

1992



ANNUAL BOOK OF ASTM STANDARDS

SECTION

6

Paints, Related Coatings, and Aromatics

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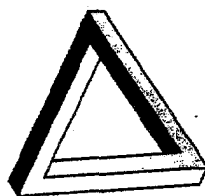


Paint—Tests for Formulated Products and
Applied Coatings

VOLUME
06.01



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ANNUAL BOOK OF ASTM STANDARDS

SECTION

6

Paints, Related Coatings, and Aromatics



VOLUME
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**Paint—Tests for Formulated Products
and Applied Coatings**

Includes standards of the following committee:

D-1 on Paint and Related Coatings and Materials

D-33 on Protective Coating and Lining Work for
Power Generation Facilities

Publication Code Number (PCN): 01-060192-14

ASTM 1916 Race Street, Philadelphia, PA 19103-1187 USA ■ (215) 299-5400 ■ TWX: 710-670-1037 ■ FAX: 215-977-9679

DUP050297153



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Library of Congress Catalog Card Number: 83-641658

ISBN 0-8031-1754-x (set)

ISBN 0-8031-1706-x (section)

ISBN 0-8031-1707-8 (volume)

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Printed in Easton, MD, U.S.A.



Foreword

Organized in 1898, ASTM has grown into one of the largest voluntary standards development systems in the world. ASTM is a not-for-profit organization which provides a forum for producers, users, ultimate consumers, and those having a general interest (representatives of government and academia) to meet on common ground and write standards for materials, products, systems, and services.

From the work of 134 standards-writing committees, ASTM publishes more than 8,500 standards each year. These standards and other related technical information are sold throughout the world.

ASTM Headquarters has no technical research or testing facilities; such work is done voluntarily by 33,000 technically qualified ASTM members located throughout the world. Membership in the Society is open to all concerned with the fields in which ASTM is active. A membership application may be found at the back of this volume. Additional information may be obtained from Member and Committee Services, ASTM, 1916 Race St., Philadelphia, PA 19103; tel. (215) 299-5454.

1992 Annual Book of ASTM Standards

The 1992 *Annual Book of ASTM Standards* consists of 68 volumes, divided among 16 sections, of which this volume is one. It contains approved ASTM standards, proposals, emergency standards, and related material. These terms are defined as follows in the *Regulations Governing ASTM Technical Committees*:

Categories:

standard—as used in ASTM, a document that has been developed and established within the consensus principles of the Society and that meets the approval requirements of ASTM procedures and regulations.

Discussion—The term “standard” serves in ASTM as an adjective in the title of documents, such as test methods or specifications, to connote specified consensus and approval. The various types of standard documents are based on the needs and usages as prescribed by the technical committees of the Society.

proposal—a document that has been approved by the sponsoring committee for publication for information and comment prior to its consideration for adoption as a standard.

Discussion—Complete balloting procedures are not required for proposals.

emergency standard—a document published by the Society to meet a demand for more rapid issuance of a specific standard document.

Discussion—The Executive Subcommittee of the sponsoring committee must recommend the publishing of an emergency standard and the Committee on Standards must concur in the recommendation. Emergency standards are not full consensus documents because they are not submitted to Society ballot.

Types:

The various types of ASTM documents are to provide a flexibility of form, communication, and usage for both the technical committees and the myriad users of ASTM documents. The type of ASTM document that is developed and titled is based on the technical content and intended use, not on the degree of consensus achieved. The three categories of ASTM documents (standard, emergency standard, and proposal) can be of the following forms and types:

classification—a systematic arrangement or division of materials, products, systems, or services into groups based on similar characteristics such as origin, composition, properties, or use.

guide—a series of options or instructions that do not recommend a specific course of action.

Discussion—Whereas a practice prescribes a general usage principle, a guide only suggests an approach. The purpose of a guide is to offer guidance, based on a consensus of viewpoints, but not to establish a fixed procedure. A guide is intended to increase the awareness of the user to available techniques in a given subject area and to provide information from which subsequent evaluation and standardization can be derived.

practice—a definitive procedure for performing one or more specific operations or functions that does not produce a test result. (Compare *test method*.)

Discussion—A practice is not a downgraded test method. Examples of practices include procedures for conducting interlaboratory testing programs or other statistical procedures; for writing statements on sampling or precision and bias; and for selection, preparation, application, inspection, necessary precautions for use or disposal, installation, maintenance, and operation of testing equipment.

specification—a precise statement of a set of requirements to be satisfied by a material, product, system, or service that indicates the procedures for determining whether each of the requirements is satisfied.

Discussion—It is desirable to express the requirements numerically in terms of appropriate units together with their limits.

terminology—a document comprising definitions of terms; descriptions of terms; and explanations of symbols, abbreviations, or acronyms.

test method—a definitive procedure for the identification, measurement, and evaluation of one or more qualities, characteristics, or properties of a material, product, system, or service that produces a test result. (Compare *practice*.)

A new edition of the Book of Standards is published annually because of additions of new standards and significant revisions to existing standards. Approximately 30 % of each volume is new or revised. Each volume contains all actions approved by the Society at least six months before the publication date. New and revised standards approved by the Society between the annual editions of any given volume are made available as separate copies. Users are cautioned to follow the most current issue of a standard except when a specific edition of a standard is cited, for example, as in a contract.

Development and Use of ASTM Standards

ASTM believes that technically competent standards result when a full consensus of all concerned parties is achieved and rigorous due process procedures are followed. This philosophy and standards development system ensure technically competent standards having the highest credibility when critically examined and used as the basis for commercial, legal, or regulatory actions.

ASTM standards are developed voluntarily and used voluntarily. Standards become legally binding only when a government body references them in regulations, or when they are cited in a contract. Any item that is produced and marked as conforming to an ASTM standard must meet all applicable requirements of that standard.

ASTM standards are used by thousands of individuals, companies, and agencies. Purchasers and sellers incorporate standards into contracts; scientists and engineers use them in laboratories; architects and designers use them in plans; government agencies reference them in codes, regulations, and laws; and many others refer to standards for guidance.

Consideration of Comments on ASTM Standards

An ASTM standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of any standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Using the Annual Book of ASTM Standards

The standards are assembled in each volume in alphanumeric sequence of their ASTM designation numbers except for Volumes 11.01, 11.02, and 05.04, which are assembled by subject matter. Volumes 05.03 and 06.03 are assembled first by committee, then in alphanumeric sequence. Each volume has a table of contents, listing the standards in alphanumeric sequence by ASTM designation; and a list by subjects, categorizing the standards according to subject. A subject index of the standards in each volume appears at the back of each volume.

Availability of Individual Standards

Each ASTM standard is available as a separate copy from ASTM. Special quantity prices and discounts can be obtained from Customer Services. When ordering, provide the ASTM standard designation and year of issue, title, quantity desired, and shipping instructions.

Caveat Statements and Policies in Standards

ASTM caveat statements on Safety Hazards and Fire Hazards are required to appear in standards where appropriate. They are located in the scope section of applicable standards. The caveats on General Statement of ASTM Policy and Patents are contained in all standards and located at the end of each standard disclaimer. For more information on the caveats see Section F2 of the Form and Style Manual for ASTM Standards.



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A complete Subject Index begins on p. 1133

This contents includes only those standards included in these books and those standards that appeared previously that have been superceded or discontinued within the past five years. Since the standards in these books are arranged in alphanumerical sequence, no page numbers are given in this contents.

The alphanumerical sequence in Volume 06.03 is divided into two sections, separating the standards of Committee D-1 and D-16.

In the serial designations prefixed to the following titles, the number following the dash indicates the year of original adoption as standard or, in the case of revision, the year of last revision. Thus, standards adopted or revised during the year 1992 have as their final number, 92. A letter following this number indicates more than one revision during that year, that is 92a indicates the second revision in 1992, 92b the third revision, etc. Standards that have been reapproved without change are indicated by the year of last reapproval in parentheses as part of the designation number, for example, (1992). A superscript epsilon indicates an editorial change since the last revision or reapproval—e1 for the first change, e2 for the second change, etc.

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§ Approved for use by agencies of the Department of Defense and, if indicated on the standard, replaces corresponding Federal or Military document. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

† Although this standard has been officially withdrawn from Society approval, a brief description is included for information only.

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PENDING STANDARDS ACTION

The following standards action was on Society ballot when this edition went to press. If approved, the action will be noted in *Standardization News* and the document will be available soon thereafter as a separate reprint.

Revision of Standards:

- D 1492 - 91
- D 3359 - 91
- D 4417 - 91

New Standards:

- D 5165 - 91
- D 5180 - 91

Practice for the Laboratory Preparation of Gelled Vehicles Using a Resin Kettle	06.02
Test Method for Quantitative Test for Turbidity in Clear Liquids	06.02



List by Subjects

1992 ANNUAL BOOK OF ASTM STANDARDS, VOLUME 06.01

PAINT—TESTS FOR FORMULATED PRODUCTS AND APPLIED COATINGS

Since the standards in this book are arranged in alphanumerical sequence, no page numbers are given in this list. The standards listed in italics are related documents included for information only and do not appear in this volume.

For List by Subjects of Volume 06.02 and 06.03, see pp. 1095 and 1100
A complete Subject Index begins on p. 1133

PAINT AND RELATED COATINGS—AS LIQUIDS

Specifications for:

- | | |
|---------------------------|---|
| D 5098 – 90 | Artists' Acrylic Emulsion Paints |
| D 4302 – 90 | Artists' Paints: Oil, Resin-Oil, and Alkyd Paints |
| D 5067 – 90 ^{ei} | Artists' Watercolor Paints |

Physical Tests

General Test Methods

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| D 969 – 85 (1989) ^{ei} | Bleeding of Traffic Paint, Determination of Degree of |
| D 4399 – 90 | Electrical Conductivity of Electrocoat Baths, Measuring |
| §D 1210 – 79 (1988) ^{ei} | Fineness of Dispersion of Pigment-Vehicle Systems |
| D 1316 – 87 | Fineness of Grind of Printing Inks by the NPRI Grindometer |
| §D 2243 – 90 | Freeze-Thaw Resistance of Water-Borne Coatings |
| D 2337 – 84 | Freeze-Thaw Stability of Multicolor Lacquers |
| D 3793 – 89 | Low-Temperature Coalescence of Latex Paint Films |
| D 2574 – 86 | Microorganisms, Resistance of Emulsion Paints to Attack in the Container |
| §D 185 – 84 (1989) ^{ei} | Particles, Coarse, in Pigments, Pastes, and Paints |
| D 5062 – 90 ^{ei} | Resin Solution, Dilutability |
| §D 869 – 85 (1989) ^{ei} | Settling of Paint, Evaluating Degree of |
| D 1309 – 88 | Settling Properties of Traffic Paints During Storage |
| D 4144 – 82 (1987) | Stability, Package, of Coatings for Ultraviolet Curing, Estimating |
| §D 1849 – 80 (1987) ^{ei} | Stability, Package, of Paint |
| D 4948 – 89 | Upper Layer Separated from a Viscous Liquid, Determination of |

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| D 3925 – 91 | Sampling Liquid Paints and Related Pigmented Coatings |
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Application Properties

Test Methods for:

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| D 4958 – 91 | Brush Drag of Latex Paints, Comparison of |
| D 2353 – 83 | Flow Ratings of Organic Coatings Using the Shell Flow Comparator (Discontinued 1992†) |
| D 2801 – 69 (1981) ^{ei} | Leveling Characteristics of Paints by Draw-Down Method (Discontinued 1990†) |
| D 4062 – 88 | Leveling of Paints by Draw-Down Method |
| D 4400 – 89a | Sag Resistance of Paints Using a Multinotch Applicator |
| D 2376 – 84 (1989) | Slump of Face Glazing and Bedding Compounds on Metal Sash |
| D 4707 – 87 | Spatter Resistance to Roller Application, Measuring |

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| D 4941 – 89 | Preparing Drawdowns of Artists' Paste Paints |
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- §D 154ⁱ
- D 483
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- §D 147

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- §D 56
- D 435ⁱ
- §D 219ⁱ
- D 421
- D 428
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- D 404
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- D 471

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- D 445
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- D 37ⁱ

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- §D 23ⁱ
- D 52ⁱ
- D 50ⁱ

Practice

- D 39ⁱ

Test Me

- D 37ⁱ
- D 40ⁱ
- D 49ⁱ

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- §D 16ⁱ

§ Approved for use by agencies of the Department of Defense and, if indicated on the standard, replaces corresponding Federal or Military document. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.
† Although this standard has been officially withdrawn from Society approval, a brief description is included for information only.

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Color and Clarity of Liquids

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| §D 1544 - 80 (1989) ⁶¹ | Color of Transparent Liquids (Gardner Color Scale) |
| D 4838 - 88 | Tinting Strength, Relative, of Chromatic Paints, Determining |
| D 2090 - 88 | Clarity and Cleanliness of Paint and Ink Liquids (see Vols 06.02, 06.03) |

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Consistency and Viscosity

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| §D 562 - 81 (1990) ⁶¹ | Consistency of Paints Using the Stormer Viscometer |
| D 4359 - 90 | Determining Whether a Material is a Liquid or a Solid |
| §D 2196 - 86 (1991) ⁶¹ | Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer |
| D 4212 - 88 | Viscosity by Dip-Type Cups |
| D 4287 - 88 | Viscosity, High-Shear, Using ICI Cone/Plate Viscometer |
| §D 1200 - 88 | Viscosity by Ford Viscosity Cup |
| D 4040 - 91 | Viscosity of Printing Inks and Vehicles by the Falling-Rod Viscometer |
| §D 1545 - 89 | Viscosity of Transparent Liquids by Bubble Time Method |

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Nonvolatile Content

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| D 5145 - 90 | Nonvolatile and Pigment Content of Electrocoat Baths |
| D 5095 - 90 | Nonvolatile Content in Silanes, Siloxanes, and Silane-Siloxane Blends Used in Masonry Water Repellant Treatments, Determination of |
| D 4713 - 87 ⁶¹ | Printing Inks, Resins Solutions, and Vehicles |
| §D 1644 - 88 | Varnishes |

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| D 2832 - 83 (1991) ⁶¹ | Paint and Related Coatings |
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Pigment Content

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| D 2371 - 85 (1990) ⁶¹ | Solvent-Reducible Paints |
| §D 2698 - 90 | Solvent-Reducible Paints Using High-Speed Centrifuge |
| D 3723 - 84 (1990) ⁶¹ | Water-Reducible Paints by Low-Temperature Ashing |

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| D 5200 - 91 | Volatile Organic Compounds of Solvent Reducible Paints in Aerosol Cans |
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| D 3960 - 91 | Paints and Related Coatings, Volatile Organic Content (VOC) |
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| D 4017 - 90 | Paints and Paint Materials by Karl Fischer Method |
| D 4942 - 89 | Water Pickup of Lithographic Printing Inks and Vehicles in a Laboratory Mixer |

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- D 4370 - 84 (1990)^{e1} Acid and Base Milliequivalent Content of Electrocoat Bath
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- D 3133 - 72 (1989)^{e1} Cellulose Nitrate in Alkyd Lacquers by Infrared Spectrophotometry, Quantitative Determination of
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D 3383-79a (1988)⁶¹

Solvent-Reducible Floor Paints, Testing

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- D 3842 - 86 (1991) Coatings for Use in Light-Water Nuclear Power Plants, Selection of Test Methods for
- D 4537 - 91 Quality and Certify Inspection Personnel for Coating Work in Nuclear Facilities, Establishing Procedures to
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- D 4538 - 90a Protective Coating and Lining Work for Power Generation Facilities

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- E 171 - 87 *Standard Atmospheres for Conditioning and Testing Materials (see Vol 15.09)*

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- D 5043 - 90 Field Identification of Coatings
- D 5009 - 89 Transfer Efficiency Under Laboratory Conditions, Evaluating and Comparing
- D 5066 - 91 Transfer Efficiency Under Production Conditions for Spray Application of Automotive Paints—Weight Basis, Determination of
- §D 95 - 83 (1990) Water in Petroleum Products and Bituminous Materials by Distillation
- E 306 - 71 (1976)^{†1} *Absolute Calibration of Reflectance Standards (see Vol 14.02)*
- E 337 - 84 (1990) *Relative Humidity by Wet- and Dry-Bulb Psychrometer (see Vols 07.01, 11.03, and 15.09)*

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- D 3980 - 88 Interlaboratory Testing of Paint and Related Materials
- D 4236 - 91 Labeling Art Materials for Chronic Health Hazards
- D 5068 - 90 Paint Brushes for Evaluation, Preparation of
- D 5069 - 90 Paint Roller Covers for Evaluation, Preparation of
- E 312 - 80 (1986) *Description and Selection of Conditions for Photographing Specimens (see Vol 14.02)*
- E 179 - 90 *Geometric Conditions, Selection of, for Measurement of Reflectance and Transmittance (see Vol 14.02)*
- E 167 - 77 (1987) *Goniophotometry of Objects and Materials (see Vol 14.02)*
- G 24 - 87 *Natural Light Exposure Tests (Sunlight and Daylight), Conducting Under Glass (see Vols 07.01 and 14.02)*

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- E 41 - 86 *Conditioning (see Vols 08.03 and 14.02)*
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Standard Test Method of Salt Spray (Fog) Testing¹

This standard is issued under the fixed designation B 117; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 811.1 of Federal Test Method Standard No. 151b. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

1. Scope

1.1 This test method sets forth the conditions required in salt spray (fog) testing for specification purposes. Suitable apparatus which may be used to obtain these conditions is described in Appendix X1. The method does *not* prescribe the type of test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results. It should be noted that there is seldom a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Comments on the use of the test in research will be found in Appendix X2. For evaluation of corrosive conditions, see Appendix X3.

NOTE 1—This method is applicable to salt spray (fog) testing of ferrous and nonferrous metals, and is also used to test inorganic and organic coatings, etc., especially where such tests are the basis for material or product specifications.

1.2 The values stated in SI units are to be regarded as standard. The inch-pound units in parentheses are provided for information.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- B 287 Method of Acetic Acid-Salt Spray (Fog) Testing²
- B 368 Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)³

¹ This test method is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved March 30, 1990. Published May 1990. Originally published as B 117 - 39 T. Last previous edition B 117 - 85 ϵ 1.

² Discontinued—See 1987 *Annual Book of ASTM Standards*, Vols 02.05 and 03.02.

³ *Annual Book of ASTM Standards*, Vol 02.05.

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁴
- D 1193 Specification for Reagent Water⁵
- D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments⁴
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁶

3. Apparatus

3.1 The apparatus required for salt spray (fog) testing consists of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control. The size and detailed construction of the apparatus are optional, provided the conditions obtained meet the requirements of this method.

3.2 Drops of solution which accumulate on the ceiling or cover of the chamber shall not be permitted to fall on the specimens being tested.

3.3 Drops of solution which fall from the specimens shall not be returned to the solution reservoir for respraying.

3.4 Material of construction shall be such that it will not affect the corrosiveness of the fog.

4. Test Specimens

4.1 The type and number of test specimens to be used, as well as the criteria for the evaluation of the test results, shall be defined in the specifications covering the material or product being tested or shall be mutually agreed upon by the purchaser and the seller.

5. Preparation of Test Specimens

5.1 Metallic and metallic-coated specimens shall be suitably cleaned. The cleaning method shall be optional depending on the nature of the surface and the contaminants, except that it shall not include the use of abrasives other than a paste of pure magnesium oxide nor of solvents which are corrosive or will deposit either corrosive or protective films. The use of a nitric acid solution for the chemical cleaning, or passivation, of stainless steel specimens is permissible when agreed upon by the purchaser and the seller. Care shall be taken that specimens are not recontaminated after cleaning by excessive or careless handling.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 15.05.

5.2 Specimens for evaluation of paints and other organic coatings shall be prepared in accordance with applicable specification(s) for the material(s) being tested, or as agreed upon by the purchaser and supplier. Otherwise, the test specimens shall consist of steel meeting the requirements of Methods D 609 and shall be cleaned and prepared for coating in accordance with applicable procedure of Methods D 609.

5.3 Specimens coated with paints or nonmetallic coatings shall not be cleaned or handled excessively prior to test.

5.4 Whenever it is desired to determine the development of corrosion from an abraded area in the paint or organic coating, a scratch or scribed line shall be made through the coating with a sharp instrument so as to expose the underlying metal before testing. The conditions of making the scratch shall be as defined in Method D 1654, unless otherwise agreed upon between the purchaser and seller.

5.5 Unless otherwise specified, the cut edges of plated, coated, or duplex materials and areas containing identification marks or in contact with the racks or supports shall be protected with a suitable coating stable under the conditions of the test, such as ceresin wax.

NOTE 2—Should it be desirable to cut test specimens from parts or from preplated, painted, or otherwise coated steel sheet, the cut edges shall be protected by coating them with paint, wax, tape, or other effective media so that the development of a galvanic effect between such edges and the adjacent plated or otherwise coated metal surfaces, is prevented.

6. Position of Specimens During Test

6.1 The position of the specimens in the salt spray chamber during the test shall be such that the following conditions are met:

6.1.1 Unless otherwise specified, the specimens shall be supported or suspended between 15 and 30° from the vertical and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested.

6.1.2 The specimens shall not contact each other or any metallic material or any material capable of acting as a wick.

6.1.3 Each specimen shall be so placed as to permit free settling of fog on all specimens.

6.1.4 Salt solution from one specimen shall not drip on any other specimen.

NOTE 3—Suitable materials for the construction or coating of racks and supports are glass, rubber, plastic, or suitably coated wood. Bare metal shall not be used. Specimens shall preferably be supported from the bottom or the side. Slotted wooden strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained, if necessary by means of secondary support at the bottom of the specimens.

7. Salt Solution

7.1 The salt solution shall be prepared by dissolving 5 ± 1 parts by weight of sodium chloride in 95 parts of water conforming to Type IV water in Specification D 1193. The salt used shall be sodium chloride substantially free of nickel and copper and containing on the dry basis not more than 0.1 % of sodium iodide and not more than 0.3 % of total

impurities. Some salts contain additives that may act as corrosion inhibitors; careful attention should be given to the chemical content of the salt. Upon agreement between purchaser and seller, analysis may be required and limits established for elements or compounds not specified in the chemical composition given above.

7.2 The pH of the salt solution shall be such that when atomized at 35°C (95°F) the collected solution will be in the pH range of 6.5 to 7.2 (Note 4). Before the solution is atomized it shall be free of suspended solids (Note 5). The pH measurement shall be made electrometrically at 25°C (77°F) using a glass electrode with a saturated potassium chloride bridge in accordance with Method E 70, or colorimetrically using bromothymol blue as indicator, or short range pH paper which reads in 0.2 or 0.3 of a pH unit (Note 6).

NOTE 4—Temperature affects the pH of a salt solution prepared from water saturated with carbon dioxide at room temperature and pH adjustment may be made by the following three methods:

(1) When the pH of a salt solution is adjusted at room temperature, and atomized at 35°C (95°F), the pH of the collected solution will be higher than the original solution due to the loss of carbon dioxide at the higher temperature. When the pH of the salt solution is adjusted at room temperature, it is therefore necessary to adjust it below 6.5 so the collected solution after atomizing at 35°C (95°F) will meet the pH limits of 6.5 to 7.2. Take about a 50-mL sample of the salt solution as prepared at room temperature, boil gently for 30 s, cool, and determine the pH. When the pH of the salt solution is adjusted to 6.5 to 7.2 by this procedure, the pH of the atomized and collected solution at 35°C (95°F) will come within this range.

(2) Heating the salt solution to boiling and cooling to 95°F for maintaining it at 95°F for approximately 48 h before adjusting the pH produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

(3) Heating the water from which the salt solution is prepared to 35°C (95°F) or above, to expel carbon dioxide, and adjusting the pH of the salt solution within the limits of 6.5 to 7.2 produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

NOTE 5—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheesecloth to prevent plugging of the nozzle.

NOTE 6—The pH can be adjusted by additions of dilute cp hydrochloric acid or cp sodium hydroxide solutions.

8. Air Supply

8.1 The compressed air supply to the nozzle or nozzles for atomizing the salt solution shall be free of oil and dirt (Note 7) and maintained between 69 and 172 kN/m² (10 and 25 psi) (Note 8).

NOTE 7—The air supply may be freed from oil and dirt by passing it through a water scrubber or at least 610 mm (2 ft) of suitable cleaning material such as sheep's wool, excelsior, slag wool, or activated alumina.

NOTE 8—Atomizing nozzles may have a "critical pressure" at which an abnormal increase in the corrosiveness of the salt fog occurs. If the "critical pressure" of a nozzle has not been established with certainty, control of fluctuation in the air pressure within plus or minus 0.7 kN/m² (0.1 psi), by installation of a suitable pressure regulator valve⁹ mini-

⁷ Registered U. S. Patent Office.

⁸ The Nullmatic pressure regulator (or equivalent) manufactured by Moore Products Co., H and Lycoming Sts., Philadelphia, PA 19124, is suitable for this purpose.

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mizes the possibility that the nozzle will be operated at its "critical pressure."⁹

9. Conditions in the Salt Spray Chamber

9.1 Temperature—The exposure zone of the salt spray chamber shall be maintained at $35 \pm 1.1 - 1.7^\circ\text{C}$ ($95 \pm 2 - 3^\circ\text{F}$). The temperature within the exposure zone of the closed cabinet shall be recorded at least twice a day at least 7 h apart (except on Saturdays, Sundays, and holidays when the salt spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

NOTE 9—A suitable method to record the temperature is by a continuous recording device or by a thermometer which can be read from outside the closed cabinet. The recorded temperature must be obtained with the salt spray chamber to avoid a false low reading because of wet-bulb effect when the chamber is open.

9.2 Atomization and Quantity of Fog—At least two clean fog collectors shall be so placed within the exposure zone that no drops of solution from the test specimens or any other source shall be collected. The collectors shall be placed in the proximity of the test specimens, one nearest to any nozzle and the other farthest from all nozzles. The fog shall be such that for each 80 cm^2 of horizontal collecting area there will be collected in each collector from 1.0 to 2.0 mL of solution per hour based on an average run of at least 16 h (Note 10). The sodium chloride concentration of the collected solution shall be 5 ± 1 weight % (Note 11). The pH of the collected solution shall be 6.5 to 7.2. The pH measurement shall be made electrometrically or colorimetrically using bromothymol blue as the indicator.

NOTE 10—Suitable collecting devices are glass funnels with the stems inserted through stoppers into graduated cylinders, or crystallizing dishes. Funnels and dishes with a diameter of 10 cm have an area of about 80 cm^2 .

NOTE 11—A solution having a specific gravity of 1.0255 to 1.0400 at 25°C (77°F) will meet the concentration requirement. The concentration may also be determined as follows: Dilute 5 mL of the collected solution to 100 mL with distilled water and mix thoroughly; pipet a 10-mL aliquot into an evaporating dish or casserole; add 40 mL of distilled water and 1 mL of 1 % potassium chromate solution (chloride-free) and titrate with 0.1 N silver nitrate solution to the first appearance of a permanent red coloration. A solution that requires between 3.4 and 5.1 mL of 0.1 N silver nitrate solution will meet the concentration requirements.

9.3 The nozzle or nozzles shall be so directed or baffled that none of the spray can impinge directly on the test specimens.

10. Continuity of Test

10.1 Unless otherwise specified in the specifications covering the material or product being tested, the test shall be continuous for the duration of the entire test period. Continuous operation implies that the chamber be closed and the spray operating continuously except for the short daily

interruptions necessary to inspect, rearrange, or remove test specimens, to check and replenish the solution in the reservoir, and to make necessary recordings as described in Section 9. Operations shall be so scheduled that these interruptions are held to a minimum.

11. Period of Test

11.1 The period of test shall be as designated by the specifications covering the material or product being tested or as mutually agreed upon between the purchaser and the seller.

NOTE 12—Recommended exposure periods are to be as agreed upon by the purchaser and seller, but exposure periods of multiples of 24 h are suggested.

12. Cleaning of Tested Specimens

12.1 Unless otherwise specified in the specifications covering the material or product being tested, specimens shall be treated as follows at the end of the test:

12.1.1 The specimens shall be carefully removed.

12.2 Specimens may be gently washed or dipped in clean running water not warmer than 38°C (100°F) to remove salt deposits from their surface, and then immediately dried. Drying shall be accomplished with a stream of clean, compressed air.

13. Evaluation of Results

13.1 A careful and immediate examination shall be made for the extent of corrosion of the dry test specimens or for other failure as required by the specifications covering the material or product being tested or by agreement between the purchaser and the seller.

14. Records and Reports

14.1 The following information shall be recorded, unless otherwise prescribed in the specifications covering the material or product being tested:

14.1.1 Type of salt and water used in preparing the salt solution,

14.1.2 All readings of temperature within the exposure zone of the chamber,

14.1.3 Daily records of data obtained from each fog-collecting device including the following:

14.1.3.1 Volume of salt solution collected in millimetres per hour per 80 cm^2 ,

14.1.3.2 Concentration or specific gravity at 35°C (95°F) of solution collected, and

14.1.3.3 pH of collected solution.

14.4 Type of specimen and its dimensions, or number or description of part,

14.5 Method of cleaning specimens before and after testing,

14.6 Method of supporting or suspending article in the salt spray chamber,

14.7 Description of protection used as required in 5.5,

14.8 Exposure period,

14.9 Interruptions in test, cause and length of time, and

14.10 Results of all inspections.

NOTE 13—If any of the atomized salt solution which has not contacted the test specimens is returned to the reservoir, it is advisable to record the concentration or specific gravity of this solution also.

⁹ It has been observed that periodic fluctuations in air pressure of $\pm 3.4\text{ kN/m}^2$ (0.5 psi) resulted in about a twofold increase in the corrosivity of the fog from a nozzle which was being operated at an average pressure of 110 kN/m^2 (16 psi). Controlling the fluctuations within $\pm 0.7\text{ kN/m}^2$ (0.1 psi), however, avoided any increase in the corrosivity of the salt fog. See Darsey, V. M. and Cavanagh, W. R., "Apparatus and Factors in Salt Fog Testing," *Proceedings, ASTM*, Vol 48, 1948, p. 153.

15. Precision and Bias¹⁰

15.1 The reproducibility of results in the salt spray test is highly dependent on the type of specimens tested and the evaluation criteria selected as well as the control of the operating variables. In any testing program, sufficient replicates should be included to establish the variability of the results. Substantial variability is often observed when similar specimens are tested in different fog chambers even though

¹⁰ Supporting data are available on loan from ASTM Headquarters. Request RR: G01-1003.

the testing conditions are nominally similar and within the ranges specified in this method.

15.2 The salt spray (fog) test is intended to reproduce the corrosion that occurs in atmospheres containing salt spray or splash. It has been widely observed, however, that rankings of different alloys or coating systems, or both, do not necessarily fall in the same order as atmospheric tests in marine or road salt splash environments. This test has been more useful in rating the relative resistance of a specific type of protective coating, for example, hot-dip zinc coatings on steel. Interpretation of the results of this method beyond this purpose must be verified by actual exposure tests.

APPENDIXES

(Nonmandatory Information)

X1. CONSTRUCTION OF APPARATUS

X1.1 Cabinets

X1.1.1 Standard salt spray cabinets are available from several suppliers, but certain pertinent accessories are required before they will function according to this method and provide consistent control for duplication of results.

X1.1.2 The salt spray cabinet consists of the basic chamber, an air-saturator tower, a salt solution reservoir, atomizing nozzles, specimen supports, provisions for heating the chamber, and suitable controls for maintaining the desired temperature.

X1.1.3 Accessories such as a suitable adjustable baffle or central fog tower, automatic level control for the salt reservoir, and automatic level control for the air-saturator tower are pertinent parts of the apparatus.

X1.1.4 The cabinet should be of sufficient size to test adequately the desired number of parts without overcrowding. Small cabinets have been found difficult to control

and those of less than 0.43-m³ (15-ft³) capacity should be avoided.

X1.1.5 The chamber may be made of inert materials such as plastic, glass, or stone, but most preferably is constructed of metal and lined with impervious plastics, rubber, or epoxy-type materials or equivalent.

X1.2 Temperature Control

X1.2.1 The maintenance of temperature within the salt chamber can be accomplished by several methods. It is generally desirable to control the temperature of the surroundings of the salt spray chamber and to maintain it as stable as possible. This may be accomplished by placing the apparatus in a constant-temperature room, but may also be achieved by surrounding the basic chamber of a jacket containing water or air at a controlled temperature.

X1.2.2 The use of immersion heaters in an internal salt solution reservoir or of heaters within the chamber is detrimental where heat losses are appreciable, because of solution evaporation and radiant heat on the specimens.

X1.2.3 All piping which contacts the salt solution or spray should be of inert materials such as plastic. Vent piping should be of sufficient size so that a minimum of back pressure exists and should be installed so that no solution is trapped. The exposed end of the vent pipe should be shielded from extreme air currents that may cause fluctuation of pressure or vacuum in the cabinet.

X1.3 Spray Nozzles

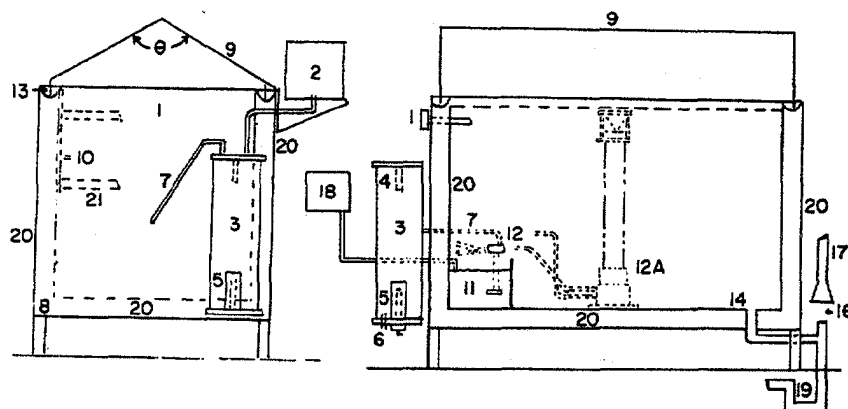
X1.3.1 Satisfactory nozzles may be made of hard rubber, plastic, or other inert materials. The most commonly used type is made of plastic. Nozzles calibrated for air consumption and solution-atomized are available. The operating characteristics of a typical nozzle are given in Table X1.1.

X1.3.2 It can readily be seen that air consumption is relatively stable at the pressures normally used, but a marked reduction in solution sprayed occurs if the level of the solution is allowed to drop appreciably during the test. Thus,

TABLE X1.1 Operating Characteristics of Typical Spray Nozzle

Siphon Height, in.	Air Flow, L/min				Solution Consumption, mL/h			
	Air Pressure, psi				Air Pressure, psi			
	5	10	15	20	5	10	15	20
4	19	26.5	31.5	36	2100	3840	4584	5256
8	19	26.5	31.5	36	636	2760	3720	4320
12	19	26.5	31.5	36	0	1380	3000	3710
16	19	26.6	31.5	36	0	780	2124	2904

Siphon Height, cm	Air Flow, dm ³ /min				Solution Consumption, cm ³ /h			
	Air Pressure, kPa				Air Pressure, kPa			
	34	69	103	138	34	69	103	138
10	19	26.5	31.5	36	2100	3840	4584	5256
20	19	26.5	31.5	36	636	2760	3720	4320
30	19	26.5	31.5	36	0	1380	3000	3710
40	19	26.6	31.5	36	0	780	2124	2904



- θ—Angle of lid, 90 to 125°
 1—Thermometer and thermostat for controlling heater (Item No. 8) in base
 2—Automatic water levelling device
 3—Humidifying tower
 4—Automatic temperature regulator for controlling heater (Item No. 5)
 5—Immersion heater, nonrusting
 6—Air inlet, multiple openings
 7—Air tube to spray nozzle
 8—Strip heater in base
 9—Hinged top, hydraulically operated, or counterbalanced
 10—Brackets for rods supporting specimens, or test table
 11—Internal reservoir
 12—Spray nozzle above reservoir, suitably designed, located, and baffled
 12A—Spray nozzle housed in dispersion tower located preferably in center of cabinet
 13—Water seal
 14—Combination drain and exhaust. Exhaust at opposite side of test space from spray nozzle (Item 12), but preferably in combination with drain, waste trap, and forced draft waste pipe (Items 16, 17, and 19).
 16—Complete separation between forced draft waste pipe (Item 17) and combination drain and exhaust (Items 14 and 19) to avoid undesirable suction or back pressure.
 17—Forced draft waste pipe
 18—Automatic levelling device for reservoir
 19—Waste trap
 20—Air space or water jacket
 21—Test table or rack, well below roof area

FIG. X1.1 Typical Salt Spray Cabinet

the level of the solution in the salt reservoir must be maintained automatically to ensure uniform fog delivery during the test.¹¹

X1.3.3 If the nozzle selected does not atomize the salt solution into uniform droplets, it will be necessary to direct the spray at a baffle or wall to pick up the larger drops and prevent them from impinging on the test specimens. Pending a complete understanding of air-pressure effects, etc., it is important that the nozzle selected shall produce the desired condition when operated at the air pressure selected. Nozzles are not necessarily located at one end, but may be placed in the center and can also be directed vertically up through a suitable tower.

X1.4 Air for Atomization

X1.4.1 The air used for atomization must be free of grease, oil, and dirt before use by passing through well-maintained filters. Room air may be compressed, heated, humidified, and washed in a water-sealed rotary pump, if the temperature of the water is suitably controlled. Otherwise

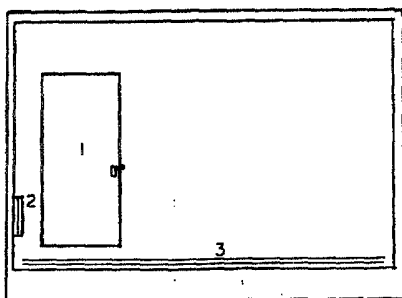
cleaned air may be introduced into the bottom of a tower filled with water, through a porous stone or multiple nozzles. The level of the water must be maintained automatically to ensure adequate humidification. A chamber operated according to this method and Appendix will have a relative humidity between 95 and 98 %. Since salt solutions from 2 to 6 % will give the same results (though for uniformity the limits are set at 4 to 6 %), it is preferable to saturate the air at temperatures well above the chamber temperature as insurance of a wet fog. Table X1.2 shows the temperatures, at different pressures, that are required to offset the cooling effect of expansion to atmospheric pressure.

X1.4.2 Experience has shown that most uniform spray chamber atmospheres are obtained by increasing the atom-

TABLE X1.2 Temperature and Pressure Requirements for Operation of Test at 95°F

	Air Pressure, psi			
	12	14	16	18
Temperature, °F	114	117	119	121
	Air Pressure, kPa			
	83	96	110	124
Temperature, °C	46	47	48	49

¹¹ A suitable device for maintaining the level of liquid in either the saturator tower or reservoir of test solution may be designed by a local engineering group, or may be purchased from manufacturers of test cabinets as an accessory.



NOTE—The controls are the same, in general as for the laboratory cabinet (Fig. X1.1), but are sized to care for the larger cube. The chamber has the following features:

- (1) Heavy insulation,
- (2) Refrigeration door with drip rail, or pressure door with drip rail, inward-sloping sill,
- (3) Low-temperature auxiliary heater, and
- (4) Duck boards on floor, with floor sloped to combination drain and air exhaust.

FIG. X1.2 Walk-in Chamber, 1.5 by 2.4 m (5 by 8 ft) and Upward in Overall Size

izing air temperature sufficiently to offset heat losses, except those that can be replaced otherwise at very low-temperature gradients.

X1.5 Types of Construction

X1.5.1 A modern laboratory cabinet is shown in Fig.

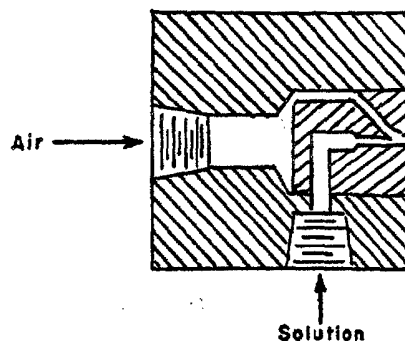


FIG. X1.3 Typical Spray Nozzle

X1.1. Walk-in chambers are not usually constructed with a sloping ceiling due to their size and location. Suitably located and directed spray nozzles avoid ceiling accumulation and drip. Nozzles may be located at the ceiling, or 0.91 m (3 ft) from the floor directed upward at 30 to 60° over a passageway. The number of nozzles depends on type and capacity and is related to the area of the test space. An 11- to 19-dm³ (3- to 5-gal) reservoir is required within the chamber, with the level controlled. The major features of a walk-in type cabinet, which differs significantly from the laboratory type, are illustrated in Fig. X1.2. Construction of a plastic nozzle, such as is furnished by several suppliers, is shown in Fig. X1.3.

X2. USE OF THE SALT SPRAY (FOG) TEST IN RESEARCH

X2.1 The detailed requirements of this method are primarily for quality acceptance and should not be construed as the optimum conditions for research studies. The test has been used to a considerable extent for the purpose of comparing different materials or finishes with an acceptable standard. The recent elimination of many cabinet variables and the improvement in controls have made the three ASTM Salt Spray Tests: Method B 117, B 287, and B 368, into useful tools for many industrial and military production and qualification programs.

X2.2 The test has been used to a considerable extent for the purpose of comparing different materials or finishes. It should be noted that there is seldom a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Informed personnel are aware of the erratic composition of basic alloys, the possibility of wide variations in quality and thickness of plated items produced on the same racks at the same time, and the consequent need for a mathematical determination of the number of specimens required to constitute an adequate sample for test purposes. In this connection it is well to point out that Method B 117 is not applicable to the study or testing of decorative chromium

plate (nickel-chromium or copper-nickel-chromium) on steel or on zinc-base die castings or of cadmium plate on steel. For this purpose Methods B 287 and B 368 are available, which are also considered by some to be superior for comparison of chemically-treated aluminum (chromated, phosphated, or anodized), although final conclusions regarding the validity of test results related to service experience have not been reached. Method B 117 is considered to be most useful in estimating the relative behavior of closely related materials in marine atmospheres, since it simulates the basic conditions with some acceleration due to either wetness or temperature or both.

X2.3 When a test is used for research, it may prove advantageous to operate with a different solution composition or concentration or at a different temperature. In all cases, however, it is desirable to control the temperature and humidity in the manner specified, and to make certain that the composition of the settled fog and that of the solution in the reservoir are substantially the same. Where differences develop, it is necessary to control conditions so that the characteristics of the settled fog meet the specified requirements for the atmosphere.

X2.4 Material specifications should always be written in terms of the standard requirements of the appropriate salt spray method, thereby making it possible to test a variety of materials from different sources in the same equipment.

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X3. EVALUATION OF CORROSIVE CONDITIONS

X3.1 General—This appendix covers test panels and procedures for evaluating the corrosive conditions within a salt spray cabinet. The procedure involves the exposure of steel test panels and the determination of their mass losses in a specified period of time. This may be done monthly or more frequently to insure consistent operation over time. It is also useful for correlating the corrosive conditions among different cabinets.

X3.2 Test Panels—The required test panels, 76 mm by 127 mm by 0.8 mm, are made from SAE 1010 commercial grade cold-rolled carbon steel (UNS G10080).

X3.3 Preparation of Panels Before Testing—Clean panels before testing by degreasing only, so that the surfaces are free of dirt, oil, or other foreign matter that could influence the test results. After cleaning, weigh each panel on an analytical balance to the nearest 1.0 mg and record the mass.

X3.4 Positioning of Test Panels—Place a minimum of two weighed panels in the cabinet, with the 127-mm length supported 30° from vertical. Place the panels in the prox-

imity of the condensate collectors. (See Section 6.)

X3.5 Duration of Test—Expose panels to the salt fog for 48 to 168 h.

X3.6 Cleaning of Test Panels After Exposure—After removal of the panels from the cabinet, rinse each panel immediately with running tap water to remove salt, and rinse in reagent grade water (see Specification D 1193, Type IV). Chemically clean each panel for 10 min at 20–25°C in a fresh solution prepared as follows:

Mix 1000 mL of hydrochloric acid (Sp. Gr. 1.19) with 1000 mL reagent grade water (D 1193, Type IV) and add 10 grams of hexamethylene tetramine. After cleaning, rinse each panel with reagent grade water (Type IV) and dry (see 12.2).

X3.7 Determining Mass Loss—Immediately after drying, determine the mass loss by reweighing and subtracting panel mass after exposure from its original mass. The mass should not vary 20 % from test to test.

X3.7.1 Data generated in the Interlaboratory Study using this method are available from ASTM as Research Report.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Last ASTM Designation: B 287 - 74 (1980)^{e1}

Standard Method of Acetic Acid-Salt Spray (Fog) Testing

This method sets forth the conditions required in acetic acid-salt spray (fog) testing for specification purposes. Formerly under the jurisdiction of Committee G-1 on Corrosion of Metals, this method was discontinued in 1988 and replaced by G 85.



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appearance
scratches
acid neutralizing
acid
general
acid value
acrylic
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Standard Terminology Relating to Paint, Varnish, Lacquer, and Related Products¹

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This standard is issued under the fixed designation D 16; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

abrasion resistance (coatings)—the ability of a coating to resist being worn away and to maintain its original appearance and structure when subjected to rubbing, scraping, or wear.

acid number (coatings)—the number of milligrams of potassium hydroxide (KOH) required to neutralize the free acids in 1 g of an oil, resin, varnish, or other substance; generally reported on the nonvolatile content.

acid value (coatings)—see **acid number**.

acrylic resin—under **resin, synthetic**, see **acrylic resin**.

adhesion promoters—materials built into the binder or added to the paint to form primary bonds to either the substrate or the previously applied coating with the specific aim of improving the dry or wet adhesion, or both.

alkyd resin—under **resin, synthetic**, see **alkyd resin**.

architectural coatings—coatings intended for on-site application to interior or exterior surfaces of residential, commercial, institutional or industrial buildings.

asbestos—see **industrial talc and industrial talc, nonasbestos type**.

associative thickener—water soluble polymers containing hydrophobic groups that are capable of nonspecific hydrophobic association similar to surfactants that elevate viscosity presumably by association between thickener particles or thickener and dispersed particles that may be present in the aqueous system such as latex particles rather than through high molecular weight or chain stiffness of the thickener molecules themselves.

autodeposition—a single-step immersion metal finishing process in which an organic coating is applied by means of unique surface chemical reactions carried out in an aqueous latex dispersion, also referred to as chemiphoresis. Components within the bath give rise to chemical reactions that slightly solubilize the metallic surface and lead to destabilization, deposition, and coalescence of the dispersed latex particles at that surface.

DISCUSSION—The deposition rate of the latex is controlled by the rate of surface solubilization. The process does not require any pretreatments such as phosphating, needs no external energy input, and gives rise to deposition wherever the solution wets the substrate. Irregularly shaped parts can be uniformly coated.

baking finish—a paint or varnish that requires baking at temperatures above 150°F (65°C) for the development of desired properties.

baking temperature—a temperature above 150°F (65°C).

batch—the total quantity of a material produced in a single final mixing operation after all production processes are complete, or just prior to filling.

bituminous varnish—under **varnish**, see **bituminous varnish**.

bleeding—the diffusion of coloring matter through a coating from the substrate; also, the discoloration arising from such diffusion. In the case of printing ink, the spreading or running of a pigment color by the action of a solvent such as water or alcohol.

blistering resistance (coatings)—the ability of a coating to resist the formation in the film of dome-shaped, liquid- or gas-filled projections resulting from local loss of adhesion and lifting of the film from the previously applied coating or the substrate.

bulking value—solid volume of a unit weight of material, usually expressed as gallons per pound. For practical purposes this is 0.120 divided by the specific gravity.

caulking compound—a soft, plastic material, consisting of pigment and vehicle, used for sealing joints in buildings and other structures where normal structural movement may occur. Caulking compound retains its plasticity for an extended period after application. It is available in forms suitable for application by gun and knife and in extruded preformed shapes.

cellulose lacquer—see **lacquer**.

chalking resistance (coatings)—the ability of a pigmented coating to resist the formation of a friable powder on its surface caused by the disintegration of the binding medium by degradative weather factors.

checking resistance (coatings)—the ability of a coating to resist slight breaks in the film that do not penetrate to the previously applied coating or to the substrate. The breaks should be called cracks if penetration extends to the previously applied coating or to the substrate. See **cracking resistance**.

chipping resistance (coatings)—the ability of a coating or layers of coatings to resist removal, usually in small pieces, resulting from impact by hard objects or from wear during service.

coating—a liquid, liquefiable or mastic composition that is converted to a solid protective, decorative, or functional adherent film after application as a thin layer.

color of an object—the aspect of the appearance of an object dependent upon the spectral composition of the incident light, the spectral reflectance or transmittance of the

¹ These definitions are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.16 on Definitions.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 16 - 11. Last previous edition D 16 - 90a.

object, and the spectral response of the observer.

hue—the attribute by which a perceived color is distinguished as red, yellow, green, blue, purple, or a combination of these. (White, gray, and black colors possess no hue.)

lightness—the attribute by which a perceived color is judged to be equivalent to a member of the continuous series of grays ranging from black to white.

saturation—the attribute by which a perceived color is judged to depart from gray of equal lightness toward a pure hue.

contrast ratio—ratio of the reflectance of a dry paint film over a black substrate of 5 % or less reflectance, to the reflectance of the same paint, equivalently applied and dried, over a substrate of 80 % reflectance.

coverage, coverage rate, covering power—ambiguous terms that are used in some instances to refer to hiding power and in others to mean spreading rate. The precise terms **hiding power** and **spreading rate** are preferred.

cracking resistance (coatings)—the ability of a coating to resist breaks of the film where the breaks extend through to the surface painted and the previously applied coating or the substrate is visible. The use of a minimum magnification of 10 diameters is recommended in cases where it is difficult to differentiate between cracking and checking. See **checking resistance**.

dirt (coatings)—see **soil**.

dirt resistance (coatings)—the ability of a coating to resist soiling by foreign material, other than microorganisms, deposited on or embedded in the dried coating.

distinctness-of-image gloss—the sharpness with which image outlines are reflected by the surface of an object.

dope—a composition, usually a cellulosic lacquer, for application on textiles and leathers.

drier—a composition that accelerates the drying of oil, paint, printing ink, or varnish. Driers are usually metallic compositions and are available in both solid and liquid forms.

drying oil—an oil that possesses to a marked degree the property of readily taking up oxygen from the air and changing to a relatively hard, tough, elastic substance when exposed in a thin film to the air.

durability—a relative term indicating degree of permanency. It may be applied to individual protective, decorative, or functional properties, for example, "the durability of gloss," but if used in a general way, for example, "the excellent durability of a paint," implies the ability of the described coating to retain, to the indicated degree, all the properties required for the continued service of the coating.

edge-tracking (coatings)—a residual, discernible pattern in a roller-applied coating characterized by trails from either or both ends of the roller.

emulsion paint—under **paint**, see **emulsion paint**.

enamel—a paint that is characterized by an ability to form an especially smooth film.

erosion resistance (coatings)—the ability of a coating to withstand being worn away by chalking or by the abrasive action of water or windborne particles of grit. The degree of resistance is measured by the amount of the coating retained. See **abrasion resistance**.

ester gum—under **resin**, synthetic, see **ester gum**.

extended pigments—organic pigments diluted with an extender (for example, alumina trihydrate, blanc fixe, or calcium carbonate).

filler—a pigmented composition for filling the pores or irregularities in a surface preparatory to application of other finishes.

fire-retardant—a descriptive term which implies that the described product, under accepted methods of test, will significantly: (a) reduce the rate of flame spread on the surface of a material to which it has been applied, or (b) resist ignition when exposed to high temperatures, or (c) insulate a substrate to which it has been applied and prolong the time required to reach its ignition, melting, or structural-weakening temperature.

filiform corrosion resistance (coatings)—the ability of a coating to resist that type of corrosion of metal substrates characterized by a definite thread-like structure and directional growth that occurs under coatings.

fire-retardant coating—a coating that will do one or more of the following: (1) reduce the flame spread on the substrate over which the coating is applied, sometimes at the sacrifice of the coating (see **intumescent coating**); (2) resist ignition of the substrate when exposed to high temperature; or (3) insulate the substrate to which the coating is applied and thereby prolong the time required to reach its ignition, melting or structural-weakening temperature.

flaking resistance (coatings)—the ability of a coating to resist the actual detachment of film fragments either from the previously applied coating or the substrate. Flaking is generally preceded by cracking, checking, or blistering and is the result of loss of adhesion. Also known as **scaling resistance**.

flattening agent—a material added to paints, varnishes, and other coating materials to reduce the gloss of the dried film.

forced drying temperature—a temperature between room temperature and 150°F (65°C).

fossil resin—under **resin**, natural, see **fossil resin**.

gallon, U. S.—a volume equal to 231 in.³ For paint, varnish, lacquer, and related products this is measured at 77°F (25°C).

glaze—a very thin coating of a paint product usually a semi-transparent coating tinted with Van Dyke brown, burnt sienna, or a similar pigment, applied on a previously painted surface to produce a decorative effect.

glazing compound—a dough-like material consisting of pigment and vehicle, used for sealing window glass in frames. It differs from putty in that it retains its plasticity for an extended period.

grinding japan—see **japan**, grinding.

grit—the coarse foreign particles in paint materials and coatings, often of irregular shape, that are hard, abrasive, and resistant to disintegration.

hiding power—the ability of a paint, or paint material as used, to hide or obscure a surface to which it has been uniformly applied. When expressed numerically, it is generally in terms of the number of square feet over which a gallon of paint, or pound of pigment, as used, can be uniformly spread to produce a specified contrast ratio (see

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contrast ratio). The term covering power has no specific relationship to hiding power, and actually has no precise meaning.

hue—under color of an object, see *hue*.

hydroxyl number—the number of milligrams of potassium hydroxide (KOH) equivalent to the hydroxyl content of 1 g of sample.

industrial talc—a mineral product varying in composition from that approaching the theoretical formula of talc, $Mg_3Si_4O_{10}(OH)_2$, to mixtures of talc and other naturally associated minerals, some of which may be fibrous as defined in ASTM Definitions D 2946, Terms Relating to Asbestos. These fibrous minerals may or may not be asbestos.

industrial talc, nonasbestos type—industrial talc of which less than 2 particles per 100 particles (by light microscopy) are asbestos fibers, where "asbestos fiber" is defined as being both a fiber by Definitions D 2946 and one of the asbestiform varieties of serpentine, riebeckite, cummingtonite (which are chrysotile, crocidolite and amosite, respectively), anthophyllite, tremolite, or actinolite. The nonasbestiform varieties of these same minerals are not asbestos.

intumescent coating—a fire-retardant coating (which see) that when heated forms a foam produced by nonflammable gases, such as carbon dioxide and ammonia. This results in a thick, highly insulating layer of carbon (about fifty times as thick as the original coating) that serves to protect the coated substrate from fire.

japan—a varnish yielding a hard, glossy, dark-colored film. Japans are usually dried by baking at relatively high temperatures.

japan, a vehicle for japan colors; frequently contains shellac.
japan color—a paste containing pigment and a grinding japan vehicle used for lettering and decoration.

japan drier—a resinate-base liquid drier.

lacquer—a coating composition that is based on synthetic thermoplastic film-forming material dissolved in organic solvent that dries primarily by solvent evaporation. Typical lacquers include those based on nitrocellulose, other cellulose derivatives, vinyl resins, acrylic resins, etc.

lake—a special type of pigment consisting essentially of an organic soluble coloring matter combined more or less definitely with an inorganic base or carrier. It is characterized generally by a bright color and a more or less pronounced translucency when made into an oil paint.

Under this term are included two (and perhaps three) types of pigment: (a) the older original type composed of hydrate of alumina dyed with a solution of the natural organic color, (b) the more modern and far more extensive type made by precipitating from solution various coal-tar colors by means of a metallic salt, tannin, or other suitable reagent, upon a base or carrier either previously prepared or coincidentally formed, and (c) a number combining both types in varying degree might be regarded as a third class.

lap (coatings)—the region where one area of a coated surface merges into an adjacent freshly-coated area during application of a single coat to the entire surface.

DISCUSSION—The objective of the painter is to avoid showing the lap.

latex paint—under paint, see *latex paint*.

lightness—under color of an object, see *lightness*.

maleic resin—under resin, synthetic, see *maleic resin*.

liquid—(flammability regulations) a substance that has a definite volume but no definite form, except such given by its container. It has a viscosity of 1×10^{-3} to 1×10^3 St (1×10^{-7} to 1×10^{-1} $m^2 s^{-1}$) at 104°F (40°C) or an equivalent viscosity at agreed upon temperature. (This does not include powders and granular materials.) Liquids are divided into two classes:

Class A, low viscosity—a liquid having a viscosity of 1×10^{-3} to 25.00 St (1×10^{-7} to 25.00×10^{-4} $m^2 s^{-1}$) at 104°F (40°C) or an equivalent viscosity at an agreed upon temperature.

Class B, high viscosity—a liquid having a viscosity of 25.01 to 1×10^3 St (25.01×10^{-4} to 1×10^{-1} $m^2 s^{-1}$) at 104°F (40°C) or an equivalent viscosity at an agreed upon temperature.

mass color—the color, when viewed by reflected light, of a pigment-vehicle mixture of such thickness as to obscure completely the background. Sometimes called over-tone or mass-tone.

mass-tone—see *mass color*.

melamine resin—under resin, synthetic see *melamine resin*.

metal marking resistance—the ability of a coating to withstand streaking or marking when a metal object is rubbed against or dragged across the surface of the coating.

mildew (fungus) resistance (coatings)—the ability of a coating to resist fungus growth that can cause discoloration and ultimate decomposition of a coating's binding medium.

mildewstat—a chemical agent that inhibits the growth of mildew.

mohair paint roller cover—a cover in which the paint applying material is woven of short pile velour that contains wool or angora goathair.

natural resin—see *resin, natural*.

nondrying oil—an oil that does not of itself possess to a perceptible degree the power to take up oxygen from the air and lose its liquid characteristics.

nonvolatile content—the portion of a coating that does not evaporate during drying or curing under specified conditions, comprising the binder and, if present, the pigment. (The percent volatile content is obtained by subtracting the nonvolatile content from 100.)

nonvolatile vehicle—the liquid portion of a paint excepting its volatile thinner and water.

oil color—an oil paint containing a high concentration of colored pigment, commonly used for tinting paint.

oil paint—under paint, see *oil paint*.

oil varnish—under varnish, see *oil varnish*.

opacity—the degree of obstruction to the transmission of visible light.

In this sense "opacity" is a relative term, it being considered that given a film sufficiently thin, in paint technology at least, there is no absolutely opaque substance.

over-tone—see *mass color*.

paint vb—to apply a thin layer of a coating to a substrate by brush, spray, roller, immersion, or any other suitable means.

paint n, general—a pigmented coating. See *coating*.

paint n, specific—a classification sometimes employed to distinguish pigmented drying oil coatings ("paints") from synthetic enamels and lacquers.

emulsion paint—a paint, the vehicle of which is an emulsion of binder in water. The binder may be oil, oleoresinous varnish, resin, or other emulsifiable binder.

latex paint—a paint containing a stable aqueous dispersion of synthetic resin, produced by emulsion polymerization, as the principal constituent of the binder. Modifying resins may also be present.

oil paint—a paint that contains drying oil or oil varnish as the basic vehicle ingredient.

paste paint—a paint in which the pigment is sufficiently concentrated to permit a substantial reduction with vehicle before use.

water paint—a paint, the vehicle of which is a water emulsion, water dispersion, or ingredients that react chemically with water.

paint brush—a paint application tool consisting of a flexible brushing part composed of long filamentary material (brushing material) bound to a handle.

DISCUSSION—Typical types include designs for varnish, enamel, sash and wall painting. They are manufactured in a range of shapes and sizes.

paint brush bristle—hair of the swine (for example: pig, hog, boar), used in brushing material.

paint brush ferrule—outer band that joins the brushing material to the handle.

paint brush filament—a synthetic polymer extrusion used in brushing material.

paint brush head—brush without the handle.

paint brush length clear—also called "length out", the exposed length of the brushing material from the ferrule to the tip end.

paint brush thickness—measurement of the brushing material across the narrow opening of the ferrule.

paint pad—a paint application tool consisting of short filamentary material usually bonded to a flat, resilient backing connected to a handle designed to apply paint by a wiping action.

paint roller—a complete paint application tool consisting of a roller frame and a roller cover designed to apply paint by a rolling action.

paint roller core—a structural tube that forms the base of the roller cover to which paint applying material is attached.

paint roller cover—a tubular sleeve consisting of a paint applying material secured to a core.

paint roller cover pile height—also called "nap length"; the length of the roller cover paint applying material from pile backing to pile face.

paint roller frame—a frame and handle assembly designed to hold a roller cover.

paste paint—under *paint*, see *paste paint*.

penta resin—under *resin*, synthetic, see *penta resin*.

phenolic resin—under *resin*, synthetic, see *phenolic resin*.

picking—a rolling up in the lap (see *lap*) when the previously applied paint film is in a semisolid (gel-like) state of

drying, causing a tacky resistance to the brush or roller and resulting in an unsightly nonuniform appearance in the final dried film.

pigment—the fine solid particles used in the preparation of paint or printing ink and substantially insoluble in the vehicle. Asphaltic materials are not pigments except when they contain substances substantially insoluble in the vehicle in which they are used.

pigment volume—the percent by volume of pigment in the nonvolatile portion of a paint or printing ink, as calculated from bulking value and composition data. The letters PV are commonly used as an abbreviation.

pinholes—small pore-like flaws in a coating that extend entirely through the applied film and have the general appearance of pin pricks when viewed by reflected light.

plasticizer—a substance added to paint, varnish, or lacquer to impart flexibility.

primer—the first of two or more coats of a paint, varnish, or lacquer system.

printing ink—a colored or pigmented liquid or paste composition that dries to a solid film after application as a thin layer by printing machinery.

print resistance (coatings)—the ability of a coating to resist taking on the imprint due to the pressure of another surface placed against it.

putty—a dough-like material consisting of pigment and vehicle, used for sealing glass in frames, and for filling imperfections in wood or metal surfaces. See *glazing compound*.

resin, natural—a solid organic substance, originating in the secretion of certain plants or insects, which is thermoplastic, flammable, nonconductive of electricity; breaks with a conchoidal fracture (when hard); and dissolves in certain specific organic solvents but not water.

fossil resin—a natural resin of ancient origin usually found in the earth.

resin, synthetic—a synthetic substance physically similar to natural resin.

acrylic resin—a synthetic resin made from derivatives of acrylic acid.

alkyd resin—a synthetic resin made from polyhydric alcohols and polybasic acids; generally modified with resins, fatty oils or fatty acids.

ester gum—a resin made from rosin or rosin acids and a polyhydric alcohol, such as glycerine or pentaerythritol.

maleic resin—a resin made from a natural resin and maleic anhydride or maleic acid.

melamine resin—a synthetic resin made from melamine and aldehyde.

penta resin—ester gum made from rosin and pentaerythritol.

phenolic resin—a synthetic resin made from phenols and aldehydes.

styrene resin—a synthetic resin made from vinyl benzene.

urea resin—a synthetic resin made from urea and an aldehyde.

vinyl resin—a synthetic resin made from vinyl compounds.

rust (coatings)—the reddish material, primarily hydrated iron oxide, formed on iron or its alloys resulting from

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exposure to humid atmosphere or chemical attack. See white rust.

rust resistance (coatings)—the ability of a coating to protect the substrate of iron or its alloys from rusting.

saturation—under color of an object, see saturation.

scaling resistance (coatings)—See flaking resistance.

sealer—a liquid composition to prevent excessive absorption of finish coats into porous surfaces; also a composition to prevent bleeding (see size).

semidrying oil—an oil that possesses the characteristics of a drying oil but to a lesser degree. There is no definite line of demarcation between drying and semidrying oils.

shade—a term descriptive of a lightness difference between surface colors, the other attributes of color being essentially constant. A lighter shade of a color is one that has higher lightness but approximately the same hue and saturation; and a darker shade is one that has a lower lightness.

Primarily, the term "shade" is derived from shadow and designates a change in appearance analogous to that produced by a local reduction in illumination. It should, therefore, when strictly used, express only the change toward a darker color. Shade of a color has been defined by several authorities as the mixture of black with that color, thus establishing its opposite character to "tint," but by extension of its relative sense it has been frequently and widely used to include lighter shades by use of the adjective "lighter" or "paler." Although such expressions apparently involve a contradiction, it is clear that while we may have a shade of a color or darker color of the same sort, it is easy to conceive of another shade not quite so dark and therefore lighter.

sheepskin paint roller cover—a cover in which the paint applying material is wool fleece still attached to its tanned natural skin.

size—usually a liquid composition to prevent excessive absorption of all paints into plaster, old wall paint, and similar porous surfaces; also a liquid composition used as a first coat on metal to improve adhesion of succeeding coats (latter usage is limited to the metal decorating industry).

The terms **sealer** and **size** are almost synonymous, but usage has established certain differences. A sealer is ordinarily a thin varnish or clear lacquer and is usually applied on wood and metal surfaces. Ordinary painter's **size** is a thin solution of glue, starch or other water-soluble substance and is usually applied on plaster surfaces, but **size** used in metal decorating is a thin varnish.

spar varnish—under varnish, see *spar varnish*.

spirit varnish—under varnish, see *spirit varnish*.

soil (coatings)—disfiguring foreign materials such as dirt, soot, or stain, other than microorganisms, deposited on or embedded in a dried film of applied coating material; also called *dirt*.

solid—(flammability regulations) a substance that has a viscosity greater than 1×10^3 St (1×10^{-1} m² s⁻¹) at 104°F (40°C) or an equivalent viscosity at an agreed upon temperature. (This includes powders and granular materials.)

spreading rate—the area covered by a unit volume of coating material frequently expressed as square feet per gallon.

stain—a discoloration, arising from foreign materials, that penetrates into the coating.

stain—a penetrating composition that changes the color of a surface, usually transparent and leaving practically no surface film.

styrene resin—under resin, synthetic, see *styrene resin*.

surfacers—a pigmented composition for filling minor irregularities to obtain a smooth uniform surface preparatory to applying finish coats; usually applied over a primer and sandpapered for smoothness.

synthetic paint roller cover—a cover in which the paint applying material is man-made material.

synthetic resin—see resin, synthetic.

talc—see industrial talc and industrial talc, nonasbestos type.

temporary coating—a coating designed to protect or decorate a substrate for a limited time that can be readily removed either by mechanical or chemical means.

thinner—the portion of a paint, varnish, lacquer, or printing ink, or related product that volatilizes during the drying process.

tint—a color produced by the mixture of white pigment or paint in predominating amount with a colored pigment or paint, not white. The tint of a color is, therefore, much lighter and much less saturated than the color itself.

tinting strength—the power of coloring a standard paint or pigment.

toner—an organic pigment that does not contain inorganic pigment or inorganic carrying base.

undertone—the color of a thin layer of pigment-vehicle mixture applied on a white background.

urea resin—under resin, synthetic, see *urea resin*.

urethane coatings—coatings based upon vehicles containing a minimum of 10 percent by weight (nonvolatile vehicle basis) of a polyisocyanate monomer reacted in such a manner as to yield polymers containing any ratio, proportion or combination of urethane linkages, active isocyanate groups, or polyisocyanate monomer. The reaction products may contain excess isocyanate groups available for further reaction at time of application or may contain essentially no free isocyanate as supplied.

Type I, one-package prereacted—urethane coatings characterized by the absence of any significant quantity of free isocyanate groups. They are usually the reaction product of a polyisocyanate and a polyhydric alcohol ester of vegetable oil acids and are hardened with the aid of metallic soap driers.

Type II, one-package moisture cured—urethane coatings characterized by the presence of free isocyanate groups and capable of conversion to useful films by the reaction of these isocyanate groups with ambient moisture.

Type III, one-package heat cured—urethane coatings that dry on cure by thermal release of blocking agents and regeneration of active isocyanate groups that subsequently react with substances containing active hydrogen groups.

Type IV, two-package catalyst—urethane coatings that comprise systems wherein one package contains a prepolymer or adduct having free isocyanate groups capable of forming useful films by combining with a relatively small quantity of catalyst, accelerator, or crosslinking agent such as a monomeric polyol or polyamine contained in a

second package. This type has limited pot-life after the two components are mixed.

Type V, two-package polyol—urethane coatings that comprise systems wherein one package contains a prepolymer or adduct or other polyisocyanate capable of forming useful films by combining with a substantial quantity of a second package containing a resin having active hydrogen groups with or without the benefit of catalyst. This type has limited pot-life after the two components are mixed.

Type VI, one-package nonreactive lacquer—urethane solution coatings characterized by the absence of any significant quantity of free isocyanate or other functional groups. Such coatings convert to solid films primarily by solvent evaporation.

varnish—a liquid composition that is converted to a transparent or translucent solid film after application as a thin layer.

bituminous varnish—a dark-colored varnish containing bituminous ingredients. The varnish may be either of the oil or spirit type.

oil varnish—a varnish that contains resin and drying oil as the basic film-forming ingredients and is converted to a solid film primarily by chemical reaction.

spar varnish—a varnish for exterior surfaces. The name originated from its use on spars of ships.

spirit varnish—a varnish that is converted to a solid film primarily by solvent evaporation.

vehicle—the liquid portion of a paint or printing ink. Anything that is dissolved in the liquid portion of a paint or printing ink is a part of the vehicle.

vernonia oil—a low-viscosity epoxidized drying oil from the seed of an African plant, *Vernonia galamensis*, containing three reactive epoxy groups and three carbon—carbon double bonds per triglyceride molecule and is characterized by its very low viscosity and melting point.

DISCUSSION—It flows easily even at temperatures below 0°C and thus needs only a fraction of the volatile solvents usually used for other drying oils. Thus, it can be used as a reactive diluent for high solids alkyds and epoxy coating formulations.

vinyl resin—under resin, synthetic, see vinyl resin.

volatile thinner—see thinner.

volume percent solids—the portion of a coating that remains as part of the dry film expressed as percent by volume.

DISCUSSION—This contrasts to another convention of expressing solids content by weight percent. Often a percent is given without specifying whether it is volume or weight. This is confusing and leads to errors in coating calculations.

water paint—under paint, see water paint.

wet adhesion—the ability of a coating film to adhere tightly to the substrate directly beneath it under wet conditions such as rain, dew, washing, etc.

wet storage stain (coatings)—See white rust.

white rust—white corrosion products (zinc hydroxide and zinc oxide) on zinc-coated articles. They form when the parts are stored so close together that condensed moisture is entrapped between them and the air circulation is inadequate to assist drying. Also called wet storage stain. See rust.

zinc-rich primer—a primer for ferrous metals, incorporating zinc dust at a concentration sufficient to make the dried film electrically conductive thus providing cathodic protection to the ferrous substrate.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



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Designation: D 95 - 83 (Reapproved 1990)^{ε1}



Designation MPMS Chapter 10.5



Designation 74/82

An American National Standard
British Standard 4385
American Association State
Highway Transportation Standard
AASHTO No. T55

Standard Test Method for Water in Petroleum Products and Bituminous Materials by Distillation¹

This standard is issued under the fixed designation D 95; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved by the sponsoring committees and accepted by the cooperating organizations in accordance with established procedures.

This test method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

^{ε1} NOTE—Paragraphs 1.3 and 11.2 were added editorially in May 1990.

1. Scope

1.1 This test method covers the determination of water in petroleum products, tars, and other bituminous materials by the distillation method. The specific products considered during the development of this test method are listed in Table 1. For bituminous emulsions refer to Test Method D 244. This test method, along with ASTM Test Method D 4006 (API Chapter 10.2 and IP 358), supersedes the previous edition of ASTM Test Method D 95 (API Standard 2560, IP 74).

NOTE 1—With some types of oil, satisfactory results may be obtained from ASTM Test Method D 4007 (API Chapter 10.3, IP 358, and ASTM Method D 1796 API Chapter 10.6).

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see 5.1.1.2 and 5.1.1.3.*

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 86 Method for Distillation of Petroleum Products²
D 244 Test Methods for Emulsified Asphalts³

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and API Committee on Petroleum Measurement and is the direct responsibility of Subcommittee D02.02 on Joint ASTM-API Committee on Static Petroleum Measurement.

Current edition approved March 25, 1983. Published January 1984. Originally published as D 95.21. Last previous edition D 95 - 70 (1980).

² *Annual Book of ASTM Standards*, Vols 05.01 and 06.03.

³ *Annual Book of ASTM Standards*, Vol 04.03.

- D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)⁴
D 4006 Test Method for Water in Crude Oil by Distillation⁵
D 4007 Test Method for Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure)⁵
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁵
D 4177 Method for Automatic Sampling of Petroleum and Petroleum Products⁵
E 123 Specification for Apparatus for Determination of Water by Distillation⁵
2.2 *API Manual of Petroleum Measurements Standards:*⁶
Chapter 8.1 Manual Sampling of Petroleum and Petroleum Products (ASTM Practice D 4057)
Chapter 8.2 Automatic Sampling of Petroleum and Petroleum Products (ASTM Test Method D 4177)
Chapter 10.2 Determination of Water in Crude Oil by the Distillation Method (ASTM Test Method D 4006)
Chapter 10.3 Determination of Water and Sediment in Crude Oil by the Centrifuge Method (Laboratory Procedure) (ASTM Test Method D 4007)
Chapter 10.6 Determination of Water and Sediment in Fuel Oil by the Centrifuge Method (ASTM Test Method D 1796)
2.3 *British Standard:*⁷
756 Dean and Stark Apparatus

3. Summary of Test Method

3.1 The material to be tested is heated under reflux with a

⁴ *Annual Book of ASTM Standards*, Vol 05.01.

⁵ *Annual Book of ASTM Standards*, Vol 05.03.

⁶ Available from American Petroleum Institute, 1220 L St., N.W., Washington, DC 20005.

⁷ Available from American National Standards Institute, 13th Floor, 11 W. 42nd Street, New York, NY 10036.

TABLE 1 Type of Solvent-Carrier Liquid Versus Material to be Tested

Type of Solvent-Carrier Liquid	Material to be Tested
Aromatic	asphalt, tar, coal tar, water gas tar, road tar, cutback bitumin, liquid asphalt, tar acid
Petroleum distillate	road oil, fuel oil, lubricating oil, petroleum sulfonates
Volatile spirits	lubricating grease

water-immiscible solvent, which co-distills with the water in the sample. Condensed solvent and water are continuously separated in a trap, the water settling in the graduated section of the trap and the solvent returning to the still.

4. Significance and Use

4.1 A knowledge of the water content of petroleum products is important in the refining, purchase, sale, and transfer of products.

4.2 The amount of water as determined by this method (to the nearest 0.05 volume %) shall be used to correct the volume involved in the custody transfer of oil.

5. Solvent-Carrier Liquid

5.1 A solvent-carrier liquid appropriate to the material being tested (see Table 1) shall be used.

5.1.1 *Aromatic Solvent*—The following aromatic solvents are acceptable:

5.1.1.1 *Industrial Grade Xylene.*

5.1.1.2 A blend of 20 volume % industrial grade toluene and 80 volume % industrial grade xylene (Warning—Flammable. Vapor harmful. See Annex A1.1.)

5.1.1.3 *Petroleum or Coal Tar Naphtha* (Warning—Extremely flammable. Harmful if inhaled. Vapors may cause fire. See Annex), free of water, yielding not more than 5% distillates at 125°C (257°F) and not less than 20% at 160°C (320°F) when tested by Method D 86 and with a relative density (specific gravity) not lower than 0.8545 at 15.56/15.56°C (60/60°F).

5.1.2 *Petroleum Distillate Solvent*—A petroleum distillate

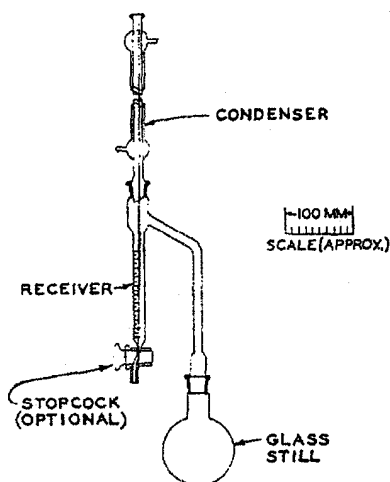


FIG. 1 Typical Assembly with Glass Still (Dean and Stark)

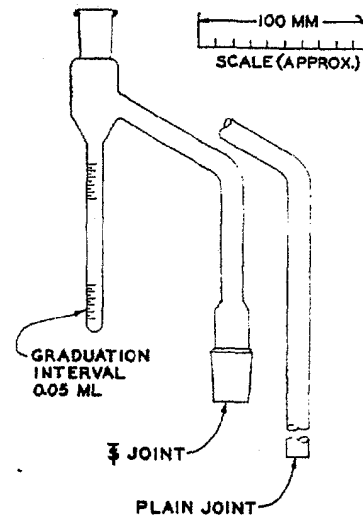


FIG. 2 Two-millilitre Receiver Showing Alternative Connections to Glass Still

solvent, 5% boiling between 90° and 100°C (194° and 212°F) and 90% distilling below 210°C (410°F), shall be used.

5.1.3 *Volatile Spirits Solvent*—The following volatile spirits solvents are acceptable:

5.1.3.1 *Petroleum Spirit*, with a boiling range of 100° to 120°C (212° to 248°F).

5.1.3.2 *Isooctane*, of 95% purity or better.

6. Apparatus

6.1 *General*—The apparatus comprises a glass or metal still, a heater, a reflux condenser, and a graduated glass trap. The still, trap, and condenser may be connected by any suitable method that produces a leakproof joint. Preferred connections are ground joints for glass and O-rings for metal to glass. Typical assemblies are illustrated in Figs. 1, 2, and 3. The stills and traps should be chosen to cover the range of materials and water contents expected.

6.2 *Still*—A glass or metal vessel with a short neck and suitable joint for accommodating the reflux tube of the trap shall be used. Vessels of 500, 1000, and 2000-mL nominal capacity have proved satisfactory.

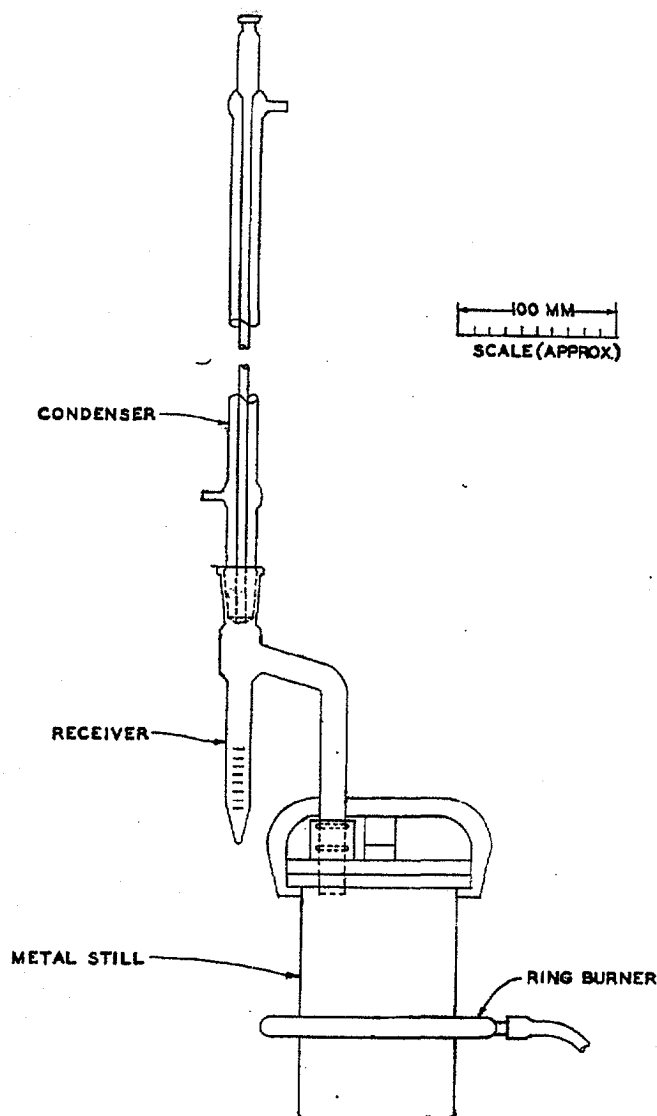
6.3 *Heater*—A suitable gas burner or electric heater may be used with the glass still. A gas ring burner with ports on the inside circumference shall be used with the metal still. The gas ring burner shall be of such dimensions that it may be moved up and down the vessel when testing materials that are likely to form or solidify in the still.

6.4 *Glassware*—Dimensions and descriptions of typical glassware for use in this method are provided in Specification E 123 and British Standard 756. A straight water-cooled condenser with a length of 400 mL is recommended in place of the length of 300 mL specified in British Standard 756.

NOTE 2—Instead of standardizing on a particular apparatus specification with respect to dimensions and style, a given apparatus will be deemed satisfactory when accurate results are obtained by the standard addition technique described in Section 7.

7. Standardization

7.1 A given assembly of apparatus will be considered



Note—Trap shall be 15 to 16 mm in inside diameter

FIG. 3 Typical Assemblies with Metal Still

satisfactory when accurate readings are obtained from the addition of known amounts of water from a calibrated buret or pipet to a clear hydrocarbon oil and tested in accordance with Section 9.

7.2 The readings shall be judged accurate if the permis-

sible limits given in Table 2 for the various sized graduated traps are not exceeded.

7.3 A reading outside the permissible limits suggests a malfunction resulting from vapor leaks, too rapid boiling, inaccuracies in calibration of the trap, or ingress of extraneous moisture. Eliminate these factors before repeating the standardization.

TABLE 2 Permissible Limits in Millilitres

Capacity of Receiver at 20°C	Volume of Water Added to Flask at 20°C	Permissible Limits for Recovered Water at 20°C
2	1	1 ± 0.05
10	1	1 ± 0.1
10	5	5 ± 0.2
25	12	12 ± 0.2

8. Sampling

8.1 Sampling is defined as all steps required to obtain an aliquot of the contents of any pipe, tank, or other system and to place the sample into the laboratory test container. Only representative samples obtained as specified in Practice D 4057 (API Chapter 8.1) and Method D 4177 (API Chapter

8.2) shall be used for this test method.

8.2 The size of the test portion should be based on the expected water content of the sample, such that the water yield does not exceed the capacity of the trap (unless a trap with a stopcock is used since excess water can be withdrawn into a graduated cylinder).

9. Procedure

9.1 Measure a suitable amount of sample to an accuracy of $\pm 1\%$ and transfer it to the still.

9.2 Measure ordinary liquid samples in a graduated cylinder of an appropriate size. Rinse the material adhering to the cylinder with one 50-mL and two 25-mL portions of the solvent-carrier liquid (see Section 5 and Table 1). Drain the cylinder thoroughly after the sample transfer and each rinsing.

9.3 Weigh solid or viscous materials directly into the still and add 100 mL of the selected solvent-carrier liquid. In cases of material with a low-water content when large samples must be used, a solvent-carrier liquid volume in excess of 100 mL may be necessary.

9.4 Glass beads or other boiling aids may be added, if necessary, to reduce bumping.

9.5 Assemble the components of the apparatus as illustrated in Figs. 1, 2, and 3 choosing the trap in accordance with the expected water content of the sample and making all connections vapor and liquid tight. If a metal still with a removable cover is used, insert a gasket of heavy paper, moistened with solvent, between the still body and the cover. The condenser tube and trap must be chemically clean to assure free drainage of water into the bottom of the trap. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture inside it. Circulate cold water through the jacket of the condenser.

9.6 Apply heat to the still, adjusting the rate of boiling so that condensed distillate discharges from the condenser at the rate of 2 to 5 drops per second. If the metal still is used, start heating with the ring burner about 76 mL (3 in.) above

the bottom of the still and gradually lower the burner as the distillation proceeds. Continue distillation until no water is visible in any part of the apparatus except in the trap, and the volume of water in the trap remains constant for 5 min. If there is a persistent ring of water in the condenser tube, carefully increase the rate of distillation or cut off the condenser water for a few minutes.

9.7 When the evolution of water is complete, allow the trap and contents to cool to room temperature. Dislodge any drops of water adhering to the sides of the trap with a glass rod or other suitable means and transfer them to the water layer. Read the volume of the water in the trap to the nearest scale division.

10. Calculation

10.1 Calculate the water in the sample, as weight or volume percent, in accordance with the basis on which the sample was taken, as follows:

$$\text{Water, \%} = V/W \times 100$$

where:

V = volume of water in trap and

W = weight (or volume of sample).

Volatile water-soluble material, if present, may be measured as water.

10.2 Report the results as the water content to the nearest 0.05% if the 2-mL receiver has been used and to the nearest 0.1% if the 10-mL or 25-mL receiver has been used with a 100-mL or 100-g sample.

11. Precision and Bias

11.1 *Precision*—The criteria described in Section 11.1.1 and 11.1.2 should be used to judge the acceptability of results (95% probability) when using the 10-mL or 25-mL traps. The precision when using the 2-mL trap has not been established.

11.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the values in Table 3 in only one case in twenty.

11.1.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the values in Table 3 in only one case in twenty.

11.2 *Bias*—A bias statement is under development.

TABLE 3 Precision

Type	Water Collected, mL	Difference, mL
Repeatability	0.0-1.0	0.1
	1.1-25	0.1 or 2 % of the mean, whichever is greater
Reproducibility	0.0-1.0	0.2
	1.1-25	0.2 or 10 % of the mean, whichever is greater

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Keep
Use v
Avoid
Avoid

A1.2 N
Warn
Harm

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENTS

A1.1 Xylene/Toluene

Warning—Flammable. Vapor harmful.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing of vapor or spray mist.
Avoid prolonged or repeated contact with skin.

Vapors may cause flash fire.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid a build-up of vapors and eliminate all sources of ignition, especially nonexplosion proof electrical apparatus and heaters.
Avoid prolonged breathing of vapors or spray mist.
Avoid prolonged or repeated skin contact.

A1.2 Naphtha

Warning—Extremely flammable.
Harmful if inhaled.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Guide for Testing Varnishes¹

This standard is issued under the fixed designation D 154; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Editorial changes made throughout in October 1989.

1. Scope

1.1 This guide covers the selection and use of procedures for testing varnishes. Some test methods are included but most sections refer to specific ASTM test methods.

1.2 Varnishes may be applied under such diverse conditions to so many different surfaces, and their dried films may be subjected to so many kinds of wear and exposure, that it is not possible to assure desired performance from a single selection of test methods and numerical results. Those skilled in varnish technology may find partial assurance of obtaining desired qualities in various types of varnishes through careful selection of the methods covered and intelligent interpretation of results.

1.3 *This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 56 Test Method for Flash Point by Tag Closed Tester²
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester²
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)³
- D 479 Test Method for Reactivity of Paint Liquids⁴
- D 523 Test Method for Specular Gloss⁵
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive⁵
- D 968 Test Method for Abrasion Resistance of Organic Coatings by Falling Abrasive⁵
- D 1200 Test Method for Viscosity by Ford Viscosity Cup⁵
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)⁶

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved May 31, 1985. Published July 1985. Originally published as D 154 - 23. Last previous edition D 154 - 76.

² Annual Book of ASTM Standards, Vols 05.01 and 06.03.

³ Annual Book of ASTM Standards, Vols 05.01 and 10.03.

⁴ Discontinued; see 1983 Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ Annual Book of ASTM Standards, Vols 05.01 and 06.03.

- D 1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus⁶
- D 1469 Test Method for Total Rosin Acids Content of Coating Vehicles⁷
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁵
- D 1542 Test Method for Qualitative Detection of Rosin in Varnishes⁸
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)⁹
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁹
- D 1546 Test Method for Performance Tests of Clear Floor Sealers⁵
- D 1639 Test Method for Acid Value of Organic Coating Materials⁵
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁵
- D 1641 Test Method for Exterior Durability of Varnishes⁵
- D 1644 Test Methods for Nonvolatile Content of Varnishes⁵
- D 1647 Test Method for Resistance of Dried Films of Varnishes to Water and Alkali⁵
- D 1729 Practice for Visual Evaluation of Color Difference of Opaque Materials¹⁰
- D 2090 Test Method for Clarity and Cleanness of Paint Liquids¹¹
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁵
- D 2369 Test Method for Volatile Content of Coatings⁵
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁵
- D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus¹²
- D 3964 Practice for Selection of Coating Specimens for Appearance Measurement⁵
- D 4039 Test Method for Reflection Haze of High Gloss Surfaces⁵
- D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser⁵
- E 308 Method for Computing the Colors of Objects by Using the CIE System¹⁰

⁷ Annual Book of ASTM Standards, Vol 06.02.

⁸ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁹ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

¹⁰ Annual Book of ASTM Standards, Vol 14.02.

¹¹ Annual Book of ASTM Standards, Vols 06.02 and 06.03.

¹² Annual Book of ASTM Standards, Vol 06.03.

LIQUID VARNISH PROPERTIES

3. Appearance

3.1 Appearance of the liquid varnish is important both for aesthetic reasons and because it indicates whether the dried film is likely to have a satisfactory appearance. Examine the liquid varnish for foreign matter, sediment or skins in accordance with Test Method D 2090.

4. Color

4.1 Most varnishes are predominantly yellow but the color of the liquid varnish is only a preliminary indication of the color of the dried varnish film. The initial color may bleach or may darken depending upon the conditions of exposure. Determine approximately and quickly the liquid color of small specimens in Gardner-Holdt tubes by comparison with the Gardner Color Standards in accordance with Test Method D 1544.

4.2 Measure the color of extremely pale varnishes by using larger specimens in 100-mL cylinders, 300 mm deep, in accordance with Test Method D 1209.

4.3 A more precise measure of color, in terms of tristimulus values, may be made on small specimens in 10-mm cells with parallel walls in accordance with Method E 308.

5. Viscosity

5.1 The viscosity of a varnish or clear vehicle is a property important in ease of application; varnishes for brush application are typically 1 to 2 St whereas varnishes with viscosities as high as 100 St may sometimes be added to lithography coatings or used as mixing vehicles for producing enamels. Viscosity is commonly measured at 77°F (25°C).

5.2 For the rapid, approximate measurement of the viscosity of transparent varnishes, determine the bubble time by Test Method D 1545. Report the viscosity either in stokes or in Gardner-Holdt letter designations as described in Table 1 of Test Method D 1545.

5.3 For a rapid, approximate measurement of the viscosity of translucent varnishes, determine the Ford cup efflux time in accordance with Test Method D 1200.

5.4 For the precise measurement of viscosity, use capillary viscometers as described in Test Method D 445.

6. Specific Gravity

6.1 Specific gravity of a varnish is the ratio of the weight of a given volume of the varnish at a given temperature to the weight of an equal volume of distilled water at the same temperature. Determine specific gravity or density at 77°F (25°C) or other agreed temperature in accordance with Test Method D 1475 which allows use of either a pycnometer or a weight per gallon cup.

7. Volatile Content

7.1 Volatile matter determination is an indication of the amount of material in the coating that will be given off to the atmosphere in the area where the coating is applied. Depending upon the method of application, the time required to vaporize the volatile and the conditions of the atmosphere surrounding the application, it is recommended that Test

TABLE 1 List of Test Methods

Test Method	Section	ASTM Designation
<i>Liquid Varnish Properties:</i>		
Appearance	3	D 2090
Color (Gardner)	4.1	D 1544
Color, Platinum-Cobalt Scale	4.2	D 1209
Color, tristimulus	4.3	E 308
Viscosity:	5	
Bubble time (Gardner-Holdt)	5.2	D 1545
Ford cup efflux	5.3	D 1200
Kinematic, high precision	5.4	D 445
Specific gravity	6.7	D 1475
Volatile content	7	D 2369
Nonvolatile matter	8	D 1644
Flash point:	9	
Tag closed-cup		D 56
Pensky Martens		D 93
Tag open-cup		D 1310
Setaflash closed tester		D 3278
Skimming	10	
Acid value	11	D 1639
Reactivity of paint liquids	12	D 479
Rosin content, quantitative	13	D 1469
Rosin content, qualitative (Lieberman-Storch and Halphen-Hicks)	13	D 1542
<i>Drying and Curing Properties:</i>		
Drying time	14	D 1640
Print free time	15	D 1640
<i>Dry Varnish Properties:</i>		
Gloss:	16	
Specular gloss		D 523
Reflective haze		D 4039
Resistance of dried films to water and alkali	17	D 1647
Abrasion resistance:	18	
Carborundum air blast		D 658
Falling abrasive		D 968
Taber abraser		D 4060
Exterior durability	19	D 1641
Color of dried film	20.1	D 2244
	20.2	D 1729
Clear floor sealers	21	D 1546

Method D 2369 be used to determine the volatile content of a varnish.

8. Nonvolatile Matter

8.1 Nonvolatile content is an indication of the amount of permanent film-forming material contained in a varnish. The normal drying of a varnish film may involve varying amounts of absorption of oxygen from the air, loss of volatile solvents, and continuing decomposition of the dried film. The net result of this process may differ somewhat from a nonvolatile determination at a temperature higher than the normal drying conditions.

8.2 With due regard to the composition of the varnish, determine the nonvolatile matter in accordance with either Method A (3 h at 220°F (105°C)) or Method B (10 min at 300°F (149°C)) of Test Methods D 1644.

8.3 As noted in Test Method D 2369, nonvolatile matter can also be calculated by subtracting the volatile content from 100.

9. Flash Point

9.1 Determine the flash point of varnishes having a

viscosity of less than 9.5 cSt at 77°F (25°C) (45 SUS at 100°F) by Test Method D 56, and of varnishes having a viscosity of more than 9.5 cSt at 77°F by Test Methods D 93. Alternatively, use Test Methods D 3278, which gives comparable results to Test Methods D 56 and D 93, and Test Method D 1310.

NOTE—Due to various U.S. Government and State regulations, it is now necessary to check with appropriate departments to determine which ASTM Test Method is applicable.

10. Skinning

10.1 Varnishes, which dry by oxidation, may form a skin in a partially filled can or in a filled can that is stored for a long time. Since skins are insoluble in the varnish, they must be removed before use if a satisfactory film is to be obtained. Use the following test to determine if a varnish has an objectionable tendency to early skin formation:

10.1.1 *Container*—A wide-mouth jar with a capacity of 8-fluid oz (235 mL) and dimensions of 4½ in. (115 mm) in height and 2 in. (50 mm) in diameter.

10.1.2 *Procedure*—Measure a 6-fluid oz (180-mL) specimen of the varnish into the glass container. Screw the cover on tightly, invert the jar, and leave in an inverted position, at rest, and in the dark (placing under a box or in a drawer is satisfactory). Examine the varnish for skinning at specified time intervals.

11. Acid Value

11.1 The acid value of a varnish is an indication of reactivity with basic pigments and, within any one type of composition, may indicate conformity to a standard method of preparation. It is not a general criterion of excellence in a protective coating.

11.2 Determine the acid value in accordance with Test Method D 1639.

12. Reactivity of Paint Liquids

12.1 Reactivity of a varnish with zinc oxide is a partial indication of the stability of the consistency of enamels made from it and various basic pigments.

12.2 Determine the reactivity in accordance with Test Method D 479.

13. Rosin Content

13.1 Improper use of rosin and its derivatives is sometimes associated with inferior performance of varnishes containing them. Qualitative tests for rosin may be employed to detect the use of a significant amount in varnishes. Quantitative determination of rosin may be used to control rosin content within limits agreeable to the purchaser and the seller.

13.2 Determine the rosin content quantitatively in accordance with Test Method D 1469 and qualitatively with Test Method D 1542.

DRYING AND CURING PROPERTIES

14. Drying Time

14.1 Small variations in film thickness, air temperature and humidity, and exposure to light and other radiation may affect drying times by ±5 %.

14.2 Determine the drying stages appropriate to the varnish under test in accordance with the applicable sections of Test Methods D 1640.

15. Print-Free Time

15.1 Varnishes intended for floors, furniture, etc., are expected to bear heavy objects for long periods without marring of the surface or adhering to the object.

15.2 Determine the print-free time in accordance with that section of Test Methods D 1640.

DRY VARNISH PROPERTIES

16. Gloss

16.1 Because varnishes are transparent or translucent, gloss must be measured on films applied to a nonreflecting substrate. The usual material is plane, black glass as described in Test Method D 2805 and similar to the gloss standards used in Test Method D 523. Gloss measurements of varnishes on wood substrates are generally not valid because the reflectance of the substrate can affect the result, but comparative tests in one laboratory of different varnishes on the same substrate may be helpful.

16.2 Gloss is usually measured in accordance with Test Method D 523 using 60° geometry. For greater sensitivity in evaluating high gloss varnishes, the 20° geometry may be used or Test Method D 4039 which uses both 20° and 60° geometries.

16.3 Prepare specimens in accordance with Practice D 3964, applying the varnish to black glass panels with a film applicator that has a clearance of 3 mils (75 µm) for varnishes with a nonvolatile content of 35 % or more and 6 mils (150 µm) for those with a nonvolatile content less than 35 %.

16.4 Allow the films to dry under the conditions specified in Test Methods D 1640 for at least 24 h. For a rapid control test the films may be force dried at 120°F (50°C) for 1 h, providing it has been established that heat acceleration does not affect the gloss of the varnish.

16.5 Measure the gloss in accordance with Test Methods D 523 or D 4039 and report.

17. Resistance of Dried Films to Water and Alkali

17.1 Performance of varnish films is indicated in part by measurement of resistances of their dried films to water, alkali, and other reagents.

17.2 Determine the resistance to water and alkali in accordance with Test Method D 1647.

18. Abrasion Resistance

18.1 The durability and general performance of varnish films on floors is influenced by many factors such as mechanical properties, film thickness, and exposure to light, cleaning materials, various types of soil, and foot or vehicular traffic, so that no one set of tests are adequate to ensure universally satisfactory service.

18.2 An indication of the resistance to abrasion in service may be determined by Test Methods D 658, D 968, or D 4060. All these test methods are suitable for interlaboratory use only when results are compared by ranking instead of numerical values.

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19. Exterior Durability

19.1 Durability of varnish films varies so widely with exposure to varying conditions of atmosphere and light or other radiation, that any one set of conditions is only a preliminary indication of general durability.

19.2 Determine the exterior durability in accordance with Test Method D 1641.

20. Color of Dried Film

20.1 The color of the dry film is usually more significant than that of the liquid varnish in establishing whether the color of a varnished object will be acceptable. This can be evaluated by determining, in accordance with Test Method D 2244, the color difference of white structural glass before and after application and drying of a varnish film.

20.2 If a varnish with a dry color known to be satisfactory is available, a standard panel can be prepared and used for visual color comparison in accordance with Practice D 1729.

21. Clear Floor Sealers

21.1 Clear floor sealers are varnishes of relatively low viscosity for application to wooden or other porous surfaces and are variously used as either the sole coating or for making the substrate more uniform for application of wax, varnish, or other coatings.

21.2 Evaluate clear floor sealers in accordance with Test Method D 1546.

22. Precision

22.1 No specific precision statement is made for this guide since this information is included in the referenced methods, if available.

23. Keywords

23.1 drying and curing properties; varnish acid value; varnish flash point; varnish nonvolatile contents; varnish physical properties; varnish specific gravity; varnish volatile contents; varnish viscosity

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Methods for Coarse Particles in Pigments, Pastes, and Paints¹

This standard is issued under the fixed designation D 185; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Methods 4091, 4101 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Editorial changes were made throughout in March 1989.

1. Scope

1.1 These test methods cover the determination of the amount of coarse particles in dry pigments and of coarse particles and skins in mixtures of pigments and vehicles.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

E11 Specification for Wire-Cloth Sieves for Testing Purposes²

3. Significance and Use

3.1 In production of paints, smoothness of the paint film is of paramount importance. Agglomerates or coarse particles larger than 45 μm are difficult to disperse and may prevent obtaining a smooth film. These test methods are a valuable quality control test for grading raw materials.

4. Apparatus

4.1 The apparatus shall consist of a 3-in. (75-mm) 45- μm (No. 325) sieve conforming to Specification E 11. A 3-in. 45- μm sieve for comparison purposes should be retained in the laboratory as a reference standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it and on the reference standard sieve a comparison test, using a pigment that has a considerable amount of coarse particles. A reserve stock of such a pigment should be kept for this purpose.

5. Procedure for Insoluble Dry Pigments, Except Metallic Aluminum and Bronze Powders

5.1 Dry the sieve in an oven at $105 \pm 2^\circ\text{C}$, cool, and then

weigh on an analytical balance, recording the weight to 1 mg.

5.2 Weigh a specimen (25 g for basic carbonate and basic sulfate white leads, 25 g for red lead and mercuric oxide, 2 g for black pigments of low specific gravity, 3 g for Prussian blues and graphite, and 10 g for all other pigments) of the pigment to be tested on an analytical balance to 1 mg. Wet the sieve on both sides with alcohol and transfer the specimen of pigment to the sieve and wet with alcohol.

5.3 Hold the sieve under a tap delivering about 300 to 500 mL of the wash liquid (water) per minute. By slightly shaking the sieve, the pigment will be rapidly carried through. A soft camel's-hair brush may be used in aiding the operation. If the sieve is held at a slight angle so that the pigment gradually collects at one edge during the washing process, and then rotated, the pigment may be brushed out rapidly, with no risk of clogging the sieve.

5.4 After most of the finely divided portion of the pigment has passed through the sieve (from 2 min to 1 h, according to the kind of pigment), place the sieve in an 8-in. (200-mm) porcelain dish containing 250 mL of the wash liquid so that the sieve is covered to a depth of about $\frac{1}{2}$ in. Brush the pigment remaining on the sieve with a soft 1-in. (25-mm) camel's-hair brush at the rate of two strokes per second during two periods of 10 s each. Raise the sieve from the dish after each 10-s period to let the liquid on the sieve run through. Change the liquid in the dish after every two brushing periods. Continue this operation until the wash liquid passing over the residue and through the sieve is clear and free from solid particles. When the washing appears to be complete, collect about 200 mL of the wash liquid, after passing over the residue and through the sieve, in a clean 400-mL beaker. Stir the liquid vigorously, and set the beaker on a black surface in the case of white pigments and on a white surface in the case of colored pigments. The washing is not considered complete until such a test fails to show any particles collected about the middle of the bottom of the beaker.

NOTE 1—Occasionally, pigments will be found that foam when water is used as the wash liquid. In such instances, during the last washing in the porcelain dish the use of a liquid that breaks down the foaming and is readily miscible with water, such as alcohol, will usually overcome this difficulty.

5.5 When the washing is complete wash the pigment particles adhering to the brush back onto the sieve and wipe off the water below the sieve. Add a few drops of alcohol and then of ether to expedite drying. Dry the sieve for 1 h at

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.31 on Pigment Specifications.

Current edition approved Oct. 26, 1984. Published December 1984. Originally published as D 185 - 37. Last previous edition D 185 - 78.

² Annual Book of ASTM Standards, Vol 14.02.

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105°C, cool, and weigh as described in 5.1. Calculate the percent of coarse particles.

6. Procedure for Metallic Aluminum and Bronze Powders

6.1 In the case of metallic aluminum and bronze powders follow the procedure described in Section 5 except use 5 g of the material as the specimen and denatured alcohol, instead of water, as the wash liquid.

7. Procedure for Carbon Black in Pellet Form

7.1 Dry, cool, and weigh the sieve as described in 5.1.

7.2 Prepare a stock solution of dispersing agent³ in water; using as high a concentration of dispersing agent as possible without losing fluidity. When solution has been obtained, filter through coarse filter paper. A quart of this solution will be sufficient for several tests.

7.3 Crush the pellets between two glass plates approximately 12-in. (305-mm) square, using a gentle rotating motion. If 10 g of carbon black are taken, crush about 2 g at a time. Weigh into a 600-mL beaker 10 g of the crushed particles. Add enough of the dispersing solution to make a heavy paste and mix well to incorporate thoroughly the carbon black. Dilute with water to about 300 mL; then pour into the clean 45- μ m (No. 325) sieve. First pass the tap water through a 45- μ m sieve.

7.4 Using a small camel's-hair brush, gently brush the mix through the sieve, running tap water slowly through at the same time. When it appears that all the dispersed black has gone through, stop the tap water and continue brushing until most of the water remaining on the sieve has gone through. Add a few millilitres of the stock solution of the dispersing agent, thoroughly mix with the brush, then turn on the tap water again and wash through as before. Repeat this operation until no dispersed black comes through the sieve. When about 0.5 g of sand-like material is left on the sieve, the end point is being approached. At this point, adding the dispersing solution and gently rubbing it into the residue with the finger still produces a colloidal dispersion of black which easily passes through the sieve. From this point on, before each addition of dispersing agent, work the residue into the center of the sieve with the tap water. Many washings and many additions of dispersing solution are required in order to reach the end point. Take care to make certain that all the black has been washed through the sieve.

7.5 Dry the sieve in an oven at 105°C for 1 h, cool, and weigh the residue. Calculate the percent of coarse particles. If the value thus obtained is greater than that specified, proceed as follows to remove any adhered carbon that will pass through the 45- μ m (No. 325) sieve: Transfer the particles on the sieve to a piece of white bond paper and gently rub the carbon onto the paper with the finger. When no further real blackening of the paper occurs, carefully transfer the residue to the balance pan, weigh, and recalculate the percent of coarse particles.

8. Procedure for Water-Soluble Pigments, Pastes in Oil, Pastes in Japan, and Mixed Paints

8.1 Dry, cool, and weigh the sieve as described in 5.1.

8.2 For water-soluble pigments use 10 ± 1 g as the specimen. For pastes in oil, pastes in Japan, and mixed paints use 25 ± 1 g as the specimen. For white leads and red lead use 50 ± 1 g as the specimen.

8.3 Weigh the specimen to 1 mg and transfer to a 250-mL beaker. Slowly add 100 mL of kerosine to the contents of the beaker, mixing thoroughly by use of a stirring rod with flattened end. Break up all lumps but do not grind the material.

8.4 Wet the sieve on both sides with kerosine; then transfer the contents of the beaker to the sieve using a wash bottle filled with kerosine.

8.5 Remove small particles retained on the stirring rod or beaker walls with a camel's-hair brush. Rinse the brush with kerosine.

8.6 When the washing is complete, wash the pigment particles adhering to the brush back onto the sieve and wipe off kerosine below the sieve. Dry the sieve for 1 h at 105°C, cool, and weigh as described in 5.1. Calculate the percent of particles.

9. Procedure for Ship-Bottom Paints Containing Resins and Alcohol

9.1 In the case of ship-bottom paints containing resins and alcohol follow the procedure described in Section 8, with specimen weight of 25 ± 1 g, but use denatured alcohol, instead of kerosine, as the wetting medium, for mixing with paint, and as the wash liquid.

10. Procedure for Cellulose Ester Lacquers

10.1 In the case of cellulose ester lacquers follow the procedure described in Section 8, with specimen weight of 25 ± 1 g of the material but use a mixture of equal parts of ethyl acetate, toluene, and denatured alcohol, instead of kerosine, as the wetting medium, for mixing with the lacquer, and as the wash liquid.

11. Procedure for Latex and Emulsion Paints

11.1 *Apparatus*—As described in Section 4 except that sieves used shall be as follows:

11.1.1 *Flat Paints*, a 3-in. (75-mm) 90- μ m (No. 170) sieve.

11.1.2 *Gloss and Semi-Gloss Paints*, a 3-in. (75-mm) 75- μ m (No. 200) sieve.

11.2 Dry, cool, and weigh the sieve as described in 5.1.

11.3 Weigh 25 g of paint to ± 1 g into a tared 250-mL beaker. Without delay, start adding 100 mL of water to the paint in the beaker, slowly and with hand stirring sufficient to thoroughly mix the paint with the water. Wet the sieve on both sides with water and transfer the contents of the beaker gradually to the sieve, using a wash bottle containing water. Small particles retained on the stirrer or beaker walls may be removed with a camel's-hair brush. Instead of tap water, use reagent water for diluting the paint sample, in the wash bottle, and for washing the paint through the sieve. The water may be delivered from a reservoir for the last step.

11.4 Follow the procedure described in 5.3. In using the camel's-hair brush, take care not to crush agglomerates that would not be broken down in normal application of the paint. Rinse the brush into the sieve at end of use.

11.5 Finally, dry, cool, and weigh the sieve for 1 h, as

³ Any dispersing agent specific for carbon black may be used, such as Darvan No. 1 available from R.T. Vanderbilt, 30 Winfield St., Norwalk, CT 06855.

described in 5.1. Then calculate the percent of coarse particles and skins.

12. Precision

12.1 Precision data are not available at this time. When available the appropriate precision statements will be added.

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Standard Practice for the Chemical Analysis of White Linseed Oil Paints¹

This standard is issued under the fixed designation D 215; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the chemical analysis of the usual white linseed oil paints. The methods included are listed in Table 1.

1.2 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 34 Practice for Chemical Analysis of White Pigments²
- D 50 Methods of Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 717 Test Methods for Analysis of Magnesium Silicate Pigment²
- D 1193 Specification for Reagent Water³
- D 1208 Test Methods for Common Properties of Certain Pigments²
- D 1301 Test Methods for Chemical Analysis of White Lead Pigments²
- D 1394 Test Methods for Chemical Analysis of White Titanium Pigments²
- D 1398 Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions²
- D 1469 Test Methods for Total Rosin Acids Content of Coating Vehicles²
- D 1542 Test Method for Qualitative Detection of Rosin in Varnishes^{4,5}
- D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids⁴
- D 2349 Method of Qualitative Determination of Nature of Thinner in Solvent-Reducible Paints³
- D 2350 Test Method for Antimony Oxide in White Pigment Separated from Solvent-Reducible Paints²
- D 2351 Test Method for Sulfide in White Pigment Separated from Solvent-Reducible Paints²

D 2352 Test Method for Sulfur Dioxide in White Pigment Separated from Solvent-Reducible Paints²

D 2369 Test Method for Volatile Content of Coatings⁵

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁵

D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints⁵

3. Purity of Reagents

3.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.2 Unless otherwise indicated, references to water shall be understood to mean Type II reagent grade water conforming to Specification D 1193.

4. Hazards

4.1 *Ammonium Hydroxide* causes severe burns and may be fatal if swallowed. Read the appropriate Material Safety Data Sheets (MSDS) before using.

4.2 *Hydrochloric and Sulfuric Acids* cause severe burns and may be fatal if swallowed. Read the appropriate MSDS before using.

4.3 *Acetic Acid* causes severe burns and may be fatal if swallowed. Read the appropriate MSDS before using.

4.4 *Nitric Acid* causes burns and may be fatal if swallowed. Vapor is extremely hazardous and may cause nitrogen oxide poisoning. Read the appropriate MSDS before using.

4.5 *Toluene* is flammable. Vapors are harmful. Use with adequate ventilation. Read the appropriate MSDS before using.

4.6 *Hydrogen Sulfide* is both an irritant and an asphyxiant. Read the appropriate MSDS before using.

4.7 *Ammonium Sulfide* evolves hydrogen sulfide on contact with acid or acid fumes. See 4.6. Read the appropriate MSDS before using.

4.8 *Barium Chloride*—Soluble barium salts are poisonous when taken by mouth. Read the appropriate MSDS before using.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

Current edition approved May 15, 1991. Published July 1991. Originally published as D 215 - 25 T. Last previous edition D 215 - 73 (1979)¹.

² *Annual Book of ASTM Standards*, Vol 06.02.

³ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.03.

⁵ *Annual Book of ASTM Standards*, Vol 06.01.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., and the "United States Pharmacopeia."

TABLE 1 List of Test Methods

Test Method	Section	ASTM Method
Preparation of Sample	4	...
Water	5	D 1208
Volatile Thinner	6	D 2369
Nature of Thinner	7	D 2349
Percentage of Pigment	8	D 2371
Percentage of Nonvolatile Vehicle	9	...
Separation of Vehicle	10	D 2372
Unsaponifiable Matter	11	D 1397
Fatty Acids	12	D 1398
Iodine Number of Fatty Acids	13	D 1959
Resin	14	D 1542
Qualitative Analysis, Single, Mixed, or Composite Pigments	15	...
Quantitative Analysis, Single Pigment	16	D 34
Quantitative Analysis, Mixed or Composite Pigments:		
Moisture and Other Volatile Matter	17	D 280
Loss on Ignition	18	D 1208
Insoluble Matter	19	...
Total Lead (Antimony)	20	...
Antimony Oxide	21	D 2350
Soluble Barium	22	...
Aluminum Oxide	23	...
Total Zinc	24	...
Soluble Calcium	25	...
Soluble Magnesium	26	...
Carbon Dioxide	27	D 1301
Total Soluble Sulfur Compounds	28	D 34
Soluble Sulfate	29	D 50
Sulfide Sulfur	30	D 2351
Sulfur Dioxide	31	D 2352
Matter Soluble in Water	32	D 1208

5. Preparation of Sample

5.1 On receipt of a sample, make a record of the label noting especially the brand, the name of the manufacturer, and any statement as to the composition of the paint and the net contents. Weigh the unbroken package, open, note odor and condition of the contents, pour into a clean container, and mix thoroughly by pouring from one container to the other, finally leaving the well-mixed sample in the second container which shall be tightly closed. The well-mixed sample shall be used at once for the analysis. The original can and cover may be cleaned with a suitable solvent, wiped dry, and then weighed. This weight subtracted from the original weight will give the net weight of the contents. If desired, the specific gravity of the paint may be determined, the weight per gallon calculated, and the volume of paint and the capacity of the container may be measured.

ANALYSIS OF PAINT

6. Water

6.1 Determine water in accordance with Test Methods D 1208.

7. Volatile Thinner

7.1 Determine the volatile matter in accordance with Test Method D 2369. Calculate the loss in weight as the percentage of water and volatile thinner. Subtract from this the percentage of water as determined in accordance with Section 6. Report the remainder as percent volatile thinner.

8. Nature of Thinner

8.1 Determine the nature of the thinner in accordance with Method D 2349.

9. Percentage of Pigment

9.1 Determine the percentage of pigment in accordance with Test Method D 2371. Preserve the pigment as prepared in a stoppered bottle for use in Sections 16 and 17.

10. Percentage of Nonvolatile Vehicle

10.1 Add together the percentages of water, volatile thinner, and pigment, and subtract the sum from 100. Report the remainder as nonvolatile vehicle.

TESTING NONVOLATILE VEHICLE

11. Separation of Vehicle

11.1 Separate the vehicle from the pigment in accordance with Method D 2372. Retain the vehicle so obtained for use in the unsaponifiable matter (see 12.1) and fatty acids (see 13.1) determinations.

12. Unsaponifiable Matter

12.1 Determine the unsaponifiable content of the vehicle in accordance with Test Method D 1398.

13. Fatty Acids

13.1 Determine the fatty acids in accordance with Method D 1398.

14. Iodine Number of Fatty Acids

14.1 Determine the iodine number of fatty acids (see 13.1) in accordance with Test Method D 1959.

NOTE 1—If appreciable amounts of rosin or of unsaponifiable matter are found to be absent in the vehicle of a paint, the iodine number of the fatty acids gives the best indication (though not proof) of the presence of linseed oil. An iodine number of less than 175 (Wijs) for the fatty acid is an indication that the nonvolatile vehicle was not pure linseed oil.

15. Rosin

15.1 Determine the presence of rosin in the fatty acids (see 13.1) in accordance with Test Method D 1542.

15.2 If desired, determine the amount of rosin quantitatively in accordance with Method D 1469.

ANALYSIS OF PIGMENT

Qualitative Analysis, Total Pigments—Single, Mixed, or Composite

16. Qualitative Analysis

16.1 Reagents:

16.1.1 *Acetic Acid.* (Precaution—See 4.3)

16.1.2 *Acid Ammonium Acetate Solution*—Mix 150 ml of acetic acid (8+2) 100 ml of water, and 95 ml of NH_4OH (sp gr 0.90).

16.1.3 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH). (Precaution—See 4.1)

16.1.4 *Ammonium Polysulfide*—Pass H_2S gas into 200 ml of NH_4OH (sp gr 0.90) in a bottle immersed in running water or in iced water until the gas is no longer absorbed; then add 200 mL of NH_4OH (sp gr 0.90) and dilute with water to 1 litre. Digest this solution with 25 g of flowers of sulfur for several hours and filter.

16.1.5 *Ammonium Sulfate* ($(\text{NH}_4)_2\text{SO}_4$).

16.1.6 *Barium Chloride* ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$). (Precaution—See 4.8)

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16.1.7 *Hydrochloric Acid (1+1)*—Mix equal volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) (Precaution—See 4.2) and water.

16.1.8 *Hydrogen Peroxide (H₂O₂), 3%*.

16.1.9 *Hydrogen Sulfide (H₂S)*. (Precaution—See 4.6)

16.1.10 *Potassium Dichromate (K₂Cr₂O₇)*.

16.1.11 *Potassium Ferrocyanide, Standard Solution*—Dissolve 22 g of pure potassium ferrocyanide (K₄Fe(CN)₆·3H₂O) in water and dilute to 1 L. To standardize, transfer about 0.2 g (accurately weighed) of pure metallic zinc or freshly ignited pure zinc oxide to a 400-mL beaker. Dissolve in 10 mL of HCl (sp gr 1.19) and 20 mL of water. Drop in a small piece of litmus paper, add NH₄OH until slightly alkaline, add HCl until just acid, and then 3 mL of HCl (sp gr 1.19). Dilute to about 250 mL with hot water and heat nearly to boiling. Run in the K₄Fe(CN)₆ solution slowly from a buret, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator shows a brown tinge after standing 1 min. A blank should be run with the same amounts of reagents and water as in the standardization. The amount of K₄Fe(CN)₆ solution required for the blank should be subtracted from the amounts used in standardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated.

16.1.12 *Potassium Iodide (KI)*.

16.1.13 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H₂SO₄). (Precaution—See 4.2)

16.1.14 *Sulfuric Acid (1+19)*—Carefully mix 1 volume of concentrated H₂SO₄ (sp gr 1.84) (Precaution—See 4.2) with 19 volumes of water.

16.2 *Procedure*:

16.2.1 The following qualitative analysis should be made and the quantitative scheme modified as required. Add acetic acid slowly to the pigment until all carbonate is decomposed (noting whether any H₂S is evolved), then add a large excess of acid ammonium acetate solution. Boil, filter, and test the filtrate for metals other than lead and zinc (especially calcium and barium). The absence of calcium in this filtrate indicates that the extending pigments contain no calcium carbonate (CaCO₃) or calcium sulfate (CaSO₄); the absence of barium indicates that the extending pigments contain no barium carbonate (BaCO₃).

NOTE 2—If the original sample contained BaCO₃, and lead sulfate (PbSO₄), CaSO₄, or other soluble sulfate, the soluble barium will form with the soluble sulfate a precipitate of BaSO₄ which will be determined as "insoluble matter." If the sample contained strontium sulfate (SrSO₄) or strontium carbonate (SrCO₃), some SrSO₄ may be counted as BaSO₄, some strontium will count as soluble barium, and some may be counted as calcium oxide (CaO). Strontium is not separated, as it probably will not be encountered, or will be present as an impurity in the barium and calcium compounds.

16.2.2 Wash the matter insoluble in acid ammonium acetate solution with another portion of this solution, and finally with hot water. This insoluble matter shall be dried, ignited, and tested for siliceous matter, BaSO₄, and titanium compounds. To test for the latter, place a small amount of the insoluble matter, or of the original sample (about 0.5 g) in a 250-mL resistant glass beaker; add 20 mL of concentrated H₂SO₄ (sp gr 1.84) and 7 to 8 g of (NH₄)₂SO₄. Mix well, and boil for a few minutes. A residue denotes the

presence of silica or siliceous matter. Cool the solution, dilute with 100 mL of water, heat to boiling, settle, filter, and wash with hot H₂SO₄ (1+19) until free from titanium. The residue may be tested for lead, barium, and silica.

16.2.3 Add H₂O₂ to a small portion of the filtrate; a clear yellow-orange color indicates the presence of titanium. Boil another portion of the filtrate with metallic tin or zinc; a pale blue to violet coloration indicates titanium.

16.2.4 Treat another portion (about 1 g) of the pigment with 20 mL of HCl (1+1) and note whether any H₂S is evolved; boil the solution for about 5 min, add about 25 mL of hot water, filter, and wash with hot water. Render a small portion of the filtrate alkaline with NH₄OH, acidify with HCl, and add a little BaCl₂ solution; a white precipitate (BaSO₄) indicates the presence of a soluble sulfate. To another portion of the filtrate add a little H₂SO₄; a white precipitate indicates the presence of lead, soluble barium, or both (some CaSO₄ may also separate). Filter, wash to remove free acid, and treat the precipitate with a few drops of KI solution; the formation of yellow lead iodide (PbI₂) indicates the presence of lead. The white precipitate may also be treated with H₂S water; the formation of black lead sulfide (PbS) indicates the presence of lead.

16.2.5 To another portion of the original filtrate (see 16.2.1) add NH₄OH until alkaline, render slightly acid with acetic acid, heat to boiling, and add a little K₂Cr₂O₇ solution; a yellow or orange-yellow precipitate indicates the presence of lead, soluble barium, or both. To another portion of the original filtrate add a few drops of K₄Fe(CN)₆ solution; a white precipitate with a bluish tinge indicates the presence of zinc. Pass into the remaining portion of the original filtrate a current of H₂S for 5 to 10 min, add an equal volume of water, and pass H₂S into the solution for about 5 min. Filter and wash with H₂S water. Digest the precipitate with ammonium polysulfide, filter, acidify the filtrate with HCl, and warm; the presence of antimony is indicated by the separation of an orange-colored precipitate. The filtrate from the H₂S precipitate may be tested for barium, calcium, and magnesium in the usual manner.

Quantitative Analysis, Single Pigment

17. Quantitative Analysis

17.1 If the sample is a single pigment, proceed in accordance with Practice D 34, for the particular pigment to be tested.

Quantitative Analysis, Mixed or Composite Pigments

18. Moisture and Other Volatile Matter

NOTE 3—On an extracted and dried pigment, this determination is of little value. If the original paint contained gypsum, a part of the combined water of the latter will be driven off in the drying of the extracted pigment and in the "moisture" determination.

18.1 Determine the moisture and other volatile matter in accordance with Test Methods D 280.

19. Loss on Ignition

NOTE 4—This determination may serve as a rough or approximate check in many cases on the carbon dioxide, water, etc.

19.1 Determine loss on ignition in accordance with Test Methods D 1208.

20. Insoluble Matter

20.1 Reagents:

20.1.1 Alcohol.

20.1.2 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). (Precaution—See 4.2)

20.1.3 *Hydrochloric Acid* (1+1)—Mix equal volumes of concentrated HCl (sp gr 1.19) (Precaution—See 4.2) and water.

20.1.4 *Sodium Carbonate* (Na_2CO_3), anhydrous.

20.1.5 *Sodium Carbonate* (10 g/L)—Mix 10 g of Na_2CO_3 with water and dilute to 1 L.

20.1.6 *Sulfuric Acid* (1+4)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) (Precaution—See 4.2) with 4 volumes of water.

20.2 Procedure:

20.2.1 Moisten 1 g of the pigment with a few drops of alcohol, cover, add 40 ml of HCl (1+1) and boil gently for 5 to 10 min. Wash the cover, evaporate to dryness, and heat at about 150°C for 30 min to 1 h to dehydrate the residue. Moisten the residue with 4 ml of concentrated HCl (sp gr 1.19), allow to stand a few minutes, dilute with 100 ml of hot water, boil a few minutes, filter hot through paper, and wash with hot water (until washings give no test for lead and chlorine).

20.2.2 Ignite the paper and residue in a platinum or porcelain crucible, cool, and weigh the total insoluble matter (Note 2). (The insoluble matter may be filtered off on a Gooch crucible, washed with hot water, dried at 105°C, cooled, and weighed; it shall then be ignited, cooled, and weighed, when it is desired to get the loss on ignition (combined water, organic matter, etc.) of the same, or if the insoluble matter is not to be further examined.) If the sample contains titanium pigment, practically all of the titanium dioxide (TiO_2) will be found in the insoluble matter along with barium sulfate (BaSO_4) and siliceous matter. Should an examination of the insoluble matter be necessary, it is advisable to remove the TiO_2 before proceeding further. The TiO_2 may be removed (or determined on a separate portion) in accordance with Test Methods D 1394.

20.2.3 After removing the TiO_2 , the residue containing siliceous matter and BaSO_4 may be ignited to remove the filter. To determine BaSO_4 , mix the ignited insoluble matter with about ten times its weight of anhydrous Na_2CO_3 (grinding the mixture in an agate mortar if necessary) and fuse in a covered platinum crucible, heating about 1 h. Let cool, place the crucible and the cover in a 200-mL glazed porcelain casserole (Note 5), add about 100 mL of water, and heat until the mass is disintegrated. Filter on paper into a 300-mL glazed porcelain casserole (leaving the crucible and the cover in the original casserole) and wash the casserole and filter thoroughly with a hot solution of Na_2CO_3 (10 g/L). Place the casserole containing the crucible and cover under the funnel, pierce the filter with a glass rod, and wash the residue into the original casserole by means of a jet of hot water. Wash the paper with hot HCl (1+1) and then with hot water. Remove the crucible and the cover. Evaporate the HCl solution to dryness, and heat at about 150°C for 30 min to 1 h. Moisten the residue with about 10 mL of concentrated HCl (sp gr 1.19), dilute with 100 mL of hot water, boil a few minutes, filter hot through paper, and wash thoroughly with hot water. Dilute the filtrate to a volume of 300 mL,

bring to boiling, and add, dropwise, 5 mL of H_2SO_4 (1+4). Allow to stand in a warm place for 1 h or so, filter on a weighed Gooch crucible, wash with hot water, ignite, cool, and weigh as BaSO_4 . Subtract the sum of the percentages of BaSO_4 and TiO_2 from the percentage of total insoluble matter and report the result as the percentage of insoluble siliceous matter (Note 6).

NOTE 5—A casserole is preferable to a beaker, as silica is dissolved from glass when in long contact with a strong sodium carbonate solution.

NOTE 6—Any soluble aluminum oxide (Al_2O_3) (or iron oxide (Fe_2O_3)) and in most cases magnesium oxide (MgO), and sometimes some calcium oxide (CaO), come from the siliceous pigment used. Magnesium oxide generally denotes the presence of asbestos.

20.2.4 To determine silica, acidify the filtrate from the barium carbonate (BaCO_3) filtration (20.2.3) with HCl, boil to expel the CO_2 , evaporate to dryness, bake to dehydrate the silica, moisten with HCl, dilute with 100 mL of hot water, and boil and filter through the same paper as was used to recover silica from the BaCO_3 portion. Wash thoroughly with hot water and proceed in accordance with Test Methods D 717.

20.2.5 If it is desired to look for magnesium, combine the filtrate obtained in accordance with 20.2.4 with the filtrate from the final BaSO_4 separation (20.2.3) and test for Al_2O_3 and MgO in the usual way. To recover MgO that may have dissolved in the procedure for the elimination of the TiO_2 , make the filtrate containing the TiO_2 just alkaline with NH_4OH , bring to boiling, filter, and wash. The filtrate may be tested for MgO . Any Al_2O_3 present will be precipitated along with the TiO_2 . To recover this, ignite and weigh as TiO_2 and Al_2O_3 . Deduct for TiO_2 present in the sample; the difference is Al_2O_3 .

21. Total Lead

21.1 Reagents:

21.1.1 *Acetic Acid*: Precaution—See 4.3.

21.1.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH). Precaution—See 4.1.

21.1.3 *Ammonium Polysulfide*—See 16.1.4.

21.1.4 *Ammonium Sulfide* ($(\text{NH}_4)_2\text{S}$). Precaution—See 4.7.

21.1.5 *Ether*.

21.1.6 *Ethyl Alcohol* (95%).

21.1.7 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl) Precaution—See 4.2.

21.1.8 *Hydrogen Sulfide* (H_2S). Precaution—See 4.6.

21.1.9 "*Lead Acid*"—Mix 300 ml of concentrated H_2SO_4 (sp gr 1.84) and 1800 ml of water. Dissolve 1 g of lead acetate in 300 mL of water and add this to the hot solution while stirring. Let stand at least 24 h and siphon through a thick asbestos filter.

21.1.10 *Nitric Acid* (1+1)—Mix equal volumes of concentrated nitric acid (HNO_3 , sp gr 1.19) (Precaution—See 4.4) and water.

21.1.11 *Nitric Acid* (1+3)—Mix 1 volume of concentrated HNO_3 (sp gr 1.19) (Precaution—See 4.4) with 3 volumes of water.

21.1.12 *Potassium Dichromate Solution* (100 g/L)—Dissolve 100 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in water and dilute to 1 L.

21.1.13 *Sodium Sulfide Solution (20 to 30 g/L)*—Dissolve 20 to 30 g of sodium sulfide (Na_2S) in water and dilute to 1 L.

21.1.14 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4). **Precaution**—See 4.2.

21.1.15 *Sulfuric Acid (1+1)*—Carefully mix 1 volume of concentrated H_2SO_4 (sp gr 1.84) (Precaution—See 4.2) with 1 volume of water.

21.2 Procedure:

21.2.1 Unite the filtrate and washings (total volume 150 to 200 mL) from the total insoluble matter (see Section 20), pass H_2S into the solution until it is saturated, add an equal volume of water, and again saturate with H_2S . Filter, wash with water containing a little H_2S , dissolve in hot HNO_3 (1+3), washing the paper with hot water. Add 10 to 20 mL of H_2SO_4 (1+1), evaporate until copious fumes of H_2SO_4 are evolved. Cool, add about 75 mL of water and then about 75 mL of ethyl alcohol (95%). Stir, let settle, filter on a Gooch crucible, wash with diluted alcohol, and dry in an oven at 105 to 110°C; or, ignite gently in a radiator⁷ or muffle, cool, and weigh as lead sulfate (PbSO_4). Calculate to lead oxide (PbO).

NOTE 7—It is not possible to determine the amount of basic lead carbonate and lead sulfate when carbonates or soluble sulfates of other metals, such as calcium, are present. Also, neither basic lead carbonate nor basic lead sulfate are definite compounds.

21.2.2 If the pigment contains antimony, filter and wash the sulfide precipitate as described in 21.2.1. Wash the precipitate with a fine jet of water from the paper into a porcelain dish or casserole, add 25 mL of ammonium polysulfide, cover the vessel, and warm the mixture at 40 to 60°C for 10 to 15 min while stirring frequently. Wash the cover, filter through the same paper, and wash with Na_2S (20 to 30 g/L) or $(\text{NH}_4)_2\text{S}$ solution. Discard the filtrate. Dissolve the residue in hot HNO_3 (1+3), and determine the lead as lead sulfate (PbSO_4) as described in 20.2.1; or, the original sulfide precipitate may be discarded and the lead determined on a separate portion of the pigment as follows: to 1 g of the sample in a covered beaker, add 40 mL of HCl (1+1) and boil gently for 5 to 10 min. Wash off the cover and evaporate to dryness. To the residue add sufficient HCl (sp gr 1.19) to dissolve the PbSO_4 (with pigments containing considerable amounts of PbSO_4 , it may be necessary to add 15 to 20 mL of HCl (sp gr 1.19)), add about 50 mL of hot water, boil a few minutes, filter hot through paper, and wash with hot water until the washings give no test for lead. (If the sample contains no insoluble matter, omit the filtration.)

21.2.3 To the filtrate add 20 mL of H_2SO_4 (sp gr 1.84) and evaporate until dense white fumes of sulfur trioxide (SO_3) are copiously evolved. Allow to cool, but not below 60°C, and then add slowly 50 mL of water while agitating the solution. Heat to boiling for several minutes in order to ensure complete solution of antimony sulfate. Allow the PbSO_4 to settle out until the supernatant liquid is clear, not letting the temperature fall below 60°C. If the liquid does not clear quickly it must be heated longer. When clear, pour the solution through a weighed porcelain Gooch crucible with asbestos mat, decanting the solution as completely as pos-

sible without allowing more than a very small amount of PbSO_4 to go over into the crucible. Add 10 mL more of concentrated H_2SO_4 (sp gr 1.84) to the PbSO_4 in the original beaker and boil for several minutes. Cool, add slowly 30 mL of water, and again heat to boiling for a few minutes. Allow the solution to cool to about 60°C and completely transfer the PbSO_4 to the Gooch crucible. Wash with "lead acid" (see 21.1.9) to remove soluble sulfates and finally wash free of acid with diluted alcohol (equal parts of ethyl alcohol or denatured alcohol and water). Dry in an oven at 105 to 110°C, or ignite gently in a radiator or muffle. Calculate to PbO , or determine as lead chromate (PbCrO_4) as described in 21.2.6.

21.2.4 If soluble compounds of barium or calcium are present, barium sulfate (BaSO_4) and calcium sulfate (CaSO_4) will be included with the PbSO_4 . If soluble silica (SiO_2) is present, it will also be included with the PbSO_4 . In such cases, the PbSO_4 precipitate after washing with diluted alcohol may be dissolved in acid ammonium acetate and the lead determined as PbCrO_4 as described in 21.2.6. For ordinary work, the amount of BaSO_4 dissolved by the acetate treatment may be disregarded.

21.2.5 If the pigment contains no soluble antimony, barium, or calcium compounds, the lead may be determined directly on the original pigment as follows: to 1 g of the sample in a covered beaker, add 25 mL of HNO_3 (1+1) and boil gently a few minutes. Wash off the cover, evaporate to dryness on a steam bath, moisten with HNO_3 , add hot water, and heat a few minutes. Filter and wash with hot water until washings are lead-free. Add 10 to 20 mL of H_2SO_4 (1+1) to the clear solution, evaporate, and determine lead as PbSO_4 as described in 21.2.1.

21.2.6 In the absence of soluble compounds of antimony, iron, aluminum, and barium, the following procedure may be used: treat 1 g of the original pigment with 25 mL of HNO_3 (1+1) and proceed as described in 20.2.1. To the clear solution, diluted to 200 mL, add NH_4OH in slight excess, acidify with acetic acid, and add 4 to 6 mL more of acetic acid. Heat to boiling and add 10 to 15 mL of a solution of $\text{K}_2\text{Cr}_2\text{O}_7$ (100 g/L). Heat until the yellow precipitate assumes an orange color, let settle, and filter on a weighed Gooch crucible. Wash by decantation until the washings are colorless, finally transferring all of the precipitate. Wash with ethyl alcohol (95 %) and then with ether. Dry to constant weight at 110°C, cool, and weigh as PbCrO_4 . Calculate to PbO .

22. Antimony Oxide

22.1 Determine antimony oxide in accordance with Test Method D 2350.

23. Soluble Barium

23.1 Boil the combined filtrate and washings, reduced to volume by evaporation if need be, from the lead sulfate (PbS) precipitate (see 21.2) to expel hydrogen sulfide (H_2S). Add a slight excess of H_2SO_4 (1+4) over the amount required to precipitate the barium, heat to boiling, let stand on a steam bath about 1 h, filter on a weighed Gooch crucible, wash with hot water, dry, ignite, cool, and weigh as barium sulfate (BaSO_4) (Notes 2 and 8). Calculate to barium oxide (BaO).

NOTE 8—The precipitate will include any BaSO_4 that may have been dissolved as such. The weighed precipitate should be tested for CaSO_4 .

⁷ U. S. Geological Survey, *Bulletin* 700 (1919), p. 33.

and if present, it should be removed by treating with hot HCl (1+3), filtering, washing, igniting, and again weighing.

24. Aluminum Oxide (Fe_2O_3 , TiO_2 , P_2O_5)

24.1 Reagents:

24.1.1 *Ammonium Chloride Solution* (25 g/L)—Dissolve at least 25 g of ammonium chloride (NH_4Cl) in water and dilute to 1 L.

24.1.2 *Ammonium Hydroxide* (1+5)—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH , sp gr 0.90) (Precaution—See 4.1) with 1 volume of water.

24.1.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). Precaution—See 4.2.

24.1.4 *Methyl Red Indicator, Alcoholic Solution* (2 g/L)—Dissolve 0.2 g of methyl red in alcohol and dilute to 100 mL.

24.1.5 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3). Precaution—See 4.4.

24.2 Procedure:

24.2.1 Boil the filtrate from the lead sulfide (PbS) precipitate (see 21.2) to expel hydrogen sulfide (H_2S), add a few drops of HNO_3 , and continue the boiling a few minutes to oxidize any iron that may be present. In case soluble barium was present, use the filtrate from 23.1. To the solution containing at least 25 g of NH_4Cl /L of solution, or an equivalent amount of HCl, add a few drops of methyl red indicator, alcoholic solution and heat just to boiling. Carefully add NH_4OH (1+5) dropwise until the color of the solution changes to a distinct yellow. Boil the solution for 1 to 2 min and filter at once. Wash the precipitate thoroughly with hot NH_4Cl solution (Note 9). Ignite the precipitate, cool, and weigh as aluminum oxide (Al_2O_3) (Note 10).

NOTE 9—For very accurate work, or when the precipitate is large, the precipitate should be dissolved in HCl (1+1) and the precipitation repeated.

NOTE 10—This precipitate may also contain ferric oxide (Fe_2O_3), titanium dioxide (TiO_2), and phosphorus pentoxide (P_2O_5).

25. Total Zinc

25.1 Reagents:

25.1.1 *Acetic Acid* (1+49)—Mix 1 volume of concentrated acetic acid (sp gr 1.05) (Precaution—see 4.3) with 49 volumes of water.

25.1.2 *Ammonium Acetate*.

25.1.3 *Ammonium Chloride* (NH_4Cl).

25.1.4 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH). Precaution—See 4.1.

25.1.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). Precaution—See 4.2.

25.1.6 *Hydrochloric Acid* (1+2)—Mix 1 volume of concentrated HCl (sp gr 1.19) (Precaution—See 4.2) with 2 volumes of water.

25.1.7 *Hydrogen Sulfide* (H_2S). Precaution—See 4.6.

25.1.8 *Potassium Ferrocyanide, Standard Solution*—See 16.1.11.

25.1.9 *Uranyl Indicator*—Dissolve 5 g of uranyl nitrate in water and dilute to 100 mL, or dissolve 5 g of uranyl acetate in water made slightly acid with acetic acid and dilute to 100 mL.

25.2 Procedure:

25.2.1 To the combined filtrate and washings from the alumina determination (see 24.2.1), add sufficient NH_4Cl to

give 5 g/100 mL of solution, and then add 1 g of ammonium acetate.⁸ Make slightly acid with acetic acid and pass in a current of H_2S to saturation. Allow the precipitate to settle completely, filter on paper, and wash with a solution of acetic acid (1+49) saturated with H_2S . Transfer the precipitate and filter to the vessel in which the precipitation was effected, add 30 mL of water and 10 mL of concentrated HCl (sp gr 1.19), heat until all zinc is in solution, add 200 mL of water and a small piece of litmus paper; add NH_4OH (sp gr 0.90) until slightly alkaline, make just acid with HCl, then add 3 mL of concentrated HCl (sp gr 1.19), heat nearly to boiling, and titrate with standard $\text{K}_4\text{Fe}(\text{CN})_6$ solution as described in 16.1.11 using uranyl indicator solution.

25.2.2 Zinc may be determined directly on the original sample as follows (Note 11): weigh accurately about 1 g (or an amount that will give a buret reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 400-mL beaker, add 30 mL of HCl (1+2), boil a few minutes, add 200 mL of water, and a small piece of litmus paper; add concentrated NH_4OH until slightly alkaline, render just acid with HCl, then add 3 mL of HCl (sp gr 1.19), heat nearly to boiling, and titrate with standard $\text{K}_4\text{Fe}(\text{CN})_6$ solution as described in 16.1.11 using uranyl indicator solution.

NOTE 11—If the sample contains antimony, it should be precipitated by H_2S in the hot acid solution, filtered off, washed, and the filtrate neutralized, etc., for zinc. The H_2S precipitate may also contain lead sulfide (PbS). If no sulfide separation is made, any cadmium present will be counted as zinc.

25.2.3 When iron is present, total zinc may be determined directly on the original sample as follows (Note 11). Weigh accurately about 1 g (or an amount that will give a buret reading approximately equal to that obtained in the standardization) of the pigment, transfer to a 250-mL beaker, moisten with alcohol, add 30 mL of HCl (1+2), boil for 2 or 3 min, and add about 100 mL of water. Add about 2 g of NH_4Cl make slightly alkaline with NH_4OH , heat to boiling, let settle on a steam bath, filter into a 400-mL beaker, and wash the residue once with hot water. Remove the 400-mL beaker and pour HCl (1+2) on the residue, catching the filtrate therefrom in the 250-mL beaker; wash a few times with hot water. Add to this filtrate 1 g of NH_4Cl and make slightly alkaline with NH_4OH , boil, let settle, filter on paper used for first filtration, and wash thoroughly with hot water, catching the filtrate and washings in the 400-mL beaker containing the first filtrate. Add a small piece of litmus paper, acidify with HCl, add 3 mL of HCl (sp gr 1.19), heat nearly to boiling, and titrate with standard $\text{K}_4\text{Fe}(\text{CN})_6$ solution as described in 16.1.11.

25.2.4 With pigments containing zinc oxide (ZnO) and zinc sulfide (ZnS), the ZnO may be determined as follows: weigh accurately 1 g of the pigment, transfer to a 250-mL beaker, moisten with alcohol, add about 100 mL of acetic acid (1+49), stir vigorously but do not heat, cover and let stand for 18 h, stirring once every 5 min for the first 30 min. Filter, wash with acetic acid (1+49) followed by water until the washings give no test for zinc with $\text{K}_4\text{Fe}(\text{CN})_6$ solution.

⁸ Gooch, F. A., *Representative Procedures in Quantitative Chemical Analysis*, 1st Ed. (1916), p. 107.

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Dilute the clear filtrate to about 200 mL with water, add 30 mL of HCl (1+2), and a small piece of litmus paper; add NH_4OH (sp gr 0.90) until slightly alkaline, render just acid with HCl, then add 3 mL of concentrated HCl (sp gr 1.19), heat nearly to boiling, and titrate with $\text{K}_4\text{Fe}(\text{CN})_6$ solution as described in 25.2.4. Calculate this result to zinc, subtract from total zinc, and calculate the difference to zinc sulfide (ZnS). (Any zinc carbonate (ZnCO_3) or zinc sulfate (ZnSO_4) is included in the ZnO.)

26. Soluble Calcium

26.1 Reagents:

26.1.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH). **Precaution**—See 4.1.

26.1.2 *Ammonium Oxalate, Saturated Solution* (NH_4)₂· C_2O_4 .

26.1.3 *Potassium Permanganate, Standard Solution* (0.1 N)—Dissolve 3.2 g of pure potassium permanganate (KMnO_4) in water and dilute to 1 L. Let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize against NIST's standard sample 40c of sodium oxalate as follows: in a 400-mL beaker, dissolve 0.25 to 0.30 g (accurately weighed) of sodium oxalate in 250 mL of hot water (80 to 90°C) and add 15 mL of sulfuric acid (H_2SO_4 , 1+1). Titrate at once with the KMnO_4 solution, stirring the liquid vigorously and continuously. The KMnO_4 must not be added more rapidly than 10 to 15 mL/min, and the last 0.5 to 1 mL must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The temperature of the solution should not be below 60°C by the time the end point is reached. (Too rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.833 gives its iron equivalent. The KMnO_4 solution should be kept in a glass-stoppered bottle painted black to keep out light.

26.1.4 *Sulfuric Acid* (1+4)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) (**Precaution**—See 4.2) with 4 volumes of water.

26.2 Procedure:

26.2.1 Heat to boiling the united filtrate and washings, reduced in volume if need be, from the ZnS determination (see 25.2.2), add 1 mL of NH_4OH and an excess of a hot saturated (NH_4)₂· C_2O_4 solution. Continue the boiling until the precipitate becomes granular; let stand about 1 h, filter, and wash with hot water. Ignite, cool, and weigh as calcium oxide (CaO) (Notes 2, 12, and 13). Alternatively, place the beaker in which the precipitation was made under the funnel, pierce the apex of the filter with a stirring rod, and wash the precipitate into the beaker with hot water. Pour warm H_2SO_4 (1+4) through the paper and wash a few times. Add about 30 mL of H_2SO_4 (1+4), dilute to about 250 mL, heat to 90°C, and titrate at once with 0.1 N KMnO_4 solution (the temperature of the solution should not be below 60°C when the end point is reached, see 25.1.3). Calculate to CaO (Notes 2, 12, and 13). (The iron equivalent of $\text{KMnO}_4 \times 0.502 = \text{CaO}$ value.)

NOTE 12—Care must be exercised in this washing, as 1 L of boiling water will dissolve over 0.01 g of calcium oxalate (CaC_2O_4).

NOTE 13—For more accurate work, the CaC_2O_4 precipitate should be ignited, cooled, cautiously moistened with water, redissolved in HCl, and the solution diluted to 100 mL. Add NH_4OH in slight excess, boil the liquid, and filter and wash if a precipitate appears. Reprecipitate the calcium with NH_4OH and (NH_4)₂· C_2O_4 , as described in 25.2.1, filter, wash, ignite, cool, and weigh; or, titrate as described.

27. Soluble Magnesium

27.1 Reagents:

27.1.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH). **Precaution**—See 4.1.

27.1.2 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). **Precaution**—See 4.2.

27.1.3 *Hydrochloric Acid* (1+3)—Mix 1 volume of concentrated HCl (sp gr 1.19) (**Precaution**—See 4.2) with 3 volumes of water.

27.1.4 *Sodium Ammonium Phosphate, Saturated Solution* ($\text{NaNH}_4\text{HPO}_4$).

27.2 Procedure:

27.2.1 Acidify the filtrate from the calcium determination (see 26.2.1) with HCl, add 10 mL of a saturated solution of $\text{Na}(\text{NH}_4)\text{HPO}_4$ and NH_4OH dropwise, with constant stirring. When the crystalline magnesium ammonium phosphate ($\text{Mg}(\text{NH}_4)\text{PO}_4$) has formed, add 5 mL excess of NH_4OH . Allow the solution to stand in a cool place for not less than 4 h, preferably overnight (Note 14). Filter and wash with water containing 2.5 % ammonia. Dissolve the precipitate in a small quantity of hot HCl (1+3), dilute the solution to about 100 mL with water, add 1 mL of a saturated solution of $\text{Na}(\text{NH}_4)\text{HPO}_4$ and NH_4OH dropwise, with constant stirring, until the precipitate is again formed, and then add 5 mL excess of NH_4OH . Let the precipitate stand in a cool place for not less than 2 h, filter on a Gooch crucible, wash with water containing 2.5 % of ammonia, ignite, cool, and weigh as magnesium pyrophosphate (MgP_2O_7) (Note 15). Calculate to magnesium oxide (MgO).

NOTE 14—The smaller the amount of magnesium present, the longer the precipitate must be allowed to settle.

NOTE 15—If the sample contained manganese, it will be caught in large part with the $\text{Mg}_2\text{P}_2\text{O}_7$. If desired, manganese may be determined by dissolving the $\text{Mg}_2\text{P}_2\text{O}_7$. If desired, manganese may be determined by dissolving the $\text{Mg}_2\text{P}_2\text{O}_7$ in nitric acid (**Precaution**—See 4.4) and applying the bismuthate method.

28. Carbon Dioxide

28.1 Determine carbon dioxide in accordance with Test Methods D 1301.

29. Total Soluble Sulfur Compounds (Note 2)

29.1 Determine total soluble sulfur in accordance with Methods D 34. This determination includes soluble sulfates, sulfur trioxide (SO_3) formed from sulfur dioxide, (SO_2), and the SO_3 that is formed from sulfide sulfur.

30. Soluble Sulfate (Note 2)

30.1 Determine soluble sulfates in accordance with Methods D 50.

31. Sulfide Sulfur

31.1 Determine sulfide sulfur in accordance with Test Method D 2351.

32. Sulfur Dioxide

32.1 Determine sulfur dioxide in accordance with Test Method D 2352.

33. Matter Soluble in Water

33.1 Determine matter soluble in water in accordance with Test Methods D 1208.

NOTE 16—The nature of the water-soluble matter may be determined by further examination, as the percentages of sulfur trioxide (SO_3) and calcium oxide (CaO) may be indicative.

NOTE 17—The water-soluble content of composite pigmentation, as determined in accordance with this method, is frequently higher than the sum of the water-soluble matter in the individual pigments. Possibly this is due to reaction in water between the individual pigments.

34. Calculation

34.1 The calculation of the component pigments of a mixed or combination pigment may be a somewhat difficult matter. Certain assumptions must be made, depending upon the complexity of the mixed pigment, as to the composition or formulas of component pigments and as to the manner in which the acidic and basic radicals are combined. Add any $\text{Al}_2\text{O}_3(\text{Fe}_2\text{O}_3)$ found in the soluble portion to the siliceous matter and report the sum as "insoluble siliceous matter" unless the soluble aluminum is high; in this case, an aluminate is probably present, and the Al_2O_3 should be reported as Al_2O_3 . If a small amount of soluble magnesium is found, it should also be added to the siliceous matter. If the soluble magnesium is high, the presence of MgCO_3 is indicated, and the MgO is calculated to MgCO_3 as described in 34.3. The insoluble siliceous matter reported should be based on the weight obtained on drying the total insoluble matter at 105°C if the combined water contained therein is to be considered.

34.2 Report TiO_2 as TiO_2 , ZnS as ZnS , and BaSO_4 as BaSO_4 . If CaCO_3 , CaSO_4 , BaCO_3 , and MgCO_3 are absent, calculate CO_2 to basic carbonate white lead ($\text{Pb}(\text{CO}_3)_2 \cdot \text{Pb}(\text{OH})_2$), and soluble SO_3 to PbSO_4 . Calculate any excess of lead to PbO , add it to the PbSO_4 , and report the sum as basic lead sulfate. Alternatively, multiply the sum of $\text{PbSO}_4 + \text{PbO}$ by 0.058 to obtain the ZnO ; add this result to the $\text{PbSO}_4 + \text{PbO}$ and report as basic sulfate white lead. (The ZnO factor is based on the assumption that the average composition of commercial basic sulfate white lead is 78.5 % PbSO_4 , 16.0 % PbO , and 5.5 % ZnO .) Lead oxide (PbO) should not be reported except in the presence of PbSO_4 unless the entire analysis is reported in the elementary or oxide form.

34.3 If the sample contains CO_2 but not soluble SO_3 , calculate total lead to basic carbonate white lead ($\text{Pb}(\text{CO}_3)_2 \cdot \text{Pb}(\text{OH})_2$); calculate residual CO_2 to CaCO_3 , then to BaCO_3 and MgCO_3 , if soluble barium and magnesium should be present in sufficient amounts to indicate the presence of these carbonates. The CO_2 result will be an index of this. A small amount of residual CaO is probably from the siliceous matter and should be added to the insoluble siliceous matter.

34.4 A small amount of soluble barium may be from the CaCO_3 used or may be due to the solubility of BaSO_4 , if this compound is present in the original pigment. This barium may be calculated to BaSO_4 and added to the BaSO_4 found in the insoluble matter.

34.5 If the sample contains soluble SO_3 but no CO_2 , calculate CaO to CaSO_4 or $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$; residual SO_3 to PbSO_4 ; add residual PbO to PbSO_4 and report the sum as basic lead sulfate. Alternatively, multiply $\text{PbSO}_4 + \text{PbO}$ by 0.058 and add the result to the $\text{PbSO}_4 + \text{PbO}$, and report the total as basic sulfate white lead.

34.6 If the sample contains CaCO_3 (MgCO_3 , BaCO_3) and also basic sulfate white lead, or CaSO_4 and basic carbonate white lead, or a mixture of these, it is not possible to determine or calculate the amount of PbCO_3 or PbSO_4 with any degree of certainty (see Notes 2 and 3). The presence of appreciable amounts of CaO and SO_3 in the water-soluble matter indicates the probable presence of CaSO_4 in the original pigment. The following arbitrary calculations may be made: calculate water-soluble SO_3 to CaSO_4 or $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, subtract this SO_3 from total soluble SO_3 , and calculate the remainder to PbSO_4 ; calculate residual CaO to CaCO_3 , and then residual CO_2 to (PbCO_3)₂· $\text{Pb}(\text{OH})_2$. If there is an excess of CO_2 , calculate to MgCO_3 or BaCO_3 , if the amounts of soluble magnesium and barium indicate the probable presence of these carbonates. Add residual PbO to PbSO_4 and calculate as described in 34.5 to basic sulfate white lead.

34.7 Report total antimony as Sb_2O_3 .

34.8. Calculate sulfide sulfur to ZnS , subtract the zinc equivalent to the sulfur from the total zinc, then subtract the zinc required for the basic sulfate white lead, and report the remainder as ZnO .

34.9 Report directly the following: moisture and other volatile matter, loss on ignition, SO_2 and matter soluble in water.

35. Keywords

35.1 analysis of paint, white oil; analysis of pigment, white oil; lead analysis; white linseed oil paints, chemical analysis

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Standard Test Methods for Clear and Pigmented Lacquers¹

This standard is issued under the fixed designation D 333; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

[†] NOTE—Keywords were added editorially in April 1991.

1. Scope

1.1 These test methods cover procedures for testing lacquers and lacquer coatings. The test methods included are listed in Table 1.

NOTE 1—In accordance with Definitions D 16, a lacquer is defined as a coating composition that is based on synthetic thermoplastic film-forming material dissolved in organic solvent(s) and that dries primarily by solvent evaporation. Typical lacquers include those based on nitrocellulose, other cellulose derivatives, vinyl resins, acrylic resins, etc.

NOTE 2—Lacquers may be applied under such diverse conditions, to so many different surfaces, and their dried films may be subjected to so many kinds of wear and exposure that it is not possible to assure desired performance from a single selection of test methods and numerical results therefrom. Those skilled in lacquer technology may find partial assurance of obtaining desired qualities in various types of lacquers through careful selection of the methods covered herein and intelligent interpretation of results therefrom.

NOTE 3—It is intended ultimately to remove all experimental procedures from Test Methods D 333 and to establish them as a guide to the selection of test methods for lacquer and perhaps to interpretation of results therefrom. Temporarily there remain in Test Methods D 333 a few tests that are too short or otherwise presently unsuitable for establishment under separate ASTM designations.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- B 117 Method of Salt Spray (Fog) Testing²
- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products³
- D 56 Test Method for Flash Point by Tag Closed Tester⁴
- D 88 Test Method for Saybolt Viscosity⁵
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester⁴

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.55 on Factor-Applied Coatings on Preformed Products.

Current edition approved June 26, 1987. Published August 1987. Originally published as D 333 - 31 T. Last previous edition D 333 - 81.

² Annual Book of ASTM Standards, Vols 03.02 and 06.01.

³ Annual Book of ASTM Standards, Vols 06.01, 06.02 and 06.03.

⁴ Annual Book of ASTM Standards, Vols 05.01 and 06.03.

⁵ Annual Book of ASTM Standards, Vol 04.04.

- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)⁶
- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings⁷
- D 523 Test Method for Specular Gloss⁷
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁷
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces⁷
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive⁷
- D 659 Method of Evaluating Degree of Chalking of Exterior Paints⁷
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints⁷
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁷
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints⁷
- D 714 Test Method for Evaluating Degree of Blistering of Paints⁷
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints⁷
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁷
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion⁷
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁷
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁷
- D 1014 Test Method for Conducting Exterior Exposure Tests of Paints on Steel⁷
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁷
- D 1200 Test Method for Viscosity by Ford Viscosity Cup⁷
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)⁸
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁷

⁶ Annual Book of ASTM Standards, Vol 05.01.

⁷ Annual Book of ASTM Standards, Vol 06.01.

⁸ Annual Book of ASTM Standards, Vols 06.01 and 06.03.

- D 1211 Test Method for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood⁷
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁷
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base⁷
- D 1474 Test Methods for Indentation Hardness of Organic Coatings⁷
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁷
- D 1535 Test Method for Specifying Color by the Munsell System⁹
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)³
- D 1644 Test Methods for Nonvolatile Content of Varnishes⁷
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁷
- D 1733 Method of Preparation of Aluminum Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products¹⁰
- D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus⁷
- D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus¹¹
- D 2090 Test Method for Clarity and Cleanness of Paint and Ink Liquids¹²
- D 2091 Test Method for Print Resistance of Lacquers⁷
- D 2199 Method for Measurement of Plasticizer Migration from Vinyl Fabrics to Lacquers⁷
- D 2204 Test Method for Perspiration Resistance of Organic Coatings¹³
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁷
- D 2620 Test Method for Light Stability of Clear Coatings⁷
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁷
- D 3170 Test Method for Chipping Resistance of Coatings⁷
- D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus¹⁴
- E 308 Method for Computing the Colors of Objects by Using the CIE System⁹

3. Significance and Use

3.1 These test methods are intended to compile as well as provide screening tests in evaluating clear and pigmented lacquers as used in different coating operations.

3.2 Each coating system may contain from a simple one coat operation to a multicoat finishing system.

3.3 The substrates may be varied, ferrous and non ferrous, plastic or wood which can affect the performance of a given coating system.

3.4 Substrate cleaning, chemically or physically, is an

TABLE 1 List of Test Methods

Test Method	Section	ASTM Method
General Requirements	5	...
Liquid Materials:		
Color of Clear Lacquers:		
Gardner Color Scale	10	D 1544
Platinum-Cobalt Scale	10	D 1209
Flash Point:		
Tag Closed Cup	12	D 56, D 93, D 3278
Homogeneity:		
Clarity and Cleanness	9	D 2090
Fineness of Pigment Grind	9	D 1210
Nonvolatile Matter	8	D 1644
Sample Preparation	6	...
Viscosity:		
Ford Cup (Efflux)	11	D 1200
Kinematic High Precision	11	D 445
Weight per Gallon (Density)	7	D 1475
Dried Films:		
Abrasion Resistance:		
Air Blast Abrasion Tester	20	D 658
Falling Sand Method	20	D 968
Chip Resistance:	30	D 3170
Color-Pigmented Coatings:		
Spectrophotometric Method	16	D 308
Munsell Color System	16	D 1535
Color Difference-Pigmented Coatings:		
Visual Method	17	D 1729
Instrumental Evaluation of Color Differences of Opaque Materials	17	D 2244
Elongation:		
Conical Mandrel	18	D 522
Cylindrical Mandrel	18	D 1737
Film Thickness:		
Nondestructive Magnetic Base	14	D 1186
Nondestructive Nonmagnetic Metallic Base	14	D 1400
Penetration Thickness Gage	14	D 1400
Dial Comparator	14	D 1005
Gloss:		
Specular Gloss	15	D 523
Hardness Indentation Method	19	D 1474
Hiding Power	28	D 2805
Household Chemical Resistance	22	D 1308
Light Stability	31	D 2620
Outdoor Exposure:		
Preparation of Coated Panels:		
Aluminum Alloy Panels	23	D 1733
Steel Panels	23	D 609
Test Procedures:		
Exposure Tests Using Steel Panels	23	D 1014
Evaluating Blistering	23	D 714
Evaluating Chalking	23	D 659
Evaluating Checking	23	D 660
Evaluating Cracking	23	D 661
Evaluating Rusting	23	D 610
Evaluating Erosion	23	D 662
Evaluating Flaking	23	D 772
Panel Preparation:		
Manual Spraying Method	13	...
Automatic Application	13	D 823
Plasticizer Migration	30	D 2199
Print Test	21	D 2091
Salt Fog	25	B 117
Temperature-Change Resistance	24	D 1211
Water Fog Testing	26	D 1735
Water Immersion Test	27	D 870

essential and critical aspect to the performance of the coating system.

3.5 Results from the various tests are not necessarily all useful in evaluating the performance of the different types of coating systems used on the many varied substrates.

⁹ Annual Book of ASTM Standards, Vol 14.02.

¹⁰ Discontinued; see 1979 Annual Book of ASTM Standards, Part 27.

¹¹ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

¹² Annual Book of ASTM Standards, Vols 06.02 and 06.03.

¹³ Discontinued; see 1976 Annual Book of ASTM Standards, Part 27.

¹⁴ Annual Book of ASTM Standards, Vol 06.03.

4. General Requirements

4.1 All tests shall be made in diffused light (not in direct sunlight), and at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, unless otherwise specified.

5. Preparation of Sample

5.1 Many clear lacquers and all pigmented lacquers contain suspended solids that have a tendency to settle to the bottom of the container. Stir any settled portion with a paddle or spatula and then shake vigorously for 10 min on a mechanical agitator. Since many lacquer solvents are extremely volatile, care should be taken during sampling and testing to avoid loss of significant amounts of volatile matter.

6. Weight Per Gallon

6.1 Determine the density as described in Test Method D 1475. This method is particularly adaptable for high-viscosity fluids or where a component is too volatile for a specific gravity balance determination. If a weight per gallon cup of 83.2-mL capacity is used, calculation of weight per gallon is simplified.

7. Nonvolatile Matter

7.1 Nonvolatile matter determination is an indication of the amount of permanent film-forming material contained in a lacquer. At ambient temperatures, drying of a lacquer film may involve gradual loss of slowly volatile solvents, hence solids determinations may differ from those resulting from a nonvolatile determination accelerated by a higher temperature.

7.2 Determine the nonvolatile content of lacquers as described in Test Methods D 1644. As an additional requirement, the specimen shall be reheated and reweighed until the weight is constant to within 1 mg. Test Method A of Test Methods D 1644 is preferred since Method B is potentially dangerous when used with lacquers.

8. Homogeneity

8.1 Good quality lacquers and their ingredients should be uniformly constituted and free from particles of foreign matter.

8.2 Determine the presence or absence of foreign matter in nonpigmented liquids for use in paints and lacquers or lacquers themselves as described in Test Method D 2090.

8.3 Determine the degree of dispersion (commonly referred to as "fineness of grind") of pigment, semiquantitatively, in pigmented coating systems in accordance with Test Method D 1210.

9. Color

9.1 The color of a clear lacquer is only a preliminary indication of the color of a dried film of lacquer. The initial color may bleach and another color may appear under certain conditions of exposure.

9.2 Determine the color of clear lacquers as described in Test Method D 1544. This method gives a comparison of the color of the sample with that of a color reference standard.

9.3 Determine the color of essentially water-white lacquers in accordance with Test Method D 1209. This method shall be used where the color-producing bodies in the lacquer have very nearly the same light-absorption characteristics as

those of the platinum-cobalt standards.

10. Viscosity

10.1 The viscosity of a lacquer is a property that can be used as a guide in determining the ease with which a given lacquer may be applied. For example, lacquers designed for spray application may be low in viscosity whereas they are high for doctor blade or roller application.

10.2 Determine the viscosity of clear and pigmented finishes designed for spray application as described in Test Method D 1200. This method is not recommended for lacquers with viscosities requiring more than 100 s efflux time.

10.3 For precise viscosity determinations in the range from 0.4 to 16 000 cSt proceed in accordance with Test Method D 445.

NOTE 4—Caution should be observed in that it is recognized that changes in viscosity may occur in lacquers upon aging.

11. Flash Point

11.1 The organic solvents used in lacquers have characteristic flash points. The flash point of a liquid is defined as the lowest temperature, corrected to 101.3 kPa (760 mmHg) of pressure, of the sample at which application of an ignition source causes the vapor of the sample to ignite under specified conditions of test.

11.2 Determine the flash point by Test Method D 56 or Test Methods D 93 for liquid storage regulations of Occupational Safety and Health Administration (OSHA) of U. S. Department of Labor and for classification of hazardous liquids for shipments under the regulations of U. S. Department of Transportation and bulk shipments by water.

11.2.1 Determine the flash point of lacquer or lacquer materials having a viscosity less than 9.5 cSt at 77°F (25°C) or 45 SUS at 100°F (37.8°C) (Method D 88) by Test Method D 56 and of lacquers having a viscosity of more than 9.5 cSt at 77°F (25°C) or 45 SUS at 100°F (37.8°C) by Method A of Test Methods D 93. Use Method B of Test Methods D 93 whenever there is a question that the heat transfer within a viscous lacquer is not sufficient to assure an accurate flash point. In addition, use Method B when testing pigmented lacquers or suspensions of solids and liquids which tend to skin under test conditions.

11.3 Test Methods D 3278, which give comparable results to Test Method D 56 and Test Methods D 93 while requiring a smaller specimen and less time to run, may be used as an alternative method.

12. Panel Preparation

12.1 In the evaluation of coated panels a uniform dry film thickness of lacquer is essential in order to eliminate any effect due to film thickness. Unless otherwise specified, the dry film thicknesses shall be as follows:

	Mils	Micrometres
Lacquer primer	0.3 to 0.6	8 to 15
Lacquer primer surfacer	1.0 to 2.0	25 to 50
Clear lacquer	1.0 to 1.2	25 to 31
Gloss pigmented lacquer	1.0 to 1.2	25 to 31
Lacquer putties	4.0 to 6.0	100 to 150

12.2 Panels may be prepared by either manual or automatic application of lacquers. First reduce lacquers with the

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specified thinner (if reduction is required) to the required dilution solids or viscosity. If no viscosity is specified, spray with standard-type guns at a viscosity of 17 to 25 s in a No. 4 Ford Cup at 25°C determined as described in Test Method D 1200.

12.3 Cold Spray—In manual application by cold spray, keep the air pressure constant and provide the air line with a moisture trap. Adjust the flow, gun distance from test panel, and spray pattern that the deposited film is kept smooth, level, and wet. Apply the number of coats or thickness, or both, specified in the product specification.

12.4 Hot Spray—The packaged material shall be applied with hot spray equipment approved by the Underwriter's Laboratory. When dilution is necessary, the material shall be reduced with the specified thinner to the viscosity required for hot spray application. The conditions of operation with respect to temperature of the heating unit, type of spray gun, atomization pressure and pressure on feed tank type, adjustment of air cap and fluid tip, distance of gun from work, and rate of flow of material at the spray nozzle shall be as agreed upon between purchaser and seller.

12.5 Automatic Application—Four methods of applying films of uniform thickness automatically are described in Test Methods D 823. The methods are as follows:

Test Method A—Automatic Spraying Machine,
Test Method B—Automatic Dip-Coater,
Test Method C—Automatic Blade Film Applicator, and
Test Method D—Motor-Driven Blade Film Applicator.

13. Film Thickness

13.1 Many properties of lacquer films vary with the thickness of the dry film. The measurement of dry film thickness may be accomplished with various apparatus depending upon the substrate to which the film is applied. Three nondestructive methods of thickness determination are given for films that are not easily removable from their substrate and thus do not lend themselves to simple measurement with a constant-pressure micrometer. Two destructive methods are given. The methods are as follows:

13.2 Nondestructive Methods:

13.2.1 Test Method D 1186. This method is also recommended for thin films, less than 0.5 mil (0.005 in.) (0.013 mm) in thickness.

13.2.2 Test Method D 1400, Method A. By the use of plastic shims of known thickness, the film thickness can be estimated to approximately $\pm 10\%$.

13.2.3 Test Methods D 1400, Method C.

13.3 Destructive Methods:

13.3.1 Test Methods D 1005.

13.3.2 Test Methods D 1400, Method B.

14. Gloss

14.1 This property relates to that aspect of the reflecting properties of a coating as determined by the brightness and configuration of reflected images.

14.2 Determine gloss of nonmetallic finishes in accordance with Test Method D 523. This method covers the measurement of the amount of light reflected specularly from a film's surface. While this property is a function of film gloss, correlation with visual observations is not always obtained.

15. Color-Pigmented Coatings

15.1 The colors of opaque objects such as painted surfaces may be specified using visual or instrumental means.

15.2 By visual means the colors of painted surfaces in terms of the Munsell Color System may be determined in accordance with Test Method D 1535. This method provides a simple alternative to the more precise and more complicated method of color specification based on spectrophotometry.

15.3 By spectrophotometer, either visual or photoelectric types, the colors of light-reflecting coatings and spectral characteristics may be determined using Method E 308.

16. Color Difference-Pigmented Coatings

16.1 The small daylight color differences between two similarly homogeneously colored, opaque objects such as painted surfaces may be determined using visual evaluating techniques or by instrumental means.

16.2 Determine by visual evaluation color differences of opaque materials using Practice D 1729. This practice specifies characteristics of light sources, illuminating and viewing conditions, size of specimens, and general procedures.

16.3 Determine small daylight color differences between opaque paint specimens in accordance with Method D 2244. This test method correlates reasonably well with visual judgments of character and perceptibility of color differences provided specimens are illuminated and viewed under conditions substantially the same as used in the instrument. For nonfluorescent opaque paint specimens or materials that do not contain metallic pigments ("bronze" powders and aluminum powders) or "pearly" pigments or other materials that produce optical phenomena that contribute to the visual effect, test in accordance with Test Method D 2244.

17. Elongation

17.1 An elongation test may be used as an indication of the flexibility of an attached organic coating. It can also show whether there is any change during aging.

17.2 Determine the elongation of attached organic coatings when applied to flat sheet metal of uniform surface texture in accordance with Test Method D 522 or Test Method D 1737.

18. Hardness

18.1 Determine film hardness of lacquers applied to plane rigid surfaces in accordance with Test Method D 1474. In this method the resistance of a coating to penetration by an indenter is measured and converted to a hardness value.

19. Abrasion Resistance

19.1 Determine the abrasion resistance as described in either Test Method D 658 or Test Method D 968. Both methods cover measurement of the resistance to abrasion of coatings applied to metal panels.

20. Print Test

20.1 A print test can be used to determine two characteristics of a film. If a film is thoroughly dry, the test will give a measure to the susceptibility of the film itself to pressure marring. However, as the test is used in a production situation, it can be used to determine the degree to which

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film has released solvents and hence whether the product can be safely packed.

20.2 Determine imprinting of dried films as described in Test Method D 2091.

21. Household Chemical Resistance

21.1 Household chemicals may alter the surface of organic coatings, for example through discoloration, change in gloss, blistering, swelling, and loss of adhesion.

21.2 Determine, qualitatively, the effects of household chemicals in accordance with Test Method D 1308.

22. Outdoor Exposure

22.1 It is important to determine the resistance to outdoor exposure of lacquers destined for exterior use prior to their application to surfaces which may be difficult or costly to refinish, or both. Systems of primer and lacquer should be tested as a whole rather than as separate components. Experience indicates that the type of substrate employed has a marked bearing on weathering results. It is the purpose of several ASTM methods to minimize the influence of substrate variation by providing uniform panel selection and uniform procedures for conducting exposure tests and for evaluating and recording results.

22.2 *Preparation of Panels*—Depending on the substrate anticipated to be coated, panels for outdoor exposure testing should be prepared and tested in accordance with the following methods:

22.2.1 Test Method D 1733.

22.2.2 Methods D 609.

22.2.3 Method D 1014.

22.3 *Test Procedures*—Many properties of organic coatings should be evaluated periodically throughout the outdoor exposure period. These properties may be evaluated as follows:

22.3.1 *Blistering*—Test Method D 714.

22.3.2 *Chalking*—Method D 659.

22.3.3 *Checking*—Test Method D 660.

22.3.4 *Cracking*—Test Method D 661.

22.3.5 *Rusting*—Method D 610.

22.3.6 *Erosion*—Test Method D 662.

22.3.7 *Flaking*—Test Method D 772.

23. Temperature-Change Resistance

23.1 A test for resistance to temperature change, or a cold cracking test (as it is sometimes called), is designed to give an indication of the resistance of a coating system to fracturing or checking, caused both by changes in temperature and humidity and by age. The degree of correlation between test results and long-term room temperature aging varies with the type of lacquer. The industry uses the test widely and feels that systems showing good cold crack resistance usually will give satisfactory performance in service. Some factors that can affect results are the type of substrate, its thickness, the rate at which the individual panels are cooled, the film thickness, and the nature of any priming or sealer coats.

23.2 Determine the temperature-change resistance in accordance with Test Method D 1211. This method specifies that the test panels be aged 10 days. If the panels are much older than this, the resistance to checking is expected to be

less and sometimes considerably so depending on the nature of the film.

24. Salt Fog

24.1 Salt spray (fog) testing of lacquers applied to metal substrates is helpful in determining their resistance to failure in service under atmospheric conditions that might prevail in sea coast areas.

24.2 Under accelerated conditions of laboratory testing, only the temperature, the pH, or concentration of the salt solution, and other physical properties can be controlled, the selection of the substrate, the coating technique, the manner in which the coating is scribed (parallel or X's), the location or position of the panels within the cabinet, the length of the test, the inspection of the panels, and the method of reporting results must generally be as agreed upon between purchaser and seller. Systems of primer and lacquer should be tested as a whole rather than as separate components.

24.3 Determine the salt spray resistance as described in Method B 117.

25. Water Fog Testing

25.1 Water fog testing of lacquers is helpful in determining their resistance to failure in service under conditions of high humidity. Failure is usually evidenced by blushing, dulling, blistering, or excessive softening that does not disappear upon evaporation of the absorbed water. Systems of primer and top-coat lacquer should be tested as a whole rather than as separate components. The use of unrealistically short drying times or excessively low temperature bakes will give erratic and misleading results.

25.2 Determine resistance to failure under conditions of high humidity in accordance with Test Method D 1735.

26. Water Immersion Testing

26.1 Water immersion testing is best suited for testing lacquers or systems that will actually be soaked in water during service. Materials that will be subjected to humid atmospheres only should be subjected to water fog testing. Failure is usually evidenced by blushing, dulling, blistering, or excessive softening that does not disappear upon evaporation of the absorbed water.

26.2 Determine, in an accelerated manner, the resistance to failure of a coating immersed in water in accordance with Practice D 870.

27. Hiding Power

27.1 Hiding power of chromatic and non-chromatic coatings can be determined using Test Method D 2805.

28. Perspiration Resistance

28.1 The effects of human perspiration on the surface of organic coatings are varied. The coating may undergo an objectionable alteration in surface appearance such as discoloration, loss of gloss, blistering, wrinkling, or roughening. The coating may lose adhesion, soften, become embrittled, or any combination of these changes may take place.

28.2 Determine the effects of perspiration on organic coatings in accordance with Test Method D 2204. This method covers an accelerated procedure for determining the resistance to failure of organic coatings on metallic panels

when exposed to human perspiration.

29. Plasticizer Migration

29.1 Plasticizers used in vinyl fabrics may migrate into lacquer films that come in contact with the fabric and cause objectionable marring and softening of the lacquer film.

29.2 Determine the tendency for plasticizer to migrate in accordance with Test Method D 2199.

30. Chip Resistance

30.1 Chip resistance testing is useful for determining the ability of the coating or coating system, or both, to withstand

flying stones or other objects. Systems of primer and topcoat should be tested as a system rather than as separate components.

31. Light Stability of Clear Coatings

31.1 The ability of clear coatings not to change color upon exposure to sunlight is an important characteristic, especially over white or light-colored substrates. Coatings for indoor use may be tested in accordance with Test Method D 2620.

32. Keywords

32.1 clear lacquers; pigmented lacquers

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

1. Scope

1.1 This standard covers the test method for determining the resistance of clear and pigmented lacquer coatings to chipping by impact.

2. Referenced Standards

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3. Terminology

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Standard Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts¹

This standard is issued under the fixed designation D 344; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides for the qualitative and quantitative visual determination of the hiding power of a test paint relative to that of a comparison paint.

1.2 This test method describes only a brushout application procedure in specific detail, but its concepts are valid for other methods of application as well.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products³
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁴
- D 2805 Test Method for Hiding Power of Paints by Reflectometry³
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Definitions D 16 and the *Paint/Coatings Dictionary*.⁶

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *relative hiding power, qualitative*—the characterization of a test paint as being better, equal, or poorer in hiding power than a comparison paint.

3.2.2 *relative hiding power, quantitative*—the spreading rate of a paint expressed as a percent of the spreading rate of

a comparison paint at equal hiding.

4. Summary of Test Method

4.1 *Qualitative*—The test and comparison paints are brushed out uniformly at the same spreading rate on black and white hiding power charts. After drying the brushouts are compared visually to see which paint has been most effective in reducing the substrate contrast.

4.2 *Quantitative*—Additional brushouts are made with the comparison paint, if and as necessary, to determine the spreading rate at which it matches the hiding of the test paint brushout.

5. Significance and Use

5.1 This test method evaluates the hiding power of a test paint relative to a comparison paint. The results have significance only within that relationship. It may be used for production control or quality comparisons.

5.2 When a paint is applied by brush or any other practical method, the opacity of the film is affected by variations in film thickness related to the application procedure and to the application characteristics of the paint. Two paints that hide equally well by this method might therefore differ considerably when applied with a doctor blade, since the latter method gives essentially perfect leveling. Different brushes or surface application conditions can likewise give different results.

NOTE 1—Test Method D 2805 describes an instrumental method for quantitatively determining hiding power without reference to a material paint standard. The paint film is applied at a uniform thickness (for example, with a doctor blade), the film thickness is measured rigorously, and the opacity is evaluated photometrically. Hiding power is thereby determined with a high degree of precision.

5.3 Test Method D 344 is less precise than Test Method D 2805, but is more practical because it is responsive to the application characteristics of paints and is simpler in concept and execution.

6. Apparatus

6.1 *Balance*, top-loading laboratory balance having a capacity of 100 g and sensitivity of 0.1 g.

6.2 *Syringe*, 10-mL capacity.

6.3 *Paint Brush*, ferrule 2 in. (50 mm) wide and $\frac{1}{16}$ in. (14 mm) thick, tapered polyester filaments $2\frac{3}{4}$ in. (70 mm) long with flagged chisel trim tip.⁷

⁷ Describes the Leneta-EZ Painter Certified Standard Paint Brush used by the round-robin participants, available from Leneta Company, P. O. Box 86, Ho-Ho-Kus, NJ 07423. Equivalent brushes may be used.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved May 26, 1989. Published August 1989. Originally published as D 344 - 32. Last previous edition D 344 - 84.

² *Annual Book of ASTM Standards*, Vols 06.01, 06.02, and 06.03.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

⁶ Published by the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

6.4 *Container*, ½-pint can or 250-mL beaker.

6.5 *Test Surface*, a smooth-surfaced paper chart having adjacent black and white areas and coated with a suitable varnish or lacquer so as to render the surface impervious to paint liquids. The black and white areas of the chart shall have luminous reflectance factors of 1 % maximum and 75 % minimum, respectively, when tested in accordance with Test Method E 97. The white areas of the charts used in any one testing program shall not differ in their reflectance factors by more than 1 %. Any suitable regular design of contrasting areas may be used. The chart⁸ shall have a defined test area of 0.1 m². Larger test areas may be used if available, with appropriate modification of the indicated calculations.

6.6 *Illumination*, a light source providing diffuse light of reasonable intensity, preferably northern sky light or an approximation of same. (See Practice D 1729).

7. Preparation of Brushouts

7.1 Select a convenient and suitable spreading rate, mutually agreeable to all parties involved in the testing program, and preferably in the range from 400 to 800 ft²/gal (9.8 to 19.6 m²/L). In any case, the spreading rate shall not be so low that the substrate contrast is difficult to see nor so high that it is difficult to apply the paint at a reasonably uniform film thickness.

NOTE 2—The gallon unit referred to here and throughout this test method is the U.S. gallon (3785.4 mL).

7.2 Determine the density *D* in grams per millilitre of the comparisons and test paints, in accordance with Test Method D 1475.

7.3 Calculate to three significant figures the volume *V* in millilitres to apply for the selected spreading rate *SR* as follows:

$$V(\text{mL}) = \frac{100}{SR(\text{m}^2/\text{L})} = \frac{4074.6}{SR(\text{ft}^2/\text{gal})}$$

7.4 Calculate to three significant figures, the weight *W* in grams corresponding to the volume *V*(mL) as follows:

$$W(\text{g}) = V(\text{mL}) \times D(\text{g/mL})$$

7.5 To prepare a brushout, first stir the paint well. Then precondition the brush by dipping it into the paint and working it out on a smooth, nonporous surface. Take up the desired volume of paint in the syringe and weigh it with the brush in the empty container. Place the test chart on a level bench top, distribute the paint from the syringe all around onto the test area of the chart, then brush the paint out uniformly within the test area. Set the brushout aside to dry in a horizontal position, protected adequately from dust and under normal room conditions of ventilation and temperature. Reweigh the empty syringe with the brush and container. The loss of weight represents the actual weight of paint applied to the test surface as opposed to the intended weight calculated from 7.3 and 7.4. If there is a discrepancy do not attempt to correct it by removing or applying paint

since this would disturb the leveling of the brushout. Instead, calculate the actual spreading rate as follows:

$$SR_A = \frac{B}{A} \times SR_B$$

where:

SR_A = actual spreading rate,

B = intended weight,

A = actual weight, and

SR_B = intended spreading rate.

8. Evaluation Procedure

8.1 *Viewing conditions*—When the test and comparison paint brushouts are thoroughly dry, place them vertically side by side against a flat surface and view them from a distance of 5 to 10 ft (1.5 to 3 m) under illumination conditions as described in 6.6.

8.2 Qualitative Evaluation:

8.2.1 If the contrast between the black and white areas of the test paint brushout is adjudged less than, equal to, or more than that of the comparison paint brushout, then the hiding power of the test paint is considered as better than, equal to, or poorer than that of the comparison paint.

8.2.2 If the actual spreading rate of the lower contrast paint-out is lower than that of the other paint-out by more than 3 %, prepare a second brushout with either paint so as to eliminate the difference, then compare again to confirm or revise the previous estimation of relative hiding power.

8.3 Quantitative Evaluation:

8.3.1 If the hiding of the test paint brushout does not match that of the comparison paint at the same spreading rate, make a series of additional comparison paint brushouts at several other spreading rates, differing in steps of approximately 15 % and providing contrasts above and below that of the test paint brushout. Refer to these and to the original comparison paint brushout as standards. If the standards are prepared correctly each increase in spreading rate will correspond to a perceptible increase in contrast. Compare the standards under the viewing conditions described in 8.1 to assure that this is the case.

8.3.2 Compare the brushout of the test paint with the standards under the specified viewing conditions to determine which standard it matches in contrast or, if none, to which pair of successive standards it is intermediate. In the latter case view the test paint brushout with the two standards on either side and rate the contrast of the test paint brushout by estimating the contrast difference between it and the lower spreading rate (lower contrast) standard, as a fraction of the contrast difference between the two standards to the nearest fourth. Keeping the test paint brushout in the middle, reverse the position of the two standards and rate the test paint brushout again in the same way. The final rating is the mean of the two to the nearest eighth.

8.3.3 Calculate the spreading rate (*SR_C*) of the comparison paint at which it is estimated to match the hiding of the test paint brushout as follows:

$$SR_C = SR_1 + X(SR_2 - SR_1)$$

where:

SR₁ = spreading rate of the lower spreading rate (lower contrast) standard,

SR₂ =

X =

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⁸ Suitable charts are available from Leneta Company, P. O. Box 86, Ho-Ho-Kus, NJ 07423.

nstead,

SR_2 = spreading rate of the higher spreading rate (higher contrast) standard, and

X = fractional contrast rating of the test paint brushout.

8.3.4 Calculate the relative hiding power of the test paint in accordance with the definition in 3.2.2 as follows:

$$HP_t = \frac{SR_t}{SR_c} \times 100$$

where:

HP_t = relative hiding power of the test paint,

SR_t = spreading rate of the test paint, and

SR_c = spreading rate of the comparison paint at equal hiding, as determined by an actual match with one of the standards or by estimation between two standards as described in 8.3.2.

9. Report

9.1 *Qualitative* (see 8.2)—Report the relative hiding power of the test paint as better than, equal to, or poorer than the comparison paint. The report may be amplified by such terms as exactly, slightly, moderately, considerably, etc., as may seem appropriate.

9.2 *Quantitative* (see 8.3)—Report the relative hiding power of the test coating to the nearest whole number. This is a purely numerical value. Physical units such as square feet per gallon or square metres per litre are not applicable.

10. Precision

10.1 *Qualitative*—Four test paints were rated versus a comparison paint by three laboratories, twice each by one laboratory and once each by the other two, for a total of 16 trials. There was only one discrepancy, which occurred with a paint that was apparently very similar in hiding power to the comparison paint. These results indicate that the method is effective, but more results would be required to develop an appropriate precision statement. Table 1 gives the results of these tests.

10.2 *Quantitative*—In an interlaboratory study of this

TABLE 1 Round Robin Results of Relative Hiding Power of Four White Paints versus a Comparison White Paint

Method	Paint	Laboratory 1		Laboratory 2	Laboratory 3
		Trial 1	Trial 2		
Qualitative	V1	A	A	A	A
	V2 ^a				
	V3	C	C	C	C
	V4	C	C	C	C
	V5	A	C	C	C
Quantitative	V1	125	117	115	120
	V2 ^a	100	100	100	100
	V3	85	93	62	96
	V4	66	82	60	67
	V5	102	90	83	95

^a Indicates that the test paint hides better than the comparison paint.

^b Comparison paint, relative hiding power equals 100.

^c Indicates that the test paint hides poorer than the comparison paint.

method four coatings were evaluated for hiding power against a fifth coating taken as the standard. In one laboratory each material was tested twice, but only once in the other two laboratories. Statistical analysis of the results revealed that the two results for each coating from the one laboratory were as variable as those from the other two. Consequently, the two results were treated as being individual results and the between-laboratory standard deviation was found to be 10 % with 12 df. Based on this standard deviation, the following criterion should be used for judging the acceptability of results at the 95 % confidence level:

10.2.1 *Reproducibility*—Two single results obtained by operators in different laboratories should be considered suspect if they differ in relative hiding power by more than 32 %.

10.2.2 *Repeatability*—A reasonable estimate of within-laboratory precision could not be obtained because only one laboratory repeated the test. However, the repeatability should be similar to the reproducibility.

11. Keywords

11.1 hiding power; relative hiding; opacity

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Standard Specification for Wood to Be Used as Panels in Weathering Tests of Coatings¹

This standard is issued under the fixed designation D 358; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 2031 of Federal Test Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This specification designates woods for weathering tests of exterior solvent-borne or water-borne paints and other materials of similar purpose. Such tests may include either outdoor exposure tests or accelerated laboratory tests. It is the purpose of this specification to minimize the influence of variation of wood of a given species on test results.

2. General Considerations

2.1 A variety of different species should be used for testing finishes because of the wide variations in the anatomy and the density between wood species. The factors have tremendous influences on finish performance. Latewood band width and density are especially important to finish performance, and these parameters vary significantly between wood species. Cedar and redwood have relatively low proportions of hard summerwood distributed in narrow bands and contain extractives that discolor some paint films. Southern pine has a large proportion of hard summerwood in wide bands.

2.2 The angle between the growth rings and the surface of the specimen is very critical to finish performance. Commercial practice utilizes the following classification system. Boards in which growth form an angle of less than 30° with the wide surface of the piece are said to be flat-sawn or flat-grained; those with the rings intersecting at angles between 30° and 60° are termed bastard-sawn; and those with angles greater than 60° are termed quarter-sawn, edge grain, or vertical grain.

WESTERN RED CEDAR

3. Species

3.1 The material for test panels shall be western red cedar (*Thuja plicata*).

4. Weight per Volume

4.1 When the wood is in equilibrium with air at 60 to 65 % relative humidity and 70 ± 1°F (21 ± 0.5°C) it shall weigh 22 to 23 lb/ft³ (350 to 370 kg/m³) or at 50 ± 5 % relative humidity and 73.5 ± 3.5°F (23 ± 2°C), which are

¹ This specification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility D01.52 on Factory-Coated Wood Products.

Current edition approved Oct. 28, 1983. Published December 1983. Originally published as D 358 - 33. Last previous edition D 358 - 77.

specified in Specification D 3924 as standard conditions for testing paint and related coatings, it shall weigh 21.4 to 22.4 lb/ft³ (343 to 359 kg/m³).

NOTE 1—The density requirement is to eliminate wood specimens that vary greatly from the average for that species. Either set of conditions may be used to establish the density. At 60 to 65 % relative humidity wood has a moisture content of approximately 12 % while at 45 to 55 % relative humidity it contains 9 to 10 %. The former is closer to the moisture content of wood exposed to most exterior climatic conditions.

5. Character of Wood

5.1 The wood shall be heartwood having edge grain and shall be free of knots and other defects. The color of the wood shall be uniform across each panel with no streaks that are excessively light or dark. The number of annual growth rings per inch along the radius of the log from which the wood was obtained shall not be less than ten. The surfaces shall be smoothly planed and sanded thoroughly with 150 grit sandpaper within two weeks of testing.

6. Thickness of Panels

6.1 The minimum thicknesses (Note 2) in proportion to width (across the grain) shall be as follows:

Panel Width, in. (mm)	Minimum Thickness, in. (mm)
Less than 3 (less than 75)	3/8 (10)
3 to 6 (75 to 150)	7/16 (11)
Over 6 (over 150)	1/4 (19)

NOTE 2—Minimum thicknesses in proportion to width (across the grain) do not apply to panels made of clapboard or siding of commercial types, which shall be used as regularly marketed.

WHITE PINE AND PONDEROSA PINE

7. Species

7.1 White pine wood shall be northern white pine (*Pinus strobus*). Western white pine (*Pinus monticola*), or sugar pine (*Pinus lambertiana*). Western white pine is sometimes called Idaho white pine. Ponderosa pine wood shall be *Pinus ponderosa*.

8. Weight per Volume

8.1 When the wood is in equilibrium with air at 60 to 65 % relative humidity and 70 ± 1°F (21 ± 0.5°C) it shall weigh 23 to 27 lb/ft³ (370 to 430 kg/m³) or at 50 ± 5 % relative humidity and 73.5 ± 3.5°F (23 ± 2°C) it shall weigh 22.4 to 26.4 lb/ft³ (359 to 423 kg/m³). (See Note 1.)

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9. Character of Wood

9.1 The wood shall be free of knots, pitch pockets, and other defects. The color of the wood shall be uniform across each panel with no streaks that are excessively light or dark. The number of annual growth rings per inch along the radius of the log from which the wood was obtained shall be not less than 15. The surfaces shall be smoothly planed and sanded thoroughly with 150 grit sandpaper within two weeks of testing.

9.2 The wood shall be selected as edge grain or flat grain as desired. The test surface of flat grain wood shall be on the bark side.

10. Thickness of Panels

10.1 The minimum thicknesses (see Note 3) in proportion to width (across the grain) shall be as follows:

Panel Width, in. (mm)	Minimum Thickness, in. (mm)
Less than 3 (less than 75)	$\frac{7}{16}$ (11)
3 to 6 (75 to 150)	$\frac{7}{16}$ (11)
Over 6 (over 150)	$\frac{3}{4}$ (19)

SOUTHERN PINE

11. Species

11.1 The material for test panels shall be southern pine (*Pinus sp.*), suitable for siding purposes. It is not to be limited to a single species, but shall conform to the requirements prescribed in Sections 12 and 13.

12. Weight per Volume

12.1 When the wood is in equilibrium with air at 60 to 65 % relative humidity and $70 \pm 1^\circ\text{F}$ ($21 \pm 0.5^\circ\text{C}$) it shall weigh 32 to 38 lb/ft³ (510 to 610 kg/m³) or at 50 $\pm$ 5 % relative humidity and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) it shall weigh 31 to 37 lb/ft³ (500 $\pm$ 590 kg/m³). (See Note 2.)

13. Character of Wood

13.1 The wood shall be free of knots, pitch pockets, and other defects. The color of the wood shall be uniform across each panel with no streaks that are excessively light or dark. The number of annual growth rings per inch along the radius of the log from which the wood was obtained shall be not less than 4 nor more than 12. The surfaces shall be smoothly planed and sanded thoroughly with 150 grit sandpaper within two weeks of testing.

13.2 The wood shall be sapwood having flat grain.

14. Thickness of Panels

14.1 The minimum thicknesses (see Note 3) in proportion to width (across the grain) shall be as follows:

Panel Width, in. (mm)	Minimum Thickness, in. (mm)
Less than 3 (less than 75)	$\frac{7}{16}$ (11)
3 to 6 (75 to 150)	$\frac{7}{16}$ (11)
Over 6 (over 150)	$\frac{3}{4}$ (19)

REDWOOD

15. Species

15.1 The material for test panels shall be redwood (*Sequoia sempervirens*).

16. Weight per Volume

16.1 When the wood is in equilibrium with air at 60 to 65 % relative humidity and $70 \pm 1^\circ\text{F}$ ($21 \pm 0.5^\circ\text{C}$) it shall weigh 28 to 29 lb/ft³ (450 to 460 kg/m³) or at 50 $\pm$ 5 % relative humidity and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) it shall weigh 27.2 to 28.2 lb/ft³ (436 to 452 kg/m³). (See Note 1.)

17. Character of Wood

17.1 The material shall be heartwood, as nearly edge grain as possible. The number of annual growth rings per inch along the radius of the log from which the wood was obtained shall be not less than 18. The wood shall be free of knots, oil streaks, and other defects such as wavy burl grain. The color of the wood shall be uniform across each panel with no streaks that are excessively light or dark. The surfaces shall be smoothly planed and sanded thoroughly with 150 grit sandpaper within two weeks of testing.

18. Thickness of Panels

18.1 The minimum thicknesses (see Note 2) in proportion to width (across the grain) shall be as follows:

Panel Width, in. (mm)	Minimum Thickness, in. (mm)
Less than 3 (less than 75)	$\frac{3}{8}$ (10)
3 to 6 (75 to 150)	$\frac{7}{16}$ (11)
Over 6 (over 150)	$\frac{3}{4}$ (19)

DOUGLAS FIR

19. Species

19.1 The material for test panels shall be Douglas fir (*Pseudotsuga menziesii*).

20. Weight per Volume

20.1 When the wood is in equilibrium with air at 60 to 65 % relative humidity and $70 \pm 1^\circ\text{F}$ ($21 \pm 0.5^\circ\text{C}$) it shall weigh 30 $\pm$ 36 lb/ft³ (480 to 580 kg/m³) or at 50 $\pm$ 5 % relative humidity and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) it shall weigh 29 to 35 lb/ft³ (460 to 560 kg/m³). (See Note 1.)

21. Character of Wood

21.1 The wood shall be free of knots, pitch pockets, and other defects. The color of the wood shall be uniform across each panel with no streaks that are excessively light or dark. The number of annual growth rings per inch along the radius of the log from which the wood was obtained shall be not less than ten. The surfaces shall be smoothly planed and sanded thoroughly with 150 grit sandpaper within two weeks of testing.

21.2 The wood shall be heartwood and selected as edge grain or flat grain as desired. The test surface of flat grain wood shall be on the bark side.

22. Thickness of Panels

22.1 The minimum thicknesses (see Note 2) in proportion to width (across the grain) shall be as follows:

Panel Width, in. (mm)	Minimum Thickness, in. (mm)
Less than 3 (less than 75)	$\frac{7}{16}$ (11)
3 to 6 (75 to 150)	$\frac{7}{16}$ (11)
Over 6 (over 150)	$\frac{3}{4}$ (19)

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

1. Scope

1.1 This standard covers the resistance of coatings to abrasion.

1.2 This standard addresses the responsibility of the user in the application of this standard.

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Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings¹

This standard is issued under the fixed designation D 522; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 6222 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{ε1} NOTE—Table 2 was editorially corrected in May 1989.

1. Scope

1.1 These test methods cover the determination of the resistance to cracking (flexibility) of attached organic coatings on substrates of sheet metal or rubber-type materials.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

3. Summary of Test Methods

3.1 The coating materials under test are applied at uniform thickness to panels of sheet metal or rubber-type materials. After drying or curing the coated panels are bent over a mandrel and the resistance to cracking of the coating is determined. In Test Method A the coated panels are bent over a conical mandrel. In Test Method B the coated panels are bent over cylindrical mandrels of various diameters.

4. Significance and Use

4.1 Coatings attached to substrates are elongated when the substrates are bent during the manufacture of articles or when the articles are abused in service. These test methods

have been useful in rating attached coatings for their ability to resist cracking when elongated. They have been useful in evaluating the flexibility of coatings on flexible substrates.

5. Test Specimens

5.1 Substrates:

5.1.1 If the purpose of the test is to determine the percent of elongation of the coating material, the substrate shall be cold-rolled steel strip 1/32 in. (0.8 mm) (22 gage) thick.

5.1.2 If the purpose of the test is to rate the coated material for resistance to cracking, the substrate may be any type of sheet metal or rubber-type material (for example, steel, aluminum, tinplate, or synthetic rubber). The thickness of the sheet metal may be less than 1/32 in. (0.8 mm) and the thickness of the rubber-type materials may be as great as 1/2 in. (13 mm).

5.1.3 The recommended panel size is 4 in. (100 mm) in width and 6 in. (150 mm) in length. The maximum size that the conical mandrel can accommodate is 4 1/2 in. (115 mm) wide and 7 1/2 in. (190 mm) long.

5.1.4 The surface preparation of the substrate shall be agreed upon between the purchaser and the seller. Prior to the application of the coating, round slightly the edges of metal panels to remove burrs in order to eliminate anomalous edge effects.

NOTE 1—If elongations are to be determined for coatings applied to substrates other than 1/32 in. (0.8 mm) thick cold-rolled steel, they must be measured with a special mandrel jig as described in Appendix X1.2 and shown in Fig. X1.1. The procedure for using the jig is described in Appendix X1.3.

5.2 Coated Panels:

5.2.1 Apply uniform coatings of the materials under test to the substrates in accordance with Test Methods D 823 and air dry or bake under conditions mutually agreeable to the purchaser and the seller. If percent elongation is to be measured, a minimum thickness of 1.0 mil is required on 1/32 in. (0.8 mm) thick cold-rolled steel. Perform a minimum of three determinations of coating thickness on each of the specimens in accordance with Test Methods D 1005, D 1186, or D 1400.

6. Conditioning and Number of Tests

6.1 Condition the test specimens for at least 24 h at 73.5 ± 3.5°F (23 ± 2°C) and 50 ± 5 % relative humidity, and test in the same environment or immediately on removal therefrom

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films. Current edition approved May 27, 1988. Published November 1988. Originally published as D 522 - 39. Last previous edition D 522 - 85.

² Annual Book of ASTM Standards, Vol 06.01.

unless otherwise specified by the purchaser and seller.

6.2 Test at least three replicate specimens in Test Method A and at least two replicate specimens in Test Method B at each mandrel diameter of interest.

TEST METHOD A—CONICAL MANDREL TEST

7. Apparatus

7.1 *Conical Mandrel Tester*, consisting of a metal cone, a rotating panel-bending arm, and panel clamps, all mounted on a metal base as illustrated in Fig. 1.

7.1.1 *Cone*, smooth steel, 8 in. (203 mm) in length, with a diameter of 1/8 in. (3 mm) at one end and a diameter of 1 1/2 in. (38 mm) at the other end.

8. Procedure

8.1 With the operating lever of the apparatus in a horizontal position, slip the test specimen between the mandrel and the drawbar with the finish side towards the drawbar. Rigidly clamp the specimen in a vertical position adjacent to the mandrel by placing the long edge behind the clamping bar in such a manner that the panel is always set up to the narrow end of the mandrel. Slip two sheets of No. 1 brown kraft wrapping paper, substrate 30, thoroughly lubricated on each side with talc, between the specimen and the drawbar and hold in position only by the pressure of the drawbar against the paper.

8.2 Move the lever through about 180° at uniform velocity to bend the specimen approximately 135°. If the purpose of the test is to measure percent elongation, the bend time should be about 15 s. To determine crack resistance under more simulated use conditions, the bend time should be some value between 1.5 s and 5 s to be agreed upon between the purchaser and the seller.

8.3 Examine the bent surface of the specimen immediately with the unaided eye for cracking. Having determined and suitably marked the end of the crack farthest from the small end of the mandrel, which shall be considered as the end point, bring the drawbar to the starting position and remove the panel from the mandrel. Measure the distance from the farthest end of the crack to the small end of the mandrel. This distance is used to compute the elongation.

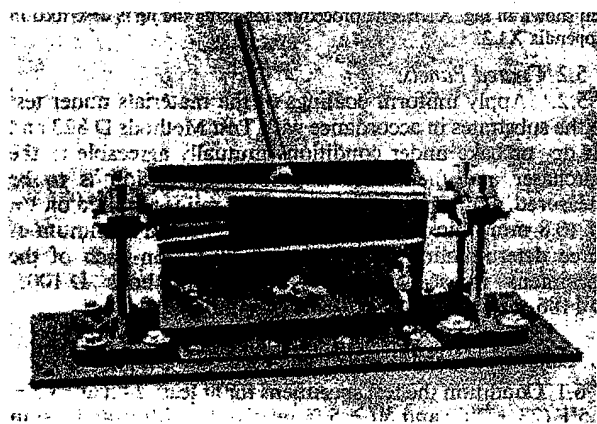


FIG. 1 Conical Mandrel Test Apparatus

The mandrel diameter at which cracking ceased is taken as the resistance to cracking value.

NOTE 2—In some cases, the measured distance is used as a measure of crack resistance.

9. Calculation

9.1 Determine the elongation of the finish from the plotted curve in Fig. 2. This curve represents the relationship between the percent elongation and the diameter of the conical mandrel for a 1.0 mil coating thickness. The relation between the distance along the conical mandrel and the corresponding diameter has also been plotted on this curve.

9.2 Adjust the percent elongation value obtained from Fig. 2 for coating thickness by adding the correction obtained from Fig. 3.

NOTE 3—*Example*—Suppose a visual examination of the finish on the bent cold-rolled steel specimens 1/32 in. (0.8 mm) in thickness shows that the end of the first crack in the coating is at a distance of 3 in. (75 mm) from the small end of the cone. From Fig. 2 determine the percent elongation of the film from the measured crack distance, in this example 5.2 %. To correct for coating thickness add the value obtained in Fig. 3. At a crack distance of 3 in. the correction per mil (25 μm) of coating thickness is 0.3 %. If the film thickness in the example is 2 mils (50 μm), the actual percent elongation is $5.2 + (2 \times 0.3) = 5.8 \%$.

10. Report

10.1 Report the following information:

- 10.1.1 Mean and range of coating elongation or resistance to cracking values for each specimen,
- 10.1.2 Mean and range of coating film thickness for each specimen,
- 10.1.3 Specimen preparation procedures used,
- 10.1.4 Test conditions, and
- 10.1.5 Mean and range of elongation or resistance to cracking, and film thickness for the replicate specimens.

11. Precision

11.1 Results are not available to determine the precision of this test method. This test method has been in use for many years and is considered acceptable for evaluating the resistance to cracking of attached coatings.

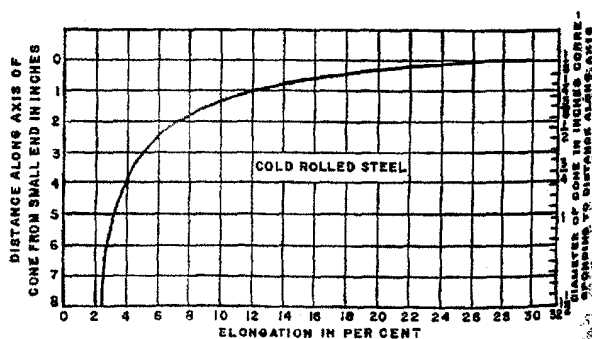


FIG. 2 Distance Along Cone and Corresponding Mandrel Size versus Percent Elongation for Specimens on Cold-Rolled Steel 1/32 in. (0.8 mm) in Thickness

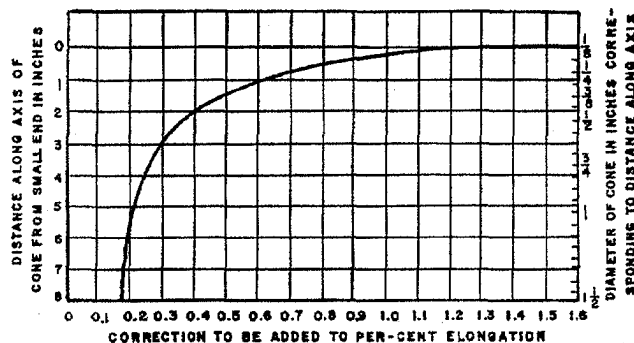


FIG. 3 Correction for Thickness of Film

TEST METHOD B—CYLINDRICAL MANDREL TEST

12. Apparatus

12.1 *Elongation Test Equipment*, consisting of rods or surfaces, including cylindrical steel diameters of 1 in. (25 mm), 3/4 in. (19 mm), 1/2 in. (12.7 mm), 3/8 in. (9.5 mm), 1/4 in. (6.4 mm), and 1/8 in. (3.2 mm), mounted on a metal base. An example of an acceptable form of this device is illustrated in Fig. 4.

13. Procedure

13.1 Place the test panel over a mandrel with the uncoated side in contact and with at least 2 in. (50 mm) overhang on either side. Using a steady pressure of the fingers, bend the panel approximately 180° around the mandrel at a uniform velocity in a time of 1.5 to 5 s, the time being that agreed upon between the purchaser and the seller. Remove and examine the panel immediately for cracking visible to the unaided eye. If cracking has not occurred, repeat the procedure using successively smaller diameter

mandrels on previously untested areas of a specimen until failure occurs or until the smallest diameter mandrel has been used.

13.1.1 This procedure can be applied as a "pass/fail" test by determining whether cracking is produced by a specified mandrel size.

13.1.2 The resistance to cracking value for a coating is taken as the mandrel diameter at which cracking ceases.

14. Calculation

14.1 Determine the elongation range of the material from Table 1 in which the elongation of a 1 mil (25 μm) thick coating on 1/32 in. (0.8 mm) cold-rolled steel is given for each mandrel diameter.

14.2 If the coating thickness exceeds 1 mil (25 μm), correct the determined elongation for film thickness using the factors for each mandrel diameter given in Table 2. Calculate the total elongation of the coating as follows:

$$E = e_1 + tc_1$$

where:

E = total elongation, %

e_1 = elongation from Table 1, %

t = thickness, mils, and

c_1 = correction factor from Table 2.

15. Report

15.1 Report the following information:

TABLE 1 Elongation

Mandrel Diameter, in. (mm)	Elongation, %
1 (25)	3.3
3/4 (19)	4.4
1/2 (12.7)	6.75
3/8 (9.5)	9.0
1/4 (6.4)	14.0
1/8 (3.2)	28.0

TABLE 2 Correction for Film Thickness

Mandrel Diameter, in. (mm)	Correction Factor
1 (25)	0.21
3/4 (19)	0.26
1/2 (12.7)	0.38
3/8 (9.5)	0.50
1/4 (6.4)	0.71
1/8 (3.2)	1.40

FIG. 4 An illustration of a cylindrical mandrel test apparatus

15.1.1 Mean and range of coating elongation or resistance to cracking values for each specimen,

15.1.2 Mean and range of coating thickness for each specimen,

15.1.3 Specimen preparation procedures used,

15.1.4 Test conditions, and

15.1.5 Mean and range of elongation, resistance to cracking, and film thickness for the replicate specimens.

16. Precision and Bias

16.1 Results are not available to determine the precision

of these test methods. These test methods have been in use for many years and are considered acceptable for evaluating the crack resistance of attached coatings.

INDEX TERMS

17. Index Terms

17.1 These test methods are indexed under the following terms: cracking; flexibility; resistance—cracking.

APPENDIX

(Nonmandatory Information)

XI. RELATIONSHIP BETWEEN ELONGATION AND MANDREL SIZE

X1.1 In the mandrel test, the specimens are elongated considerably past their elastic limit. Therefore, the elongations obtained are considerably higher than elongation values calculated from a theoretical equation that is based on the assumption that the elastic limits of the specimens are not exceeded.

X1.2 The elongation values corresponding to mandrel diameters in Figs. 2 and 3 of these test methods were obtained by direct measurements with a special mandrel jig.

X1.3 This jig shown in Fig. X1.1 consists of two forks for holding the mandrel and specimen in position and a roller with which the specimen is tightly wrapped around and held against the mandrel.

X1.4 *Measuring Elongation With Jig*—Make two parallel

gage scratches with a sharp stylus on the edge of the specimen so located that when it is wrapped around the mandrel the included angle between the scratches will be approximately 180°. In locating the scratches, it is necessary to make sure that they will be within the section of the specimen that, after bending, is tightly wrapped against the mandrel. Having located the scratches, measure the linear distance between them to within 2 μm. Clamp the specimen in the jig and bring the roller down tight against it. Draw the roller around causing the specimen to be tightly wrapped and secured against the surface of the mandrel. In order to hold the jig in this position hold the forks together with a C-clamp. Then rigidly mount the clamped jig on the table of the measuring machine.³ Each of the gage scratches shall be made to coincide with the vertical cross-hair of the measuring eyepiece while the horizontal cross-hair is tangent to the curvature of the upper surface of the specimen. Take the angle reading at each of these points. The difference between the two angle readings is a measure of the included angle formed by the two gage scratches, and the radii drawn coincident with the scratches. Read the angle readings in minutes and seconds to a precision of 5 s. Also determine the total diameter of the mandrel and the bent specimen. From these data calculate the percent elongation E at the surface of the specimen as follows:

$$L = \alpha\pi/180r$$

$$E = [(L - d)/d] \times 100$$

where:

α = angle, °

L = arc length,

r = radius of curvature for system (mandrel radius + panel thickness + coating thickness), and

d = original distance between scratches.

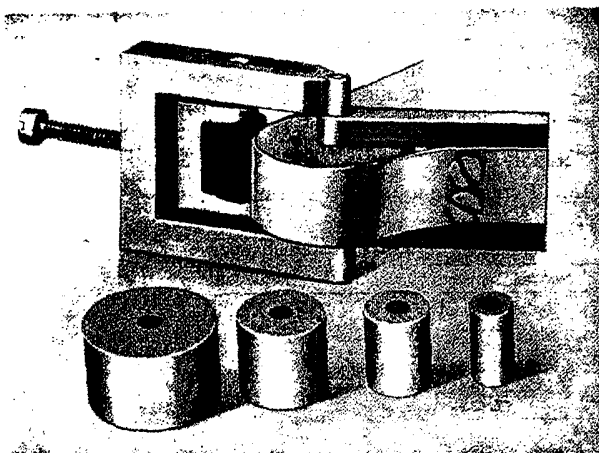


FIG. X1.1 Jig for Determining Elongation of Bent Metal Specimens

³ A star comparator has been found suitable for this purpose.

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Standard Test Method for Specular Gloss¹

This standard is issued under the fixed designation D 523; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the measurement of the specular gloss of nonmetallic specimens for glossmeter geometries of 60°, 20°, and 85° (1-7).²

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

D 3964 Practice for Selection of Coating Specimens for Appearance Measurements³

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials³

D 4039 Test Method for Reflection Haze of High-Gloss Surfaces⁴

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵

E 430 Method for Measurement of Gloss of High-Gloss Surfaces by Goniophotometry⁵

3. Terminology

3.1 Definitions:

3.1.1 *relative luminous reflectance factor*—the ratio of the luminous flux reflected from a specimen to the luminous flux reflected from a standard surface under the same geometric conditions. For the purpose of measuring specular gloss, the standard surface is polished glass.

3.1.2 *specular gloss*—the relative luminous reflectance factor of a specimen in the mirror direction.

4. Summary of Test Method

4.1 Measurements are made with 60°, 20°, or 85° geometry

(8, 9). The geometry of angles and apertures is chosen so that these procedures may be used as follows:

4.1.1 The 60° geometry is used for intercomparing most specimens and for determining when the 20° geometry may be more applicable.

4.1.2 The 20° geometry is advantageous for comparing specimens having 60° gloss values higher than 70.

4.1.3 The 85° geometry is used for comparing specimens for sheen or near-grazing shininess. It is most frequently applied when specimens have 60° gloss values lower than 10.

5. Significance and Use

5.1 Gloss is associated with the capacity of a surface to reflect more light in some directions than in others. The directions associated with mirror (or specular) reflection normally have the highest reflectances. Measurements by this test method correlate with visual observations of surface shininess made at roughly the corresponding angles.

5.1.1 Measured gloss ratings by this test method are obtained by comparing the specular reflectance from the specimen to that from a black glass standard. Since specular reflectance depends also on the surface refractive index of the specimen, the measured gloss ratings change as the surface refractive index changes. In obtaining the visual gloss ratings, however, it is customary to compare the specular reflectances of two specimens having similar surface refractive indices. Since the instrumental ratings are affected more than the visual ratings by changes in surface refractive index, non-agreement between visual and instrumental gloss ratings can occur when high gloss specimen surfaces differing in refractive index are compared.

5.2 Other visual aspects of surface appearance, such as distinctness of reflected images, reflection haze, and texture, are frequently involved in the assessment of gloss (1), (6), (7). Method E 430 includes techniques for the measurement of both distinctness-of-image gloss and reflection haze. Test Method D 4039 provides an alternative procedure for measuring reflection haze.

5.3 Little information about the relation of numerical-to-perceptual intervals of specular gloss has been published. However, in many applications the gloss scales of this test method have provided discriminations between coated specimens that have agreed well with visual discriminations of gloss (10).

5.4 When specimens differing widely in perceived gloss of color, or both, are compared, nonlinearity may be encountered in the relationship between visual gloss difference ratings and instrumental gloss reading differences.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved March 31, 1989. Published May 1989. Originally published as D 523 - 39 T. Last previous edition D 523 - 85¹.

² The boldface numbers in parentheses refer to the list of references at the end of this test method.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁵ Annual Book of ASTM Standards, Vols 6.01 and 14.02.

TABLE

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TABLE 1 Angles and Relative Dimensions of Source Image and Receptors

	In Plane of Measurement			Perpendicular to Plane of Measurement		
	$\theta, ^\circ$	$2 \tan \theta/2$	Relative Dimension	$\theta, ^\circ$	$2 \tan \theta/2$	Relative Dimension
Source image	0.75	0.0131	0.171	2.5	0.0436	0.568
Tolerance $\pm$	0.25	0.0044	0.057	0.5	0.0087	0.114
60° receptor	4.4	0.0768	1.000	11.7	0.2049	2.668
Tolerance $\pm$	0.1	0.0018	0.023	0.2	0.0035	0.046
20° receptor	1.8	0.0314	0.409	3.6	0.0629	0.819
Tolerance $\pm$	0.05	0.0009	0.012	0.1	0.0018	0.023
85° receptor	4.0	0.0698	0.909	6.0	0.1048	1.365
Tolerance $\pm$	0.3	0.0052	0.068	0.3	0.0052	0.068

6. Apparatus⁶

6.1 Instrumental Components—The apparatus shall consist of an incandescent light source furnishing an incident beam, means for locating the surface of the specimen, and a receptor located to receive the required pyramid of rays reflected by the specimen. The receptor shall be a photosensitive device responding to visible radiation.

6.2 Geometric Conditions—The axis of the incident beam shall be at one of the specified angles from the perpendicular to the specimen surface. The axis of the receptor shall be at the mirror reflection of the axis of the incident beam. The axis of the incident beam and the axis of the receptor shall be within 0.1° of the nominal value indicated by the geometry. With a flat piece of polished black glass or other front-surface mirror in the specimen position, an image of the source shall be formed at the center of the receptor field stop (receptor window). The length of the illuminated area of the specimen shall be not more than one third of the distance from the center of this area to the receptor field stop. The dimensions and tolerance of the source and receptor shall be as indicated in Table 1. The angular dimensions of the receptor field stop are measured from the receptor lens in a collimated-beam-type instrument, as illustrated in Fig. 1, and from the

⁶ A list of manufacturers of glossmeters can be obtained from ASTM Headquarters.

test surface in a converging-beam-type instrument, as illustrated in Fig. 2. See Figs. 1 and 2 for a generalized illustration of the dimensions. The tolerances are chosen so that errors in the source and receptor apertures do not produce an error of more than one gloss unit at any point on the scale (5).

6.2.1 The important geometric dimensions of any specular-gloss measurement are:

6.2.1.1 Beam axis angle(s), usually 60° , 20° , or 85° .

6.2.1.2 Accepted angular divergences from principal rays (degree of spreading or diffusion of the reflected beam).

NOTE 1—The parallel-beam glossmeters possess the better uniformity of principle-ray angle of reflection, but the converging-beam glossmeters possess the better uniformity in extent of angular divergence accepted for measurement.

NOTE 2—*Polarization*—An evaluation of the impact of polarization on gloss measurement has been reported (11). The magnitude of the polarization error depends on the difference between the refractive indices of specimen and standard, the angle of incidence, and the degree of polarization. Because the specimen and standard are generally quite similar optically, measured gloss values are little affected by polarization.

6.3 Vignetting—There shall be no vignetting of rays that lie within the field angles specified in Table 1.

6.4 Spectral Conditions—Results should not differ significantly from those obtained with a source-filter photocell combination that is spectrally corrected to yield CIE luminous efficiency with CIE source C. Since specular reflection is, in general, spectrally nonselective, spectral corrections need to be applied only to highly chromatic, low-gloss specimens upon agreement of users of this test method.

6.5 Measurement Mechanism—The receptor-measurement mechanism shall give a numerical indication that is proportional to the light flux passing the receptor field stop with $\pm 1\%$ of full-scale reading.

7. Reference Standards

7.1 Primary Standards—Highly polished, plane, black glass of refractive index of 1.567 for the sodium D line shall be assigned a specular gloss value of 100 for each geometry. The gloss value for glass of any other refractive index shall be computed from the Fresnel equation (5). For small differences in refractive index, however, the gloss value is a linear function of index, but the rate of change of gloss

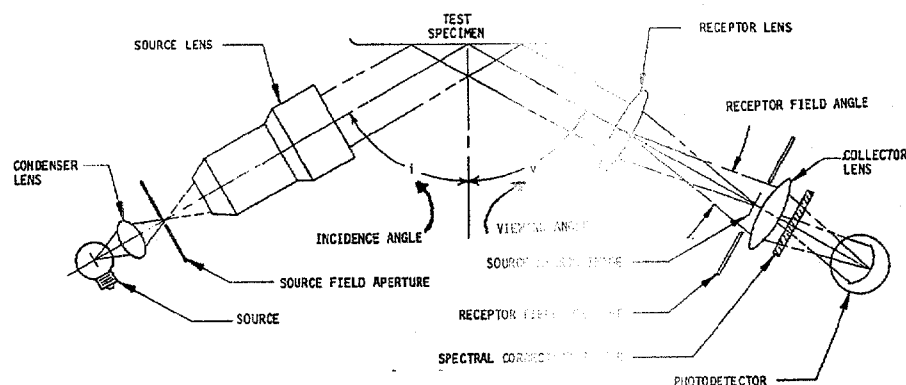


FIG. 1 Diagram of Parallel-Beam Glossmeter Showing Angular and Source Mirror-Image Position

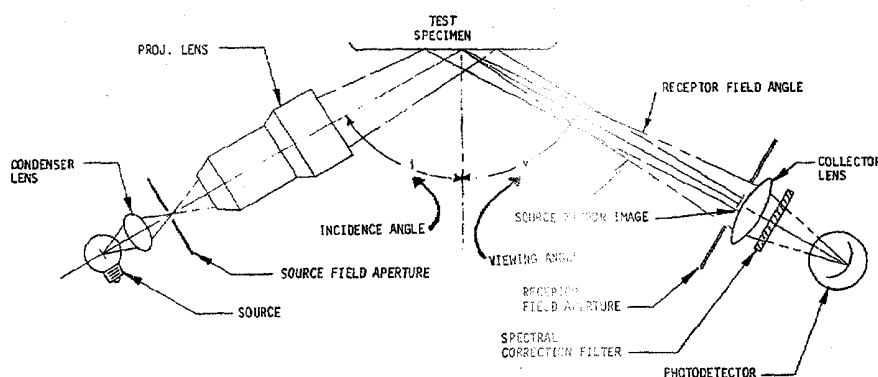


FIG. 2 Diagram of Converging-Beam Glossmeter Showing Apertures and Source Mirror-Image Position

with index is different for each geometry. Each 0.001 increment in refractive index produces a change of 0.27, 0.16, and 0.016 in the gloss value assigned to a polished standard for the 20, 60, and 85° geometries, respectively. For example, glass of index 1.527 would be assigned values of 89.2, 93.6, and 99.4, in order of increasing geometry.

NOTE 3—Polished black glass has been reported to change in refractive index with time largely due to chemical contamination (10). The original values can be restored by optical polishing with cerium oxide. A wedge of high-purity quartz provides a more stable reference standard than glass.

7.2 *Working Standards*⁷—Ceramic tile, depolished ground opaque glass, emery paper, and other semigloss materials having hard and uniform surfaces are suitable when calibrated against a primary standard on a glossmeter known to meet the requirements of this test method. Such standards should be checked periodically for constancy by comparing with primary standards.

7.3 *Store standards* in a closed container when not in use. Keep them clean and away from any dirt that might scratch or mar their surfaces. *Never* place standards face down on a surface that may be dirty or abrasive. Always hold standards at the side edges to avoid getting oil from the skin on the standard surface. Clean the standards in warm water and a mild detergent solution brushing gently with a soft nylon brush. (Do not use soap solutions to clean standards, because they can leave a film.) Rinse standards in hot running water (temperature near 150°F (65°C)) to remove detergent solution, followed by a final rinse in distilled water. *Do not wipe standards.* The polished black glass high-gloss standard may be dabbed gently with a lint-free paper towel or other lint-free absorbent material. Place the rinsed standards in a warm oven to dry.

8. Preparation and Selection of Test Specimens

8.1 This test method does not cover preparation techniques. Whenever a test for gloss requires the preparation of test specimens, use the procedures given in Test Methods D 823.

⁷ Gloss standards are available from Byk/Gardner, 2435 Linden Lane, Silver Spring, MD 20910 and Hunter Associates Laboratory, Inc., 11495 Sunset Hills Road, Reston, VA 22090.

NOTE 4—To determine the maximum gloss obtainable from a test material, such as a paint or varnish, use Methods B or C of Test Methods D 423.

8.2 Select specimens in accordance with Practice D 3964.

9. Instrument Calibration

9.1 Operate the glossmeter in accordance with the manufacturer's instructions.

9.2 Verify the instrument zero by placing a black cavity in the specified position. If the reading is not within ± 0.1 of zero, adjust it algebraically from subsequent readings or adjust the instrument to read zero.

9.3 Calibrate the instrument at the start and completion of every period of glossmeter operation, and during the operation at sufficiently frequent intervals to assure that the instrument response is practically constant. To calibrate, adjust the instrument to read correctly the gloss of a highly polished standard, properly positioned and oriented, and then read the gloss of a working standard in the mid-gloss range. If the instrument reading for the second standard does not agree within one unit of its assigned values, check cleanliness and repeat. If the instrument reading for the second standard still does not agree within one unit of its assigned value, repeat with another mid-range standard. If the discrepancy is still more than one unit, do not use the instrument without readjustment, preferably by the manufacturer.

10. Procedure

10.1 Position each specimen in turn beneath (or on) the glossmeter. If specimens have brush marks or similar texture marks, place them in such a way that the directions of the marks are parallel to the plane of the axes of the incident and reflected beams.

10.2 Obtain at least three readings on a 3 by 6-in. (75 by 150-mm) area of the test specimen. If the range is greater than 10 gloss units, take additional readings and calculate the average, discarding divergent results as in the section on Outliers of Practice D 3980. For larger specimens, take a proportionately greater number of readings.

11. Corrections

11.1 Apply diffuse corrections only upon agreement between the producer and the user. To apply the correction

subtract it from the glossmeter reading. To measure the correction, illuminate the specimen perpendicularly and view at the incident angle with the receiver aperture specified in 6.2 for the corresponding geometry. To compute the correction, multiply the 45°, 0° directional reflectance of the specimen, determined in accordance with Test Method E 97, by the effective fraction of the luminous flux reflected by the perfect diffuse reflector and accepted by the receiver aperture. The luminous flux entering the receiver aperture from the perfect white diffusor would give the following gloss indications for each of the geometries:

Geometry, °	Gloss of Perfect White Diffuser
60	2.5
20	1.2
85	0.03

12. Report

12.1 Report the information following:

12.1.1 Mean specular gloss readings and the geometry used.

12.1.2 If uniformity of surface is of interest, the presence of any specimen that exhibits gloss readings varying by more than 5 % from their mean.

12.1.3 Where preparation of the test specimen has been necessary, a description or identification of the method of preparation.

12.1.4 Manufacturer's name and model designation of the glossmeter.

12.1.5 Working standard or standards of gloss used.

13. Precision

13.1 On the basis of studies of this test method by several laboratories in which single determinations were made on different days on several ceramic tiles and painted panels differing in visually perceived gloss, the pooled within-laboratory and between-laboratories standard deviations were found to be those shown in Table 2. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.1 **Repeatability**—Two results, each of which are single determinations obtained on the same specimen by the

TABLE 2 Standard Deviation of Gloss Determinations

Type of Gloss ^a	No. of Specimens	Degrees of Freedom		Standard Deviations	
		Within-Laboratory	Between-Laboratories	Within-Laboratory ^a	Between-Laboratories ^b
20°	4	40	34	0.4	1.2
60°	4	40	34	0.3	1.2
85°	2	16	6	0.2	0.6

Type of Gloss ^a	No. of Painted Panels	Degrees of Freedom		Standard Deviations	
		Within-Laboratory	Between-Laboratories	Within-Laboratory ^a	Between-Laboratories ^b
20°	8	80	72	0.6	2.2
60°	22	220	136	0.3	1.2
85°	5	48	18	0.3	2.4

^a Single determinations.

^b For means of three determinations.

TABLE 3 Maximum Acceptable Differences for Two Results

Type of Gloss ^a	Repeatability (Within Laboratories) ^a		Reproducibility (Between Laboratories) ^b	
	Ceramic Tiles	Painted Panels	Ceramic Tiles	Painted Panels
20°	1.1	1.7	3.5	6.4
60°	0.9	0.9	3.4	3.5
85°	0.6	0.8	2.0	7.2

^a Single determinations.

^b For means of three determinations.

same specimen should be considered suspect if they differ by more than the maximum acceptable differences given in Table 3.

13.1.2 **Reproducibility**—Two results, each the mean of three determinations, obtained on the same specimen by different laboratories should be considered suspect if they differ by more than the maximum acceptable differences given in Table 3. This does not include variability due to preparation of panels in different laboratories.

Non-ferrous metal types of paint, particularly semi-gloss, the measurement is affected by method of film preparation and drying conditions. The reproducibility of results from such materials may be poorer than the values given in Table 3.

REFERENCES

- (1) Hunter, R. S., "Methods of Determining Gloss," *Proceedings, ASTM*, Vol 36, 1936, Part II, p. 783. Also, *Journal of Research*, Nat. Bureau Standards, Vol 18, No. 1, January 1937, p. 19 (*Research Paper RP958*). Six somewhat different appearance attributes are shown to be variously associated with gloss. Therefore, as many as six different photometric scales may be required to handle all gloss measurement problems. (This paper is out of print).
- (2) Hunter, R. S., and Judd, D. B., "Development of a Method of Classifying Paint According to Gloss," *ASTM Bulletin*, No. 97, March 1939, p. 11. A comparison is made of several geometrically different photometric scales for separating paint finishes for gloss. The geometric conditions of test later incorporated in Test Method D 523 are recommended.
- (3) Wetlaufer, L. A., and Scott, W. E., "The Measurement of Gloss," *Industrial and Engineering Chemistry, Analytical Edition*, Vol 12, November 1940, p. 647. A goniophotometric study of a number of paint finishes illuminated at 45°; a study of gloss readings affected by variation of aperture for 45 and 60° incidence.
- (4) Hunter, R. S., "The Gloss Measurement of Paint Finishes," *ASTM Bulletin*, No. 139, January 1948, p. 72. History of Test Method D 523.
- (5) Hunter, R. S., K. L., III, and Nimeroff, I., "Measurement of Specular Gloss," *Journal of Research*, Nat. Bureau Standards, Vol 57, 1952, p. 1. A study of the effect of aperture variation on glossmeter readings, including definitions of terms used in connection with specular gloss measurement, the Fresnel equation in a form readily used for calculation, and the deviation of diffuse correction factor.
- (6) Hunter, R. S., "Gloss Evaluation of Materials," *ASTM Bulletin*, No. 100, December 1952, p. 48. A study of the history of gloss measurement, ASTM and other societies, describing the background and the geometry of these methods. Contains photographs showing the characteristics of a variety of methods.
- (7) Hunter, R. S., *The Measurement of Appearance*, Wiley-Interscience, New York, 1975, Chapter 6, "Scales for Gloss and

Other Geometric Attributes," and Chapter 13, "Instruments for the Geometric Attributes of Object Appearance."

- (8) Horning, S. C., and Morse, M. P., "Measurement of the Gloss of Paint Panels," *Official Digest*, Federation of Paint and Varnish Production Clubs, March 1947, p. 153. A study of the effect of geometric conditions on results of gloss tests with special attention to high-gloss panels.
- (9) Huey, S., Hunter, R. S., Schreckendgust, J. G., and Hammond, H. K., III, "Symposium on Gloss Measurement," *Official Digest*, Vol 36, No. 471, April 1964, p. 343. Contains discussion of industrial experience in measurement of 60° specular gloss (Huey), high-gloss

measurement (Schreckendgust), evaluation of low-gloss finishes with 85° sheen (Hammond), and gloss standards and glossmeter calibration (Hammond).

- (10) Billmeyer, F. W., and O'Donnell, F. X. D., "Visual Gloss Scaling and Methodology and Feeling Analysis of Painted Specimens," *Color Research and Application*, Vol 12, 1987, pp 315-326. Compares visual gloss ratings with instrumental measurements of gloss, color, distinctness of image gloss, and haze for series of black, gray, and white painted specimens. The data are analyzed by multidimensional scaling.
- (11) Budde, W., "Industry Problems in Gloss Measurements," *Journal of Coatings Technology*, Vol 52, June 1980, pp. 44-48.

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Standard Test Method for Consistency of Paints Using the Stormer Viscometer¹

This standard is issued under the fixed designation D 562; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense pursuant to the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Editorial changes were made throughout in May 1990.

1. Scope

1.1 This test method covers the determination of the consistency of paints and related coatings using the Stormer viscometer.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

- 2.1 *ASTM Standard:*
E 1 Specification for ASTM Thermometers²

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *consistency*—load in grams to produce a rotational frequency of 200 r/min.

3.1.2 *Krebs units (KU)*—values of a scale commonly used to express the consistency of paints generally applied by brush or roller. This scale is a log function of the “load to produce 200-r/min” scale.

4. Summary of Test Method

4.1 The load required to produce a rotational frequency of 200 r/min for an offset paddle rotor immersed in a paint is determined.

5. Significance and Use

5.1 This test method provides values that are useful in specifying and controlling the consistency of paints, such as consumer or trade sales products.

6. Apparatus

6.1 *Viscometer*,³ Stormer, with the paddle-type rotor as illustrated in Figs. 1 and 2. The stroboscopic timer attachment in Fig. 1 can be removed and the instrument used without it but with a sacrifice of speed and accuracy. The

stroboscopic timer gives the 200 r/min reading directly.

6.2 *Container*—1-pt (500-mL), 3 3/8 in. (85 mm) in diameter.

6.3 *Thermometer*—An ASTM Stormer Viscosity thermometer with a range from 20 to 70°C and conforming to the requirements of Thermometer 49C, as prescribed in Specification E 1.

6.4 *Stopwatch*—A suitable timer measuring to 0.2 s.

6.5 *Test Oils*—A set covering the range from 5 to 1000 g.

7. Test Procedure

7.1 *Test Oils*—Standard oils, calibrated in absolute viscosity (poises), that fall within the viscosity range of the coatings to be measured. These oils should differ in viscosity by at least 5 P.

NOTE 1—The pertinent range of the Stormer is covered by oils having viscosities of 4 P (40 KU), 10 P (85 KU), and 14 P (95 KU).

7.1.1 *Test Oils*—Standards are silicone, hydrocarbon, linseed, and castor oils. Silicone and hydrocarbon oils calibrated in poises are commercially available.⁴ Uncalibrated linseed and castor oils may be calibrated with any apparatus that reports measurements of absolute viscosity.⁵

7.1.2 *Conversion*—The load to produce 200 r/min to each oil bearing the viscosity value in poises to load in grams by the following equation:⁶

$$L = (610\eta + 906.6\rho)/30$$

where

η = viscosity of oil in poises and

ρ = density of oil in g/cm³.

8. Test Results

8.1 *Procedure*—Remove rotor and weight carrier from the viscometer. Wind the string is wound evenly on the drum and the rotor is set up itself.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 562 - 47 T. Last previous edition D 562 - 55 (1976).

² Annual Book of ASTM Standards, Vols 05.03 and 14.01.

³ The viscometer may be obtained from Arthur H. Thomas Co., Vine Street and 15th St., Philadelphia, PA 19105; Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219; or Byk-Gardner, Inc., Gardner Laboratory, 2435 Mount Airy Road, Gaithersburg, MD 20878.

⁴ Calibration of test oils are available from the Cannon Instrument Co., P. O. Box 100, Uxbridge, MA 01561. Silicone oils are available from the Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

⁵ Available from various oil suppliers.

⁶ Corbett, J. A., and Peterson, D. H., “Calculation of Viscosity From Stormer Viscosity Data,” *Journal of Applied Engineering Chemistry*, Vol 34, 1942, p. 163.

⁷ Jackson, G. W., and Pearson, W. H., “A Method for the Standardization of Krebs Units of Stormer Viscometers,” *ASTM Bulletin*, No. 161, 1949.

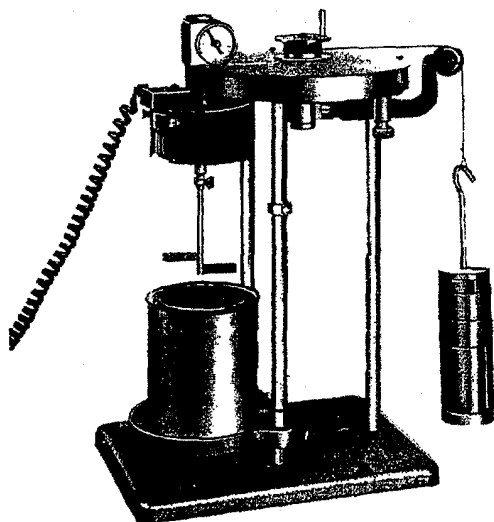


FIG. 1 Stormer Viscometer with Paddle-Type Rotor and Stroboscopic Timer

8.2 Attach a 5-g weight onto the string and then release the brake. If the viscometer starts to run from this dead start and continues to run through several revolutions of the string drum, it is satisfactory for use. If it does not start unaided when the 5-g weight is applied, the instrument should be reconditioned.

8.3 Check the dimensions of the paddle-type rotor. They should be within ± 0.004 in. (0.1 mm) of the dimensions shown in Fig. 2.

8.4 Select two standard oils having assigned values of load to produce 200 r/min within the range of the values expected for the coatings to be measured (see 7.1).

8.5 Adjust the temperature of the standard oils to $25 \pm 0.2^\circ\text{C}$. The temperature of the Stormer apparatus should be the same. If the specified temperature cannot be obtained, record the temperature of the oil at the beginning and end of test to 0.2°C .

8.6 Determine the load in grams to produce 200 r/min with each of the two oils, using either Procedure A described in Section 9 or Procedure B described in Section 10.

8.6.1 If the oil temperature was not at $25 \pm 0.2^\circ\text{C}$ during the test, correct the measured load in grams for the deviation from that temperature.

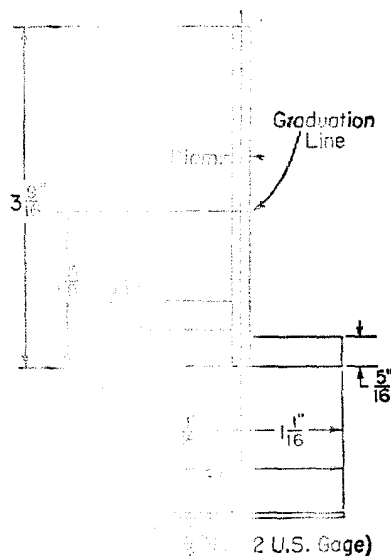
NOTE 2—Load corrections for deviations of oil temperature from the specified temperature can be made by means of a previously established plot of load versus oil temperature (see Appendix X1).

8.7 If the measured load (corrected for any temperature deviation from standard) is within $\pm 15\%$ of the assigned load values for the oils, the Stormer apparatus can be considered to be in satisfactory calibration.

9. Procedure A (Without Stroboscopic Attachment)

9.1 Thoroughly mix the sample and strain it into a 1-pt (500-mL) container to within $\frac{1}{4}$ in. (20 mm) of the top.

9.2 Bring the temperature of the specimen to $25 \pm 0.2^\circ\text{C}$ and maintain it at that temperature during the test. The



All dimensions shall be to a tolerance of ± 0.004 in. (0.1 mm).

FIG. 2 Paddle-Type Rotor for Use With Stormer Viscometer

temperature of the specimen and apparatus should be the same.

9.2.1 If the specified temperature cannot be obtained, record the temperature of the specimen at the beginning and end of test to 0.2°C .

9.3 When the temperature of the specimen has reached equilibrium, start the test by placing the specimen on the platform of the viscometer immediately on the paddle-type rotor is immersed in the specimen to the mark on the shaft of the rotor.

9.4 Place a weight on the spindle of the viscometer and determine a load that will produce 100 revolutions in the range of 25 to 35 s.

9.5 Using the load determined in 9.4, select two loads that will produce 100 revolutions (time to give 100 revolutions) within the range of 27 to 33 s. Make these measurements at the beginning and end of the test, that is, permit the rotor to make an additional 100 revolutions before starting the timing for 100 revolutions.

9.6 Repeat the test as described in 9.5 until two readings for each load are obtained that differ within 0.5 s.

10. Procedure B (With Stroboscopic Timer)

10.1 Follow the steps in Section 9 (to 9.3) for the preparation of the specimen.

10.2 Connect the stroboscopic attachment of the viscometer to a stroboscopic timer.

10.3 Place a weight on the spindle of the viscometer and determine a load that will produce 100 revolutions in the range from 25 to 35 s.

10.4 Using the load determined in 10.3, select a weight (to the nearest 0.1 g) that will produce the 200-r/min pattern

TABLE 1 Krebs' Stormer Chart with Interpolations

Seconds for 100 Revolu- tions	Load, g																				Krebs Units																			
	75	100	125	150	175	200	225	250	275	300	325	350	375	400	425	450	475	500	525	550	575	600	625	650	675	700	725	750	775	800	825	850	875	900	950	1000				
1.5	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50		
1.0	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51		
0.5	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52		
0.2	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53		
0.1	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54		
0.05	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55		
0.02	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56		
0.01	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57		

(Fig. 3) on the stroboscopic timer, that is, where the lines appear to be stationary.

10.4.1 Lines moving in the direction of paddle rotation indicate a speed greater than 200 r/min and therefore, weight should be removed from the hanger. Conversely, lines moving opposite to direction of paddle rotation indicate a speed less than 200 r/min and weight should be added.

NOTE 3—There are other patterns that appear at speeds other than 200 r/min (See Fig. 4). The pattern for 200 r/min should be determined before running any tests.

10.5 Repeat the determination in 10.4 until a consistent value of load is obtained (that is, to within 5 g).

11. Calculation

11.1 Procedure A:

11.1.1 Calculate the load to within 5 g, to produce 100 revolutions in 30 s by interpolating between the load weights recorded for the readings made between 27 and 33 s for 100 revolutions.

11.1.2 Correct the load determined for any deviation of the specimen temperature from the specified temperature (see Appendix X1).

11.1.3 If desired, determine from Table 1 the KU corresponding to the load to produce 100 revolutions in 30 s.

NOTE 4—Table 1 has been constructed so that it is not necessary to interpolate between loads to obtain the KU corresponding to the load to produce 100 revolutions in 30 s. The table provides KU values computed for a range of 27 to 33 s for 100 revolutions.

11.2 Procedure B:

11.2.1 If desired, determine from Table 2 the KU value corresponding to the load to produce 200 r/min.



FIG. 3 Stroboscopic Lines Opening When Timer is Adjusted to Exactly 200 r/min

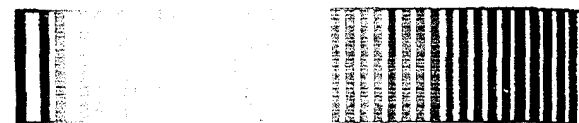


FIG. 4 Stroboscopic Lines Appearing as Multiples that May be Reached at 200 r/min Reached

12. Report

12.1 Report the following information:

12.1.1 The load in grams to produce 200 r/min (100 revolutions in 30 s).

12.1.2 The specimen temperature.

12.1.3 The specimen temperature of the specimen during the test and whether the specimen was corrected for any deviation from 25°C.

12.1.4 The KU value if Procedure A or Procedure B was used.

13. Precision

13.1 On the basis of the study in which determinations were made on three solvent-reducible, two water-reducible, and two non-reducible materials at each of six laboratories on each of two different days, the within-laboratory coefficient of variation was found to be 1 % in load grams or 1.5 % in KU, and the between-laboratory coefficient of variation was found to be 1 % in load grams or 4 % in KU.

13.2 On the basis of the above coefficients, the following criteria should be used to determine the acceptability of results at the 95 % confidence level.

13.2.1 If two results are obtained on the same material, the difference between the two test times should be considered unacceptable if it is more than 9 % in load grams or 12 % in KU.

13.2.2 If four or more results, each the mean of four individual results, are obtained on the same material, the difference between the mean of four individual results should be considered suspicious if it is more than 3 % in load grams or 12 % in KU.

14. Keywords

14.1 This standard covers KU; Stormer viscometer; viscometer.

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85 57

90 58
95 60

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TABLE 2 Krebs Units Corresponding to Load Values (For use with Stormer Viscometer)

Grams KU	Grams KU	Grams KU	Grams KU	Grams KU	Grams KU	Grams KU	Grams KU	Grams KU	Grams KU	Grams KU
	100 61 105 62	200 82 205 83	300 95	400 104	500 112	600 120	700 128	800 131	900 136	1000 140
	110 63 115 64	210 83 215 84	310 96	410 105	510 113	610 121	710 129	810 132	910 136	1010 140
	120 65 125 67	220 85 225 86	320 97	420 106	520 114	620 122	720 130	820 133	920 137	1020 140
	130 68 135 69	230 86 235 87	330 98	430 106	530 114	630 122	730 130	830 133	930 137	1030 140
	140 70 145 71	240 88 245 88	340 99	440 107	540 115	640 123	740 131	840 133	940 138	1040 140
	150 72 155 73	250 89 255 90	350 100	450 108	550 116	650 124	750 132	850 134	950 138	1050 141
	160 74 165 75	260 90 265 91	360 101	460 109	560 117	660 125	760 133	860 135	960 138	1060 141
	170 76 175 77	270 91 275 92	370 102	470 110	570 118	670 126	770 134	870 135	970 139	1070 141
	180 78 185 79	280 93 285 93	380 102	480 110	580 118	680 126	780 134	880 135	980 139	1080 141
	190 80 195 81	290 94 295 94	390 103	490 111	590 119	690 127	790 135	890 136	990 140	1090 141

APPENDIX

(Nonmandatory Test Method)

X1. EFFECT OF SPECIMEN TEMPERATURE ON STORMER CONSISTENCY

X1.1 For maximum accuracy in determining the effect of specimen temperature on Stormer consistency, measurements should be performed at three different specimen temperatures covering the range of interest. The change in load or KU per 1°C change can be determined from these results.

X1.2 It has been observed that the consistency of an oil is considerably more sensitive to temperature than is the consistency of a paint.

X1.3 Some typical effects of temperatures on the consistency of oil and paint are given below:

temperatures are given below:

	Mean Value at 25°C		Change per 1°C Change	
	Load, g	KU value	Load, g	KU value
High consistency oil	149	72	14	2.5
Medium consistency oil	217	85	18	2.0
Low consistency oil	286	93	11	1.5
High consistency paint	195	81	8	1.0
Medium consistency paint	440	108	40	2.0
Low consistency paint	300	95	4	0.5
Very low consistency paint	425	105	4	0.5

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Standard Practice for Preparation of Cold-Rolled Steel Panels for Painting with Varnish, Conversion Coatings, and Related Coating Products¹

This standard is issued under the fixed designation D 609; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of subsequent superscript, epsilon (ε) indicates an editorial change since the last revision or approval.

1. Scope

1.1 This practice covers various types of cold rolled steel panels and the procedures to be followed in their preparation for testing paint, varnish, lacquer, conversion coatings, and related products.

1.2 The procedures are as follows:

Procedure A—Conversion coatings (phosphates, chromates, etc.)

Procedure B—Vapor degreasing

Procedure C—Solvent brushing

Procedure D—Solvent wiping

Procedure E—Alkaline cleaning

1.3 This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

A 109 Specification for Steel, Carbon, Cold-Rolled Strip²

A 366/A 366M Specification for Steel, Carbon, Cold-Rolled Sheet, Commercial Quality²

**D 235 Specification for Mineral Spirits (Petroleum Spirits)
(Hydrocarbon Drycleaning Solvents)³**

3. Summary of Practice

3.1 Several procedures for preparation and cleaning of steel test panels are described as shown in Table 1.

4. Significance and Use

4.1 The procedures described in this practice are designed to provide steel panels with a uniform and reproducible surface for testing of paint, varnish, lacquer, conversion coatings, and related products.

5. Test Panels

5.1 Prepare the test panels from rust and stain-free cold-rolled steel as described in 5.2, 5.3, 5.4, and Table 1. The panels shall be made to a size and thickness as agreed upon between the purchaser and seller. Edges shall be

smooth and uniform. The steel may have been coated with a suitable rust preventive compound for protection during transport and storage. However, long-term storage of the steel on the surface may cause an oil/solvent stain or a rusted stain. Since such stains inhibit chemical treatments, the steel selected for panels shall be free of oil stains and shall be free of processing variation caused by pickling.

5.2.2.2. *Steel mill finish*—The metallic finish produced by steel mill rolls has a grainy, crystalline texture. This finish is typical of cold-rolled steel and is often painted surfaces on automobiles, appliances, and other products. It can be partially characterized by measuring the average peak-to-valley distance and the number of peaks per inch. However, conversion coating and treatment of these surfaces may vary because of differences in grain size, grain boundaries, and surface condition.

also be polished (or "ground") after the last pass of the mill. Flat polishing is accomplished by rubbing the surface with a fine, flat, polished surface and provide a smooth surface for testing. Flat polishing is a derivative of the surface on which the specimen is polished significantly improves the performance of certain types of surface stress measurement. The nature of the steel surface must be free of any level of surface contamination that is completely removed as described in the following section. In any case, a minimum of 0.001 in. of surface must be removed. Care must be taken to ensure that the polishing apparatus and after cleaning the specimen is complete removal of grit and debris from the polished surface.

smooth finish produced by the sanding process. This smooth finish has the properties such as low surface roughness, which is desirable to prevent corrosion.

When the film is removed from the panel, it should be washed in running water for 10 minutes by immersing a 10" x 10" piece of film in a jar of distilled water. The film should be dried by hanging it over the edge of a sink and allowing it to dry in the open air. Do not use heat, blower, or desiccants or other drying agents.

1. **Identify the conversion cost.** The conversion cost is the cost of converting raw materials into finished goods. It is the cost of the process that transforms the raw materials into the final product.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct-responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Aug. 24, 1990. Published November 1990. Originally published as D 609 - 41. Last previous edition D 609 - 73 (1980)

² Annual Book of ASTM Standards, Vol 01.03.

³ *Annual Book of ASTM Standards*, Vols 05.01 and 06.03.



TABLE 1

NOTE— μ in. = microinches; nm = nanometer.

Type No.	Description	Thickness, in. (mm)	Mean	ASTM Specification
1	Cold-rolled steel, as rolled (matte)	0.02 to 0.038 (0.6 to 1.0)	100–150	A 109, A 366
2	Cold-rolled steel, flat-polished one side	0.024 to 0.038 (0.6 to 1.0)	100–150	A 109, A 366
3	Cold-rolled steel, as rolled (smooth)	0.010 to 0.024 (0.25 to 0.6)	100–150	A 109, A 366

manufacturer's directions as to the application of the conversion coating. Preparation of test panels may consist of one or more steps of cleaning, rinsing, or conditioning prior to the application of the conversion coating. Additional rinsing with water will usually be required after the conversion coating is applied. To prevent rust, force dry immediately after rinsing.

6.3 Procedure B: Vapor Degreasing—Suspend the panels in a vapor-phase type degreaser containing stabilized 1,1,1-trichloroethane and allow them to remain above the boiling solvent. When the panels attain vapor temperature, condensation of vapor onto the panels no longer occurs. Prewiping with a clean lintless cloth saturated with mineral spirits may be necessary, since vapor degreasing generally does not remove solid particulate matter such as dirt, etc. (see Note).

NOTE—Other organic solvents or combinations of solvents that will produce a water-break free surface may be used if agreed upon between the purchaser and the supplier.

6.4 Procedure C: Solvent Brushing—Power-brush scrub the panel with mineral spirits (Specification D 235). When all soluble and loosely adhering soil has been washed off, flush with clean mineral spirits. Dry at a temperature of 125

°F (50°C) for one hour (see Note).

6.5 Procedure D: Solvent Wiping—Using clean lintless cloth, wipe the panel with mineral spirits, thoroughly rub the panel with a clean lintless cloth, ensuring soil has been removed. Store at a temperature of 125°F (50°C) for one hour (see Note).

6.6 Procedure E: Aqueous Alkaline Cleaning—Use aqueous alkaline cleaning solutions as appropriate. Clean the panels with an aqueous alkaline solution or immersion. Follow the recommendations of the manufacturer of the rinsing with water. Rinse the panels with clean water. Dry the panels at 125°F (50°C) for one hour (see Note).

Do not use panels that are not to be used for testing. Do not use panels impregnated with oil or grease. Do not use panels impregnated with volatile organic compounds. Do not use panels impregnated with waterproof bag or other material. Do not use panels impregnated with fingerprints, do not use panels impregnated with fingerprints. Handle the panels with clean hands and clean gloves.

The American Society for Testing and Materials takes no position regarding the validity of claims for patents or for the right to make or use any process, product, or apparatus mentioned in this standard. Users of this standard are advised that the validity of such claims is not the responsibility of the Society, and the risk of infringement of such rights, are entirely those of the user.

This standard is subject to revision at any time by the responsible technical committee. If not revised, either reapproved or withdrawn. Your comments are invited. Comments should be addressed to ASTM Headquarters. Your comments will be considered by the technical committee, which you may attend. If you feel that your comments are not fully represented in the views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Method for Evaluating Degree of Rusting on Paint

This standard is issued under the fixed designation D 610; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of supersession. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reissuance.

This method has been approved for use by agencies of the Department of Defense under Standard No. 141A. Consult the DoD Index of Specifications and Standards for the location of this standard within the Index and for any subsequent revision. Approved by the Department of Defense.

⁶¹ NOTE—Editorial changes were made throughout, including the title, in October.

1. Scope

1.1 This test method covers the evaluation of the degree of rusting on painted steel surfaces using visual standards. These visual standards² were developed in cooperation with the Steel Structures Painting Council (SSPC) to further standardization of methods.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 Adjunct:

D610 Degree of rust (four photos)²

3. Significance and Use

3.1 The amount of rusting beneath or through a paint film is a significant factor in determining whether a coating system should be repaired or replaced. This test method provides a standardized means for quantifying the amount of rust present.

4. Interferences

4.1 The colored photographic reference standards and the associated rust-grade scale cover only rusting not accompanied by blistering and evidenced by visible rust.

4.1.1 Rust blistering beneath paint may be graded using the same scale by assuming the rust was completely visible and noting that the rusting was rust blistering.

4.2 The use of reference standards²

4.2.1.1. The following must not be used:

4.2.1. The user may make accurate fault.

4. Iron is not needed for iron or not should

4.2.4 *Area and perimeter of a rectangle* The area of a rectangle is the product of its length and width. The perimeter of a rectangle is the sum of its four sides.

[illegible]

5. Procedure

5.3.1 The following information must be included in the geographic reference section of the report:

- (a) A detailed description of the area covered by the verbal description;

Note: The area under the curve of the exponential function $y = 2^x$ from $x = 0$ to $x = 10$ and the area under the curve of the exponential function $y = 2^x$ from $x = 0$ to $x = 100$ have the same value. The area under the curve of the exponential function $y = 2^x$ from $x = 0$ to $x = 100$ is the same as the area under the curve of the exponential function $y = 2^x$ from $x = 0$ to $x = 10$. The area under the curve of the exponential function $y = 2^x$ from $x = 0$ to $x = 10$ is the same as the area under the curve of the exponential function $y = 2^x$ from $x = 0$ to $x = 100$.

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5. The area required for the processing of the waste shall be based upon the quantity of waste to be processed and the type of processing equipment to be used.

5. The blistering was as if it were

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Painting.

This test method has been jointly approved by ASTM and the Steel Structures Painting Council.

Current edition approved Oct. 25, 1985. Published December 1985. Originally published as D 610 - 41. Last previous edition D 610 - 68 (1981).

² The colored photographic reference standards are available at a nominal cost from ASTM Headquarters (request Adjunct No. 12-406100-00), and from the Steel Structures Painting Council, 4400 5th Ave., Pittsburgh, PA 15213.

3 Coatings and Surface Treatments

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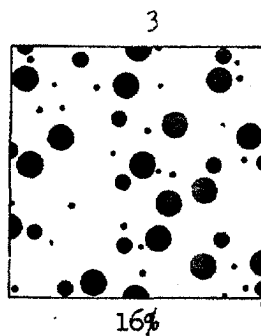
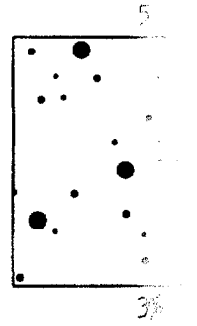
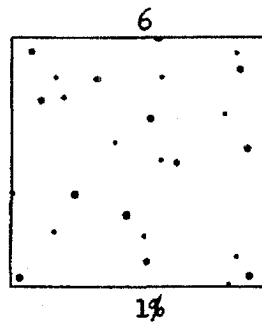
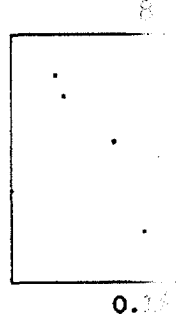
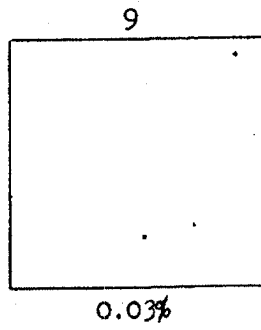


FIG. 1 Examples of

TABLE 1 Scale and Description of Rusting

Rust Grades ^A	Description	ASTM Scale ^B	Photomicrograph ^C
10	no rusting or less than 0.01 % of surface rusted	unrust	
9	minute rusting, less than 0.03 % of surface rusted	No. 1	
8 ^D	few isolated rust spots, less than 0.1 % of surface rusted	No. 2	
7	less than 0.3 % of surface rusted	non	
6 ^C	extensive rust spots but less than 1 % of surface rusted	No. 3	
5	rusting to the extent of 3 % of surface rusted	non	
4 ^D	rusting to the extent of 10 % of surface rusted	No. 4	
3 ^E	approximately one sixth of the surface rusted	non	
2	approximately one third of the surface rusted	non	
1	approximately one half of the surface rusted	non	
0 ^F	approximately 100 % of surface rusted	unrust	

^A Correspond to Swedish Pictorial Standards for Rusting (1955) (black and white).

^B Corresponds to SSPC Initial Surface Conditions E and British Iron and Steel Research Association.

^C Corresponds to SSPC Initial Surface Conditions F and BISRA 1.0 %.

^D Corresponds to SSPC Initial Surface Condition G.

^E Rust grades below 4 are of no practical importance in grading performances of paints.

^F Corresponds to SSPC Initial Surface Condition H.

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Standard Test Method for Abrasion Resistance of Organic Coatings¹ Abrasive¹

This standard is issued under the fixed designation D 658; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A superscript epsilon (ϵ) indicates an editorial change since the last revision or approval. A

1. Scope

1.1 This test method covers the determination of the resistance of organic coatings to abrasion produced by an air blast of abrasive on coatings applied to a plane, rigid surface, such as a metal or glass panel.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 968 Test Method for Abrasion Resistance of Organic Coatings by Falling Abrasive²
- D 1005 Test Methods for Measurement of Dry Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- E 11 Specification for Wire-Cloth Sieves For Testing Purposes³

3. Terminology

3.1 Description of Term Specific to This Standard:

3.1.1 *abrasion resistance*—the weight of abrasive required to wear through a unit film thickness of the coating.

4. Summary of Test Method

4.1 An abrasive under the action of a controlled air stream is allowed to impinge on a coated panel until the substrate becomes visible. The amount of abrasive used per unit film thickness is reported as the abrasion resistance of the coating.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Feb. 22, 1991. Published April 1991. Originally published as D 658 - 42 T. Last previous edition D 658 - 81(1986)¹.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.02.

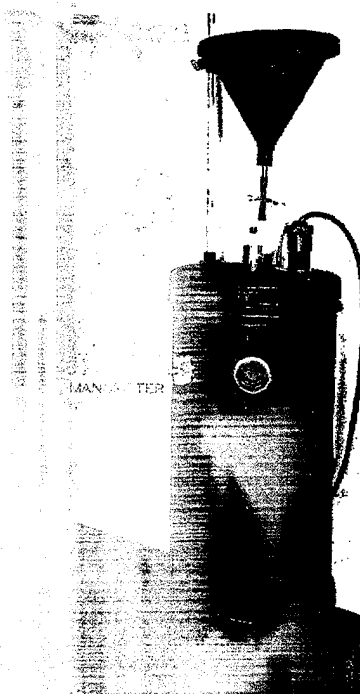


Fig. 1. Test apparatus (continued)

5. The test method described in Fig. 1 and Fig. 2 are two similar methods for determining the abrasion resistance of organic coatings. The test method described in Fig. 3 is a modification of the test method described in Fig. 1 and Fig. 2. The test method described in Fig. 3 is useful in the testing of coatings.

6. The test method described in Figs. 1 and 2 is a modification of the test method described in Fig. 3. The test method described in Fig. 3 is useful in the testing of coatings.

7. The test method described in Fig. 3 is a modification of the test method described in Fig. 1 and Fig. 2. The test method described in Fig. 3 is useful in the testing of coatings.

8. The test method described in Fig. 3 is a modification of the test method described in Fig. 1 and Fig. 2. The test method described in Fig. 3 is useful in the testing of coatings.

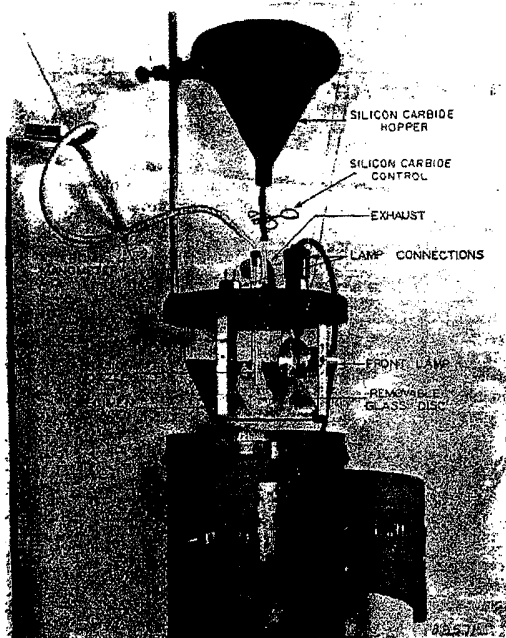


FIG. 2 Abrasion Test Apparatus (Open)

the holder. Insect screen (approximately 270 openings per square inch) is suitable for this purpose.

6.2 Air Supply, oil-free, under pressure, capable of maintaining a selected pressure within ± 200 Pa.

6.3 *Standard Abrasive*⁵—Silicon carbide grain passing a No. 170 (90- μ m) sieve and retained on a No. 200 (75- μ m) sieve.

NOTE 2—To obtain maximum accuracy and precision of results, the abrasive should be sieved for a minimum of ½ h with vigorous agitation, and only that portion which passes through a No. 170 (90-µm) sieve and is retained on a No. 200 (75-µm) sieve should be used in the test. The retained portion of the abrasive is considered satisfactory if a minimum of 85 % by weight meets the grain size requirements of 90 to 75 µm. Use the sieves described in Specification E 11.

7. Specimens

7.1 Apply uniform coatings of the material to be tested to a plane, rigid panel, such as metal or glass. Prepare a minimum of two coated panels for the material.

7.2 Cure the coated panels under conditions of humidity and temperature as agreed upon by the purchaser and seller.

NOTE 3—The coatings should be applied in accordance with Test Methods D 823, or as agreed upon between the purchaser and the seller.

NOTE 4—The thickness of the dry coatings should be measured in accordance with Test Methods D 1005, D 1400, or D 1186.

NOTE 5—For the apparatus shown in Figs. 1 and 2, the coated panels should not be larger than 5 by 4 by ¼ in. (125 by 100 by 6.3 mm) thick.

⁵ Suitable silicon carbide grain is Carborundum grain #180GG obtainable from the Carborundum Co., P. O. Box 423, Electro Minerals Div., Niagara Falls, NY 14302, or 180-mesh silicon carbide obtainable from City Chemical Corp., 132 W. 22nd St., New York, NY 10011. Other grades of abrasive may be used when mutually agreed upon between the purchaser and the seller.

8. Summary

8.1.4.1. The inner tube of the oil-free air supply shall be

8.2 [redacted] will provide
approval [redacted]

8.21 ± 2 g/min. The pressure in the pur-
chaser was 45 ± 2 kPa (100
mmHg).

Not surprisingly, the results showed that the higher the perceived usefulness of the system, the more likely the user was to use it. The results also showed that the higher the perceived ease of use of the system, the more likely the user was to use it. The results also showed that the higher the perceived ease of use of the system, the more likely the user was to use it.

8. The proposed changes to the procedure for the collection of the amount of the fine are aimed at the simplification of the procedure for the collection of the amount of the fine by allowing the collection of the amount of the fine in the amount of the fine.

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9. Conclusion

9. The buyer and seller shall be held to the terms of the contract as written and as amended by the buyer and seller at 23 ± 2°C and 50 ± 5% relative humidity in the same environment as the test material.

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10. Procedure

10.1 Place one test panel on the specimen stage. Pour a quantity of abrasive into the abrasive holder (funnel) of the apparatus. Lower the nozzle to touch the surface of the test specimen and close the door of the tester. Turn on the air supply and adjust it to the pressure reading established in the standardization of the apparatus.

10.2 Release the stream of abrasive by removing the pinchcock attached to the tubing, and allow to flow until the coating is worn through to the base metal. Consider the end point as having been reached when a small spot approximately 2 mm in diameter is worn through in the center of the wear pattern. When this point is reached, immediately shut off the abrasive stream and determine the weight of abrasive used.

NOTE 7—The amount of abrasive used can be determined either by (1) weighing the abrasive in the funnel before and after a run, or by (2) opening the bottom of the abrasion tester, collecting the abrasive used in a container, and weighing the abrasive.

10.3 Repeat 10.1 and 10.2 for two additional locations on the test specimen.

10.4 Measure the thickness of the coating adjacent to each wear pattern.

NOTE 8—The silicon carbide grain from previous tests may be reused, provided it is sieved through a No. 100 (150- μ m) sieve before it is returned to the holder in order to eliminate all of the large particles of the coating that chipped off during the abrasion test. The silicon carbide grain shall be replaced after it has been used five times.

10.5 Repeat 10.1 to 10.4 on at least one additional panel coated with the material under test.

11. Calculation

11.1 For each abrasion determination on a coated panel, calculate the abrasion resistance, A , in grams per mil as follows:

$$A = W/T$$

where:

W = weight of abrasive used, g (to one decimal place) and
 T = thickness of coating, mils (to one decimal place).

11.2 Calculate the mean of the abrasion resistance values

The American Society for Testing and Materials takes no position with any item mentioned in this standard. Users of this standard should be aware that some of the items may be the subject of patent rights, and the risk of infringement of such rights, are entirely their own.

This standard is subject to revision at any time by the responsible technical committee. It may be revised, amended, or withdrawn. Your comments are invited and should be addressed to ASTM Headquarters. Your comments will be considered for inclusion in a possible future revision of this standard. If you feel that your comments are not being given the proper consideration, please contact the responsible technical committee, which you may attend. If you feel that your views are not being given the proper consideration, please contact the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Method of Evaluating Degree of Chalking of Exterior Paints

This method covers the evaluation of the degree of chalking on white and light-colored exterior paints. It describes the procedure used to transfer to a test fabric the chalk which is then compared to a set of standard chalking test panels. Formerly under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, this method was discontinued in 1990 and replaced by ASTM Test Methods D 4214, for Chalking of Exterior Paints on Films.¹

¹ Annual Book of ASTM Standards, Vol 06.01.

1. Scope

1.1 The test method checks the degree of chalking on exterior paint films intended for use on exterior surfaces.

2. Referen

2.1 *ASTM D 661 Test Method for Evaluating Degree of Chalking of Exterior Paints*
Result

3. Definiti

3.1 *check* films by s through th sary in eva visible (as observed u

4. Signific

4.1 Since this method is an illustration of failure.

5. Types

5.1 Major types are:

5.1.1 *Irregular* breaks develop a pattern.

5.1.2 *Linear* the breaks are parallel lines.

5.1.3 *Surface* surface of

¹ This test method is part of the *Standard Test Methods for Evaluating Degree of Chalking of Exterior Paints*, published by the American Society for Testing and Materials, 11 West 42nd Street, New York, NY 10018-1500.
Committee D01.27
Current edition published as D

² Annual Book of ASTM Standards, Vol 06.01.



Standard Test Method for Evaluating Degree of Checking of Exterior Paints¹

This standard is issued under the fixed designation D 660; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A second number in parentheses indicates the year of last superscript epsilon (e) indicates an editorial change since the last revision.

This test method has been approved for use by agencies of the Department of Defense in accordance with the Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 The illustrated reference standards included in this test method are representative of degrees and types of checking of exterior paint films. These standards are primarily intended for comparative evaluation.

2. Referenced Documents

2.1 ASTM Standards:

- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²
- D 1150 Single and Multi-Panel Forms for Recording Results of Exposure Tests of Paints²

3. Definition

3.1 *checking*—that phenomenon manifested in paint films by slight breaks in the film that do not penetrate through the last applied coating. Where precision is necessary in evaluating a paint film, checking may be described as *visible* (as seen with the naked eye) or as *microscopic* (as observed under a magnification up to ten diameters).

4. Significance and Use

4.1 Since checking is not easily recognizable, this test method is intended to provide, through definition and illustrations, a means of evaluating the degree of this film failure.

5. Types of Checking

5.1 Many types of checking are recognized, of which some are:

5.1.1 *Irregular Pattern Type*—Checking in which the breaks develop in the surface of the film in no definite pattern.

5.1.2 *Line and Short Parallel Type*—Checking in which the breaks in the surface of the film are generally arranged in parallel lines.

5.1.3 *Switch Type*—Checking in which the breaks in the surface of the film form short and long irregular lines

or irregular patterns, resembling a cracked pattern.

5.1.4 *Three-Pronged Type*—Checking in which the breaks in the surface of the film form a definite three-pronged pattern, resembling a three-pronged fork or a three-pronged star.

5.1.5 *Geometric Pattern Type*—Checking in which the breaks in the surface of the film form definite geometric patterns, such as squares, rectangles, or circles.

5.1.6 *Short Break Type*—Checking in which the breaks in the surface of the film form individual short breaks, which may be arranged in a definite pattern.

5.1.7 *Long Break Type*—Checking in which breaks in the surface of the film form long, narrow patterns. These patterns may be arranged in a definite pattern.

5.1.8 *Oval Type*—Checking in which the breaks in the surface of the film form oval shapes.

5.1.9 *Irregular Pattern Type*—Checking in which the breaks in the surface of the film form an irregular pattern.

5.1.10 *Three-Pronged Type*—Checking in which the breaks in the surface of the film form a definite three-pronged pattern, resembling a three-pronged fork or a three-pronged star.

5.1.11 *Geometric Pattern Type*—Checking in which the breaks in the surface of the film form definite geometric patterns, such as squares, rectangles, or circles.

5.1.12 *Short Break Type*—Checking in which the breaks in the surface of the film form individual short breaks, which may be arranged in a definite pattern.

5.1.13 *Long Break Type*—Checking in which breaks in the surface of the film form long, narrow patterns. These patterns may be arranged in a definite pattern.

5.1.14 *Oval Type*—Checking in which the breaks in the surface of the film form oval shapes.

5.1.15 *Irregular Pattern Type*—Checking in which the breaks in the surface of the film form an irregular pattern.

5.1.16 *Three-Pronged Type*—Checking in which the breaks in the surface of the film form a definite three-pronged pattern, resembling a three-pronged fork or a three-pronged star.

5.1.17 *Geometric Pattern Type*—Checking in which the breaks in the surface of the film form definite geometric patterns, such as squares, rectangles, or circles.

5.1.18 *Short Break Type*—Checking in which the breaks in the surface of the film form individual short breaks, which may be arranged in a definite pattern.

5.1.19 *Long Break Type*—Checking in which breaks in the surface of the film form long, narrow patterns. These patterns may be arranged in a definite pattern.

5.1.20 *Oval Type*—Checking in which the breaks in the surface of the film form oval shapes.

5.1.21 *Irregular Pattern Type*—Checking in which the breaks in the surface of the film form an irregular pattern.

5.1.22 *Three-Pronged Type*—Checking in which the breaks in the surface of the film form a definite three-pronged pattern, resembling a three-pronged fork or a three-pronged star.

5.1.23 *Geometric Pattern Type*—Checking in which the breaks in the surface of the film form definite geometric patterns, such as squares, rectangles, or circles.

5.1.24 *Short Break Type*—Checking in which the breaks in the surface of the film form individual short breaks, which may be arranged in a definite pattern.

5.1.25 *Long Break Type*—Checking in which breaks in the surface of the film form long, narrow patterns. These patterns may be arranged in a definite pattern.

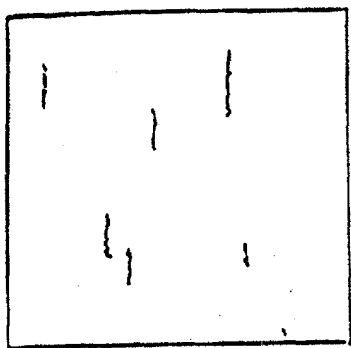
5.1.26 *Oval Type*—Checking in which the breaks in the surface of the film form oval shapes.

5.1.27 *Irregular Pattern Type*—Checking in which the breaks in the surface of the film form an irregular pattern.

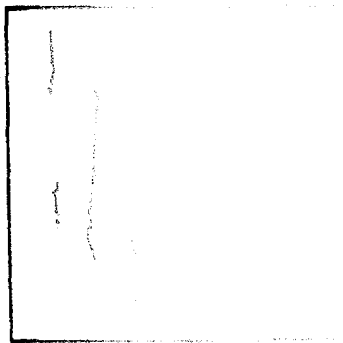
¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Oct. 19, 1987. Published December 1987. Originally published as D 660 - 44 T. Last previous edition D 660 - 44 (1981)^{e2}.

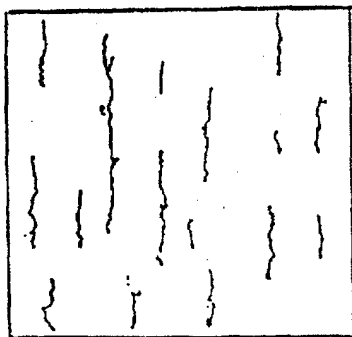
² Annual Book of ASTM Standards, Vol 06.01.



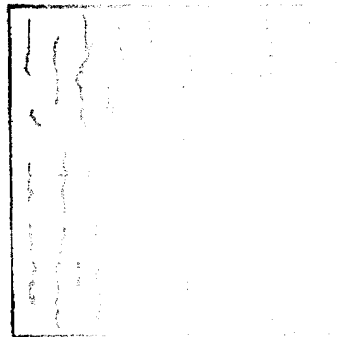
ASTM 8
TNO 2



ASTM 4
TNO 6

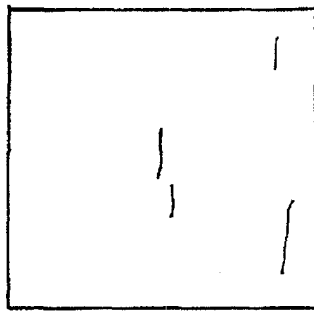


ASTM 8
TNO 2

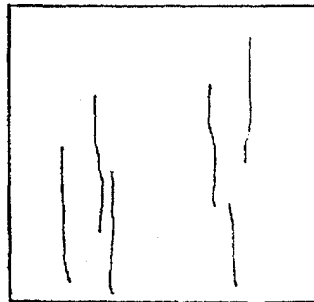


ASTM 4
TNO 6

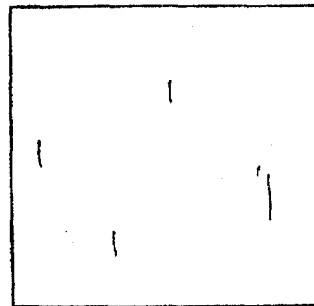
FIG. 1 Illustrated Reference



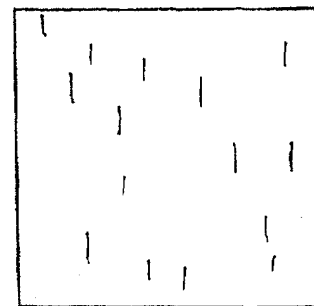
ASTM 8
TNO 2



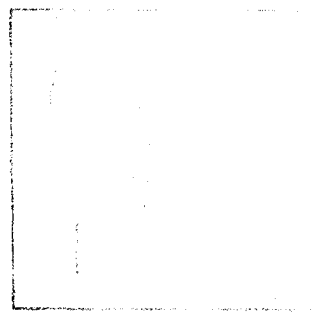
ASTM 4
TNO 6



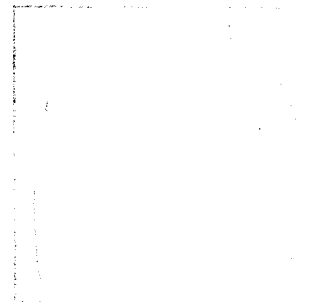
ASTM 8
TNO 2



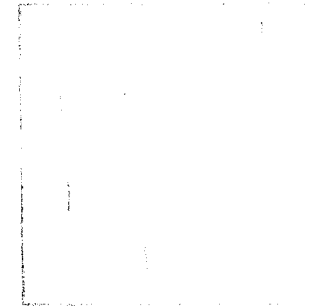
ASTM 4
TNO 6



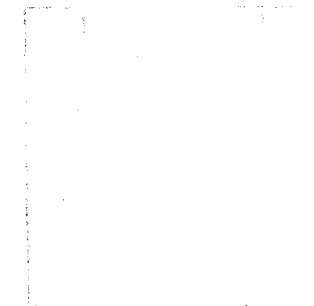
ASTM 8
TNO 2



ASTM 4
TNO 6



ASTM 8
TNO 2

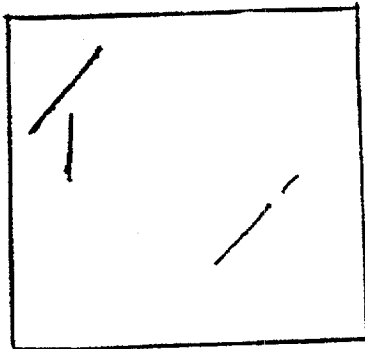


ASTM 4
TNO 6

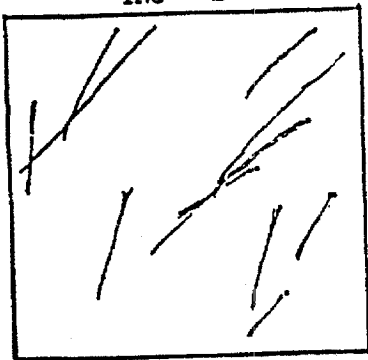
5.1.1 LONG
TANGENTIAL

5.1.2 SHORT
TANGENTIAL

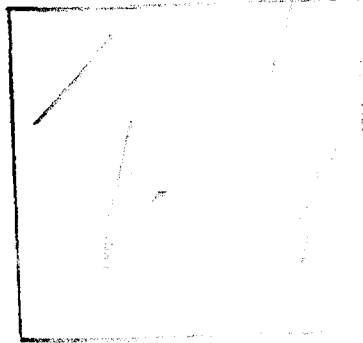
FIG. 1



ASTM 8
TNO 2



ASTM 4
TNO 6



3.1.3 SWITCH

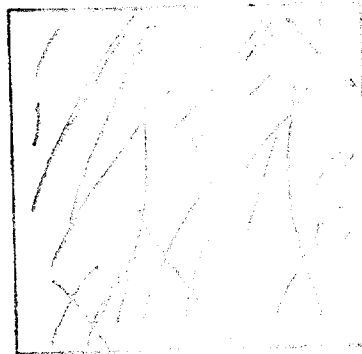
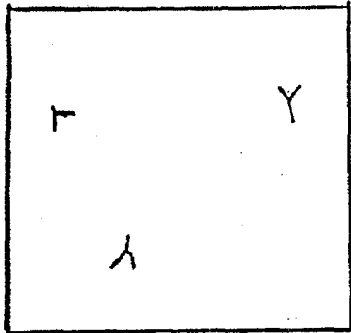
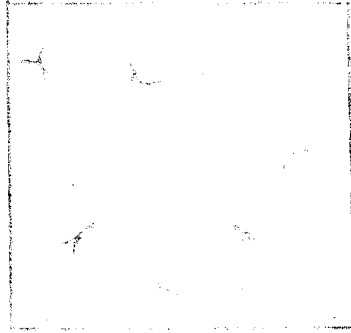


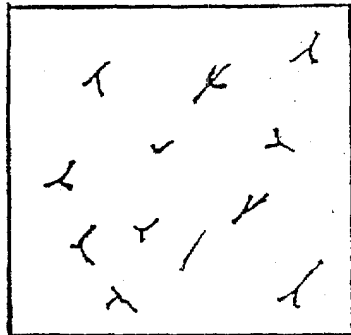
FIG. 1 (Continued)



ASTM 8
TNO 2



5.1.4 CROW FOOT



ASTM 4
TNO 6

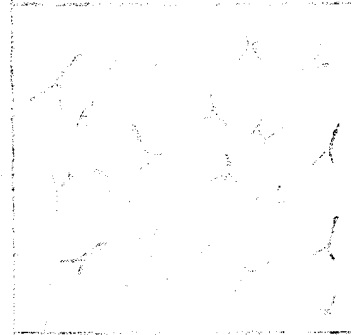
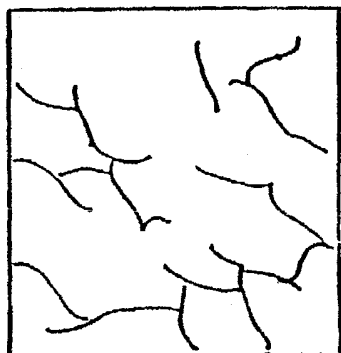


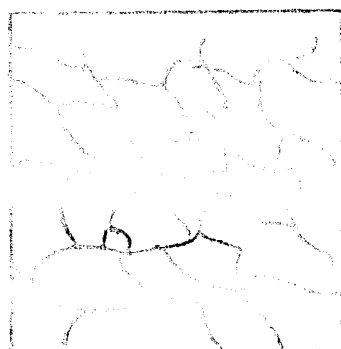
FIG. 1 (Continued)

ASTM 8
TNO 2



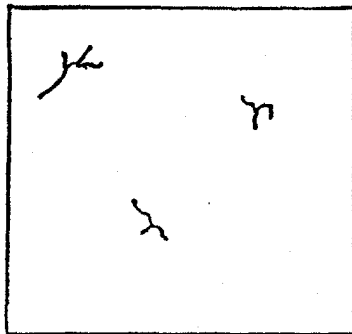
ASTM 4
TNO 6

ASTM 8
TNO 2

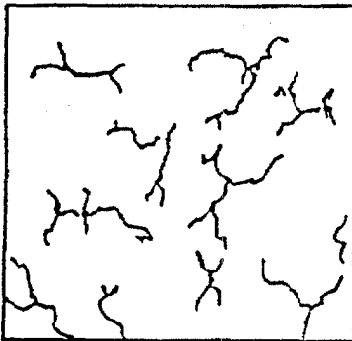


ASTM 4
TNO 6

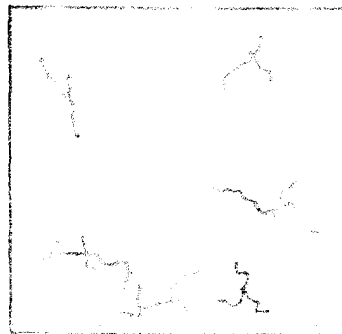
FIG. 1 (continued)



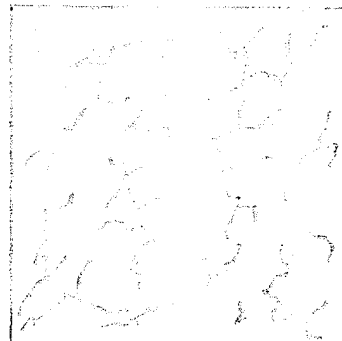
ASTM 8
TNO 2



ASTM 4
TNO 6



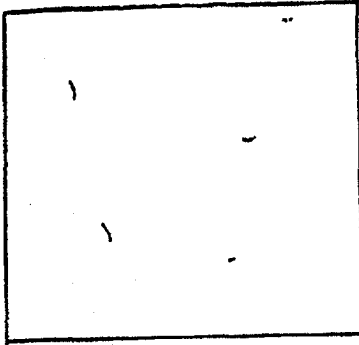
ASTM 8
TNO 2



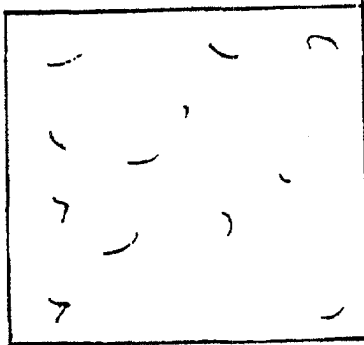
ASTM 4
TNO 6

5.1.6 SHRINKAGE

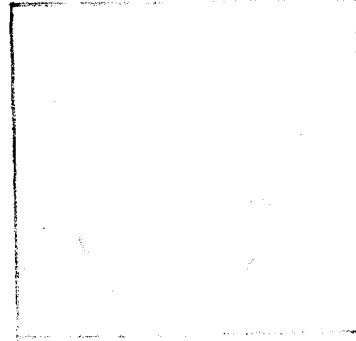
FIG. 1 (Continued)



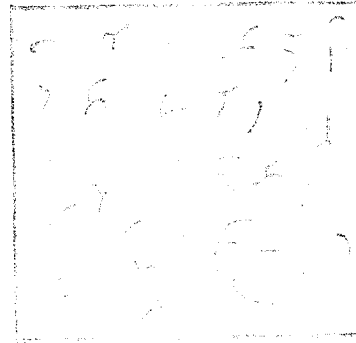
ASTM 8
TNO 2



ASTM 4
TNO 6



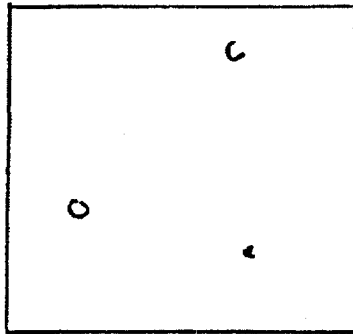
ASTM 4
TNO 6



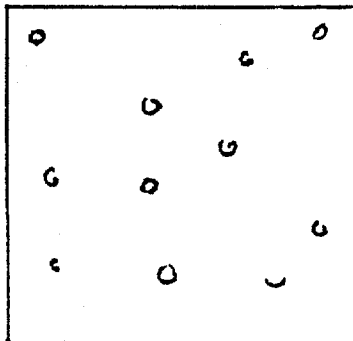
ASTM 4
TNO 6

5.1.7 SHORT,
RANDOM

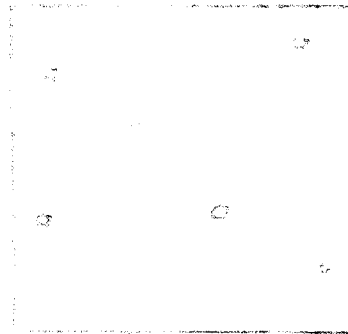
FIG. 1 (Continued)



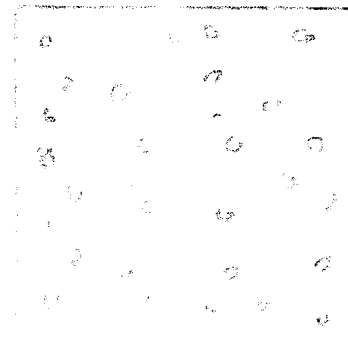
ASTM 8
TNO 2



ASTM 4
TNO 6



ASTM 6
TNO 4



ASTM 3
TNO 7

5.1.8 SIGMOID

FIG. 1

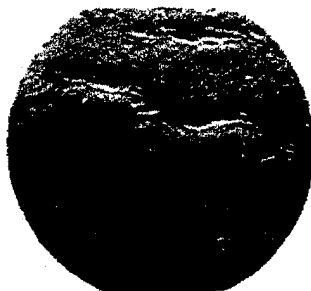


FIG. 2 No. 8 (Checking Magnified 10 Diameters)

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Evaluating Degree of Cracking of Exterior Paints¹

This standard is issued under the fixed designation D 661; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^ε) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 6471 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

^{ε1} NOTE—Footnote 3 was editorially changed in December 1988.

1. Scope

1.1 This test method covers the evaluation of the degree of cracking of exterior paints by comparison with photographic standards.

2. Referenced Documents

2.1 ASTM Standards:

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²

D 1150 Single- and Multi-Panel Forms for Recording Results of Exposure Tests of Paints²

2.2 Other Standards:

Pictorial Standards of Coating Defects Handbook³

3. Definition

3.1 *cracking*—that phenomenon manifested in paint films by a break extending through to the surface painted. Where this is difficult to determine, the break should be called a crack only if the underlying surface is visible. The use of a magnification of 10 diameters is recommended in cases where it is difficult to differentiate between cracking and checking (see Test Method D 660).

4. Significance and Use

4.1 Cracking failure of paint films can occur in use. This test method provides a means of evaluating the degree of the failure by comparing the pictorial standards.

5. Types of Cracking

5.1 Three types of cracking are recognized:

5.1.1 *Irregular Pattern Type*—Cracking in which the breaks in the film are in no definite pattern.

5.1.2 *Line Type*—Cracking in which the breaks in the film are generally arranged in parallel lines, usually either horizontally or vertically, over the surface of the film. These breaks often follow the line of brush marks.

5.1.3 *Sigmoid Type*—Cracking in which the breaks in the film form a pattern consisting of curves meeting and intersecting, usually on a relatively large scale.

6. Use of Photographic Reference Standards

6.1 The photographic reference standards that are part of this test method and are provided in the *Pictorial Standards of Coating Defects Handbook* are representative of the degree of cracking of exterior paint films. Figures 1 and 2 are for illustration purposes only and should not be used for evaluation.

6.2 The use of the photographic reference standards³ illustrated in Fig. 1 requires the following precautions:

6.2.1 The accompanying photographic reference standards show line-type cracking only. Irregular and sigmoid-type cracking may also be interpreted from these photographs.

6.2.2 Care must be taken not to confuse various types of failure that may be present on the same surface. This is particularly true in observing cracking and checking. Cracking may very often be an advanced stage of checking and is very often in evidence along with checking and other failures.

6.2.3 It must be realized that the degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison. On larger surfaces it is recommended that ratings be made at several locations and the mean and range reported.

6.2.4 Paint films may collect excessive quantities of dirt, which may mask the type and degree of failure. If necessary, dirt should be removed by careful and gentle brushing with a moderately soft brush.

6.2.5 In examining wood panels for cracking failure, the possibility of wood failure should be recognized. This takes the form of a cracking or splitting of the wood itself with a resultant rupture of the paint film. Also, some panels will develop "resin spewing" which will cause early failure by cracking. These points should be taken into consideration in any evaluations.

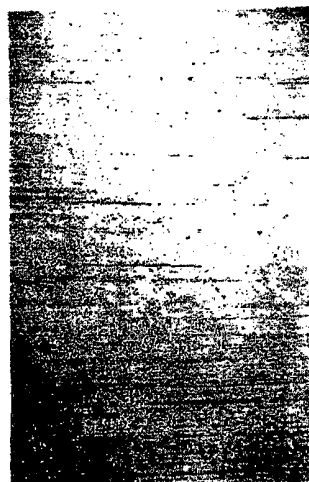
6.3 For convenience in recording the data obtained, the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

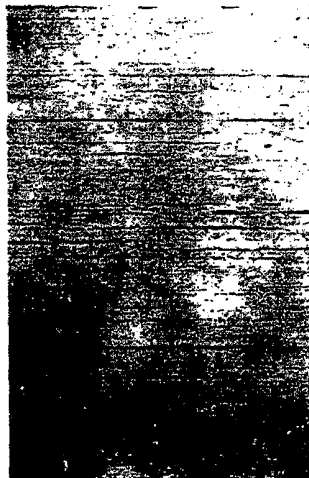
Current edition approved Aug. 29, 1986. Published October 1986. Originally published as D 661 - 42 T. Last previous edition D 661 - 44 (1981)^{ε1}.

² *Annual Book of ASTM Standards*, Vol 06.01.

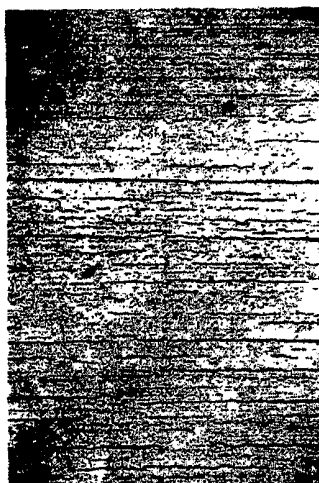
³ Copies of the pictorial photographic reference standards are contained in the publication *Pictorial Standards of Coating Defects* and may be obtained from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422. The silver halide-gelatin photographs are intended to be the only primary reference standards for this method. The reproductions of them in this test method are for the purpose of illustration only.



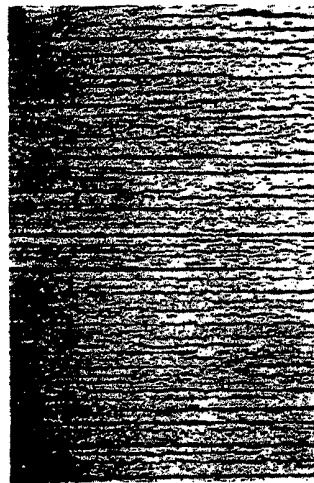
No. 8



No. 6



No. 4



No. 2

FIG. 1 Degrees of Cracking

records may be kept on forms such as Standard D 1150.⁴

7. Precision and Bias

7.1 No precision or bias statement has been established for this test method.

⁴ These record sheets may be obtained from ASTM Headquarters (order Adjunct No. 12-411500-11 and 12-411500-21) and from the Federation of

Societies for Coatings Technology, 1315 Walnut St., Suite 832, Philadelphia, PA 19107.

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Standard Test Method for Evaluating Degree of Erosion of Exterior Paints¹

This standard is issued under the fixed designation D 662; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 6431 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

^{e1} NOTE—Footnote 4 was editorially changed in December 1988.

1. Scope

1.1 This test method covers the evaluation of the degree of erosion of exterior paints by comparison with photographic standards.

2. Referenced Documents

2.1 ASTM Standards:

D 659 Method of Evaluating Degree of Chalking of Exterior Paints²

D 1150 Single- and Multi-Panel Forms for Recording Results of Exposure Tests of Paints²

2.2 Other Standards:

*Pictorial Standards of Coating Defects Handbook*³

3. Definition

3.1 *erosion*—that phenomenon manifested in paint films by the wearing away of the finish to expose the substrate or undercoat. The degree of failure is dependent on the amount of substrate or undercoat visible. Erosion occurs as the result of chalking. (See Method D 659 for evaluation of chalking.)

4. Significance and Use

4.1 Erosion failure of paint films can occur in use. This test method provides a mean of evaluating the degree of failure by comparing to pictorial standards.

5. Types of Erosion

5.1 Only one type of erosion is recognized, as defined in Section 3.

6. Use of Photographic Reference Standards

6.1 The photographic reference standards that are part of this test method and are provided in the *Pictorial Standards of Coating Defects Handbook* are representative of the degree of erosion of exterior paint films. Figure 1 is for illustration purposes only and should not be used for evaluation.

6.2 The use of the photographic reference standards³ illustrated in Fig. 1 requires the following precautions:

6.2.1 Care must be taken not to confuse various types of failure that may be present on the same surface.

6.2.2 It must be realized that the degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison. On larger surfaces it is recommended that ratings be made at several locations and the mean and range reported.

6.2.3 The photographic standards used represent various degrees of erosion of a white brushing type paint over a dark primer. This system was necessary to provide sufficient contrast for photographic purposes. The erosion of a film to its normal substrate is, however, readily visible to the naked eye so it may easily be compared to the standards and given a numerical rating.

6.2.4 In doubtful cases, erosion is sometimes more visible in a damp film than in a dry film. Also, with severe erosion, it is often easier to rate the degree of erosion in a damp film than in a dry film.

6.2.5 While erosion of a sprayed film is more regular in its wearing away, a numerical rating can be given to it by interpreting the amount of erosion in terms of these standards.

6.3 For convenience in recording the data obtained, the records may be kept on forms such as Standard D 1150.⁴

7. Precision and Bias

7.1 No precision or bias statement has been established for this test method.

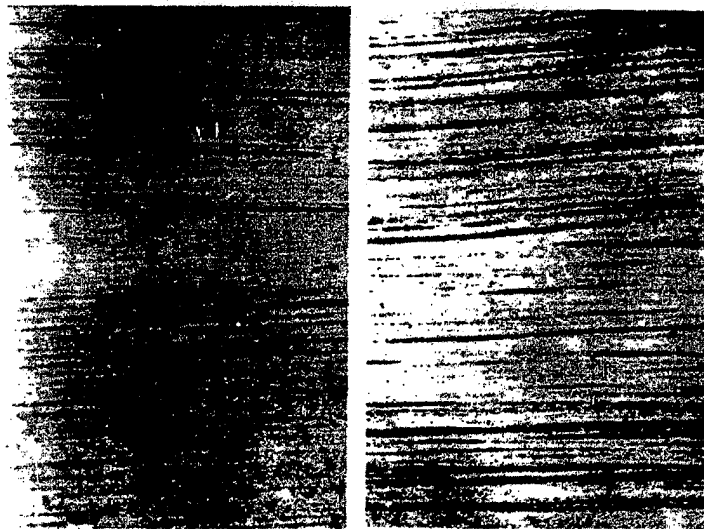
¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Aug. 29, 1986. Published October 1986. Originally published as D 662 - 42 T. Last previous edition D 662 - 44 (1981)^{e1}.

² *Annual Book of ASTM Standards*, Vol 06.01.

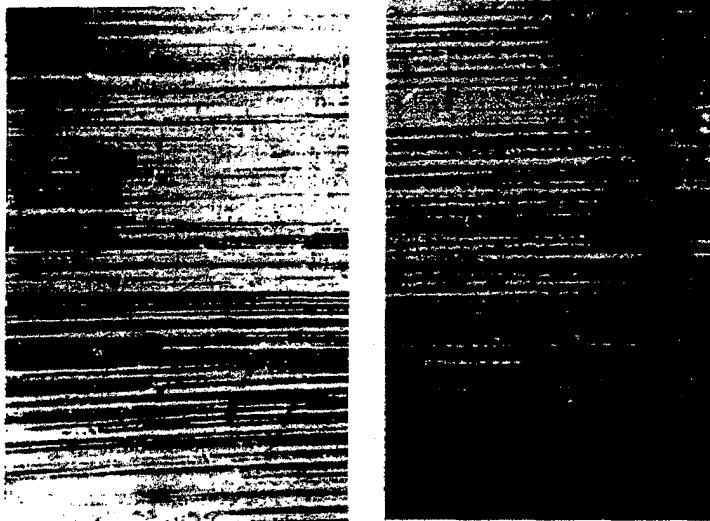
³ Copies of the pictorial photographic reference standards are contained in the publication *Pictorial Standards of Coating Defects* and may be obtained from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422. The silver halide-gelatin photographs are intended to be the only primary reference standards for this method. The reproductions of them in this test method are for the purpose of illustration only.

⁴ These record sheets may be obtained from ASTM Headquarters (order Adjunct No. 12-411500-11 and 12-411500-21) and from the Federation of Societies for Coatings Technology.



No. 8

No. 6



No. 4

No. 2

FIG. 1 Degrees of Erosion

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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1. Scope

1.1 This test method determines the relative resistance of a material to erosion by a rotating wheel.

1.2 This test method is applicable to materials, and addresses the respective appropriate applications.

2. Refer

2.1 ASTM D 2000
Automatic

3. Signif

3.1 This test method is used only for the results of

4. Appar

4.1 This test method is performed on a steel cylinder with two dimensions.

4.2 This test method is performed on a cylinder of complete g.

4.3 This test method is shown

4.4 This test method is performed on rubber or HK 715

4.5 This test method is performed on

¹ This test method and Related Committee D01.4. Current edition published as I
² Annual book
³ An apparatus Co., Station 9



Standard Test Method for No-Pick-Up Time of Traffic Paint¹

This standard is issued under the fixed designation D 711; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers a laboratory test for determining the no-pick-up time of a traffic paint by a rubber wheel.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*
D2000 Classification System for Rubber Products in Automotive Applications²

3. Significance and Use

3.1 This test method serves as a control test and should be used only as such. There is no direct correlation between the results of this test and field applications.

4. Apparatus

4.1 The apparatus³ as shown in Fig. 1 shall consist of a steel cylinder of the shape and dimensions as indicated, fitted with two replaceable O-rings and a ramp of shape and dimensions as shown.

4.2 The detailed dimensional requirements of the steel cylinder are given in Fig. 1. The total weight of the assembly complete with O-rings shall be 11 lb 14 oz $\pm$ 1 oz (5386 $\pm$ 28 g).

4.3 The detailed dimensional requirements of the ramp are shown in Fig. 1.

4.4 The replaceable O-rings shall be made of synthetic rubber or rubber-like material meeting the requirements of HK 715 of Specification D 2000.

4.5 The dimensional requirements of the O-ring are as

follows:

Outside diameter	4 1/2 in. (104 mm)
Inside diameter	3 3/8 in. (85 mm)
Cross section	3/8 in. (9.5 mm)

5. Procedure

5.1 Prepare a test stripe at least 3 in. (75 mm) in width of the paint to be tested by a mechanical spreader, or other suitable means on a clean plate glass panel at a wet film thickness of 15 ± 0.5 mils. (Use a plate glass panel approximately 4 by 8 by 1/8 in. (100 by 200 by 3 mm).)

5.1.1 Record the time of application. Allow the panel to dry in a horizontal position under the laboratory conditions specified (73.5 $\pm$ 3.5°F (23 $\pm$ 2°C) and 50 $\pm$ 5 % relative humidity).

5.1.2 Test the paints used with drop-in beads without beads unless otherwise specified or agreed upon between the purchaser and the seller.

5.2 Butt the glass plate against the ramp. At regular intervals remove the wheel from its rest, hold against the rest as a starting point, then free roll the weighted wheel down the inclined ramp and over the paint film with each roll of the wheel over a new wheel path. Position the wheel properly so that a clean surface of the wheel will come into contact with the paint film. As many as three passes of the wheel can be made before the wheel needs to be cleaned. This can be done with a rag saturated with acetone. It is best to set aside the wheel after washing until all the solvent has evaporated.

5.3 Note the end point for no-pick-up time when no paint adheres to the rubber rings of the test wheel when it is rolled over the paint film. As the end point is approached, roll the weighted wheel over the paint film every 30 s.

6. Report

6.1 Report the time elapsed between application of the paint and the end point as the drying time for no-pick-up of the traffic paint.

7. Precision and Bias

7.1 *Precision*—Because of the poor precision of this test method, if it is used in a specification, the maximum deviation from the maximum no-pick-up time specified should be agreed upon between the purchaser and the seller.

7.2 *Bias*—Bias cannot be determined.

8. Keywords

8.1 auto no-track time; drying time; traffic paint

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Paints.

Current edition approved May 26, 1989. Published July 1989. Originally published as D 711 - 43. Last previous edition D 711 - 84.

² *Annual Book of ASTM Standards*, Vols 09.01 and 09.02.

³ An apparatus meeting the requirements is available from Paul N. Gardner Co., Station 9, P.O. Box 6633, Fort Lauderdale, FL 33316.

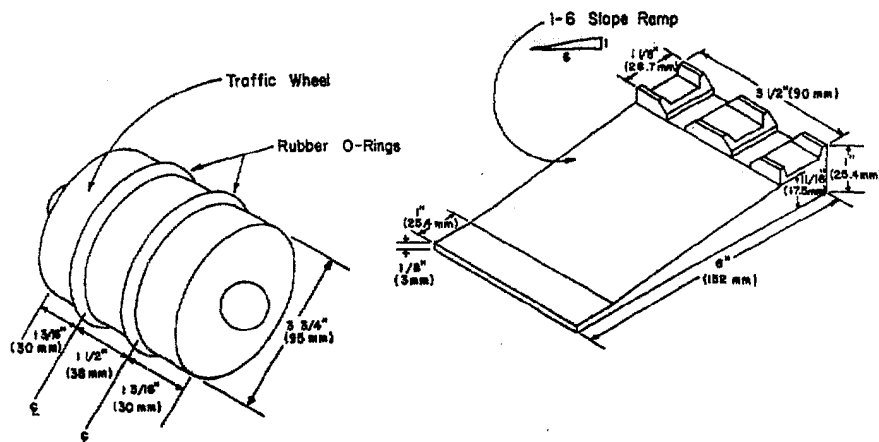


FIG. 1 Traffic Paint Drying Time Wheel and Ramp—Dual Model

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Standard Practice for Conducting Road Service Tests on Fluid Traffic Marking Materials¹

This standard is issued under the fixed designation D 713; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

NOTE—Paragraphs 9.4 and 9.5 were inadvertently deleted and were reinstated in February 1990.

1. Scope

1.1 This practice covers the determination of the relative service life of fluid traffic marking materials such as paint, thermoplastic, epoxy, and polyester products under actual road conditions using transverse test lines. Materials under test are applied under prescribed conditions and periodic observations are made using prescribed performance criteria.

1.2 *This practice may involve hazardous materials, operations and equipment. This practice does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this practice to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 711 Test Method for No-Pick-Up Time of Traffic Paint²

D 913 Test Method for Evaluating Degree of Resistance to Wear of Traffic Paint²

3. Significance and Use

3.1 This practice is an accelerated evaluation of bead retention and wear characteristics of fluid traffic marking materials. It is used to determine the useful life of such markings in the field. The same procedures are applicable to evaluating longitudinal lines to determine service life.

4. Type and Location of Pavement for Tests

4.1 Select sections where traffic is moderate and free-rolling with no grades, curves, intersections, or access points near enough to cause excessive braking or turning movements, where wear is uniform with full exposure to the sun throughout daylight hours, and there is good drainage. Select surfaces that are representative of the pavements upon which the fluid traffic marking material will be used in practice. Such surfaces include portland cement concrete, sheet asphalt, bituminous concrete, rock asphalt, and bituminous surface treatment.

5. Conditions at Time of Application

5.1 Clean the test area thoroughly of all foreign material. Do not apply traffic paint when the pavement surface is damp or wet nor when the pavement temperature is below 50°F (10°C). Application between 10 a.m. and 3 p.m. is recommended. During application record air and pavement temperature hourly.

6. Measurement of Wet Film Thickness

6.1 To aid in obtaining the correct film thickness, a length of roofing paper placed by the side of the road can be used. Place a rigid metal test panel on the roofing paper and in the path of the test line. A 12 by 12-in. (300 by 300-mm) metal panel $\frac{1}{16}$ in. (1.5 mm) in thickness is satisfactory. Immediately after the test line is applied by the motorized striper, read the wet film thickness. If the wet film thickness is not satisfactory, adjust the spray pressure and repeat until the target wet film thickness is attained. It is important that no glass beads or other interfering materials be present that would give a false wet film reading. When the wet film thickness is correct, apply a test line across a tared metal panel and weigh immediately. A balance must be immediately available and be thoroughly shielded from wind as well as be of 1500-g capacity with 0.1 g or better sensitivity. As a basis for determination of glass bead application (7.1) the weight of a paint line 4 by 12 in. (without consideration for solvent loss) can be calculated as follows:

$$W = 0.0943 \times t \times g$$

where:

W = weight of paint line, g,

t = mil thickness, and

g = weight per gallon, lb.

7. Measurement of Glass Beads

7.1 After the completion of 6.1, apply another test line to a tared panel with the motorized striper, this time also adding the glass beads, and weigh immediately. The weight difference between this measurement and that in 6.1 gives the amount of glass beads on the panel. The process can be repeated if an adjustment in the bead application rate is needed. The weight of applied glass beads can be calculated as follows:

$$W = 1.418 \times B$$

where:

W = weight of glass beads, g, and

B = glass beads per gallon of paint, lb.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved February 15, 1990. Published April 1990. Originally published as D 713 - 43 T. Last previous edition D 713 - 87.

² *Annual Book of ASTM Standards*, Vol 06.01.

8. Application Procedure

8.1 Apply the test stripes 4 in. (100 mm) in width and transversely on the road. At the option of the purchaser, the test stripes may be applied to the pavement at an angle of 45° to the direction of the traffic, or longitudinally in each wheel path, in order to increase the area of contact with traffic.

8.2 Apply test stripes (trained personnel under the supervision of the purchaser) by using a pavement-marking machine similar to the production pavement marking equipment. Apply the test stripes to at least two sections of each road surface selected to ensure against undetected road surface problems. When more than one specimen is tested at more than one location, change the sequence of placement to minimize the effect of time of day and time period before the test deck is opened to traffic.

8.3 Apply at least two lines of each specimen in each section for better statistical reliability. A tared panel as used in the measurement of glass beads (see 7.1) should be placed between the wheel track of one line's application as a double check for material and glass bead application rates. Weights between this reading and that found in 7.1 should agree within 0.5 g. The purchaser may wish to place another smaller panel between the wheel tracks to retain for future reference.

8.4 The road surface test lines have a wet film thickness within ± 0.5 mil of that required by the purchaser.

NOTE 1—If no wet film thickness is specified, 15.0 mil is recommended.

8.5 A fluid marking material with which the purchaser has had considerable production experience is included in the test series as a control.

8.6 Glass beads are placed in the paint film within ± 0.5 lb/gal of that required by the purchaser. All glass beads are supplied by the purchaser.

NOTE 2—If no bead application rate is specified, 6.0 lb/gal of paint is recommended.

9. Performance Criteria

9.1 *Auto-No-Track Time*—The auto no-track time is determined by passing over the freshly applied line in a simulated passing maneuver with a standard size passenger car with regular treads (no snow treads). A line showing no visual pick-up and redeposition of the materials onto the pavement surface when viewed from a distance of 50 ft in the highway direction is considered as showing no pick-up and conforming to the drying time requirements.

9.1.1 The test line is applied at the same temperature, the same wet film thickness, and the same rate of glass beads as will be specified by the purchaser in production application.

9.1.2 The no-track maximum time is measured when the pavement temperature is from 60 to 120°F (15 to 50°C) and under local humidity conditions, providing that the pavement is dry.

NOTE 3—A laboratory no-track test is described in Test Method D 711. This test is a laboratory control test and has little or no correlation with field results. Some paints have a tendency to skin over rapidly allowing the specimen to meet the maximum specified time for the laboratory test, but would be unsatisfactory at that time on a road surface.

9.2 *Appearance*—The impression of the observer of the

general condition of the test lines when viewed without any detailed inspection, from a distance of at least 10 ft. It is a measure of satisfactory or unsatisfactory appeal to the observer. It includes a comparison of the color of the surface under consideration with the original color, taking into account changes due to yellowing, bleeding, darkening, fading, dirt collection, mold growth, etc. This determination is made in each wheel track in an area extending 9 in. (229 mm) each side of the point of greatest wear. The appearance is rated either acceptable or unacceptable.

9.3 *Durability*—The durability is equal to one tenth of the percentages of material remaining on the pavement (when examined by the unaided eye). This determination is made in each wheel track in an area extending 9 in. (229 mm) each side of the point of greatest wear. The percent of paint remaining on the pavement is considered as the percent of the prescribed area of test stripe in which the substrate is not exposed. Make the evaluations in accordance with Test Method D 913.

NOTE 4—In the absence of a specification by the purchaser, failure shall be a rating less than 4 (less than 40 % material remaining on the pavement).

9.4 *Night Visibility*—Night visibility designates the apparent brightness when examined at night under tungsten illumination from the side of the road, with eye and light source separated by 1½ in. (40 mm) that corresponds to an observation angle of approximately ½°. Night visibility determinations are made in the wheel track areas used for rating durability, and are based on a factor of 10 for an unexposed standard panel placed next to the test stripe and 0 for no apparent brightness.

NOTE 5—In the absence of a specification by the purchaser, failure shall be a rating less than 4.

NOTE 6—A new test method for night visibility using a retro-reflectometer is being developed which is expected to have much greater precision.

9.5 *Length of Useful Life*—The length of useful life is determined by the number of days of duration between the date the sample was applied to the road surface and the date any one of the designated measurements falls below the specified minimum rating.

10. Evaluation Procedure

10.1 Make periodic inspections of the test sections in accordance with 9.2, 9.3, and 9.4. Record at each inspection the general daytime appearance (including color), film condition, and night visibility (retro-reflectance).

10.2 Inspect the test lines at regular monthly intervals. As the test lines approach failure, they should be evaluated every 2 weeks (weather permitting). The test lines must be evaluated until failure. Different types of traffic paint and markings do not wear out in a linear manner. Hence, it is not possible to extrapolate to failure some intermediate data. The winter season is also a must when evaluating test lines.

11. Calculation

11.1 Make cost calculations as follows:

$$C = M/L \times F$$

where:

C = cost per foot per day of useful life,

M = c
L = le
F = fe

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M = cost of paint per gallon,
 L = length of useful life in days as previously observed, and
 F = feet per gallon actually applied.

NOTE 7—The cost of the glass beads is usually not included in the calculation because it will be a constant when comparing the relative performance of a specified class of paint. Various types of paints and other fluid marking materials, however, can have different bead wetting characteristics, and beads with different wetting characteristics also are available, either of which could influence the useful life of the line. When testing more than one type of material or one type of bead, the

optimum cost/performance may be at a different bead application rate than that recommended in 8.6.

11.2 To determine the relative performance of the different fluid traffic marking materials during the course of the test, the following weighted rating, R , can be used:

$$R = 0.30A + 0.30D + 0.40N$$

where:

A = appearance as defined in 9.2 and determined using a rating from 0 to 10,

D = durability as defined in 9.3, and

N = night visibility as defined in 9.4.

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Standard Test Method for Evaluating Degree of Blistering of Paints¹

This standard is issued under the fixed designation D 714; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 6461 of Federal Test Method Standard No. 141 A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method employs photographic reference standards to evaluate the degree of blistering that may develop when paint systems are subjected to conditions which will cause blistering. While primarily intended for use on metal and other nonporous surfaces, this test method may be used to evaluate blisters on porous surfaces, such as wood, if the size of blisters falls within the scope of these reference standards. When the reference standards are used as a specification of performance, the permissible degree of blistering of the paint system shall be agreed upon by the purchaser and the seller.

2. Significance and Use

2.1 A phenomenon peculiar to painted surfaces is the formation of blisters relative to some system weakness. This test method provides a standard procedure of describing the size and density of the blisters so that comparisons of severity can be made.

3. Reference Standards

3.1 The photographic reference standards are glossy prints.² Figures 1 to 4 are reproductions of these standards and are included to illustrate two characteristics of blistering: size and frequency.

3.2 *Size*—Reference standards have been selected for four steps as to size on a numerical scale from 10 to 0, in which

No. 10 represents no blistering. Blistering standard No. 8 represents the smallest size blister easily seen by the unaided eye. Blistering standards Nos. 6, 4, and 2 represent progressively larger sizes.

3.3 *Frequency*—Reference standards have been selected for four steps in frequency at each step in size, designated as follows:

Dense, *D*,
Medium dense, *MD*,
Medium, *M*, and
Few, *F*.

NOTE 1—A quantitative physical description of blistering would include the following characteristics determined by actual count:

Size distribution in terms of mensuration units,
Frequency of occurrence per unit area,
Pattern of distribution over the surface, and
Shape of blister

For the usual tests, an actual count is more elaborate than is necessary.

4. Procedure

4.1 Subject the paint film to the test conditions agreed upon by the purchaser and the seller. Then evaluate the paint film for the degree of blistering by comparison with the photographic reference standards in Figs. 1 to 4.

5. Reporting

5.1 Report blistering as a number (Note 2) designating the size of the blisters and a qualitative term or symbol indicating the frequency.

5.2 Intermediate steps in size or frequency of blisters may be judged by interpolation.

5.3 When the distribution of blisters over the area has a nonuniform pattern, use an additional phrase to describe the distribution, such as "small clusters," or "large patches."

NOTE 2—The number refers to the largest size blister that is numerous enough to be representative of the specimen. For example, photographic standard No. 4, "Dense," has blisters ranging in size from about No. 7 to No. 4, inclusive.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 714 - 43 T. Last previous edition D 714 - 56 (1981).

² Glossy prints of the photographic reference standards showing types of blistering are available at a nominal charge from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103. Request Adjunct No. 12-407140-00.

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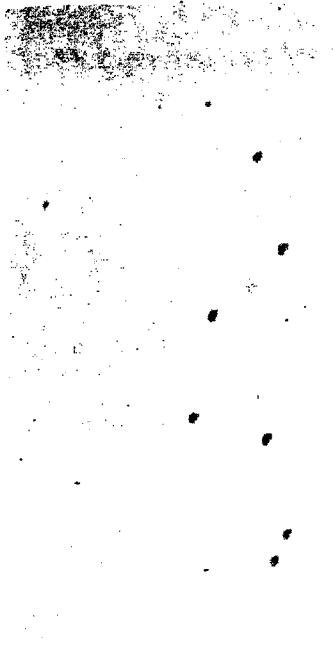
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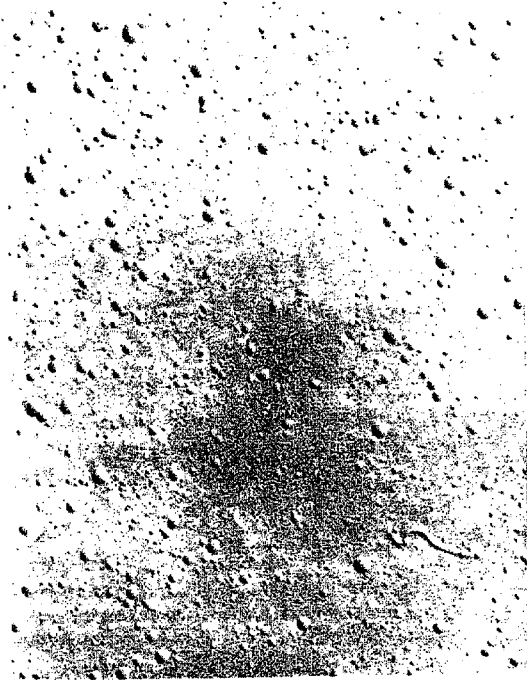


FIG. 1 Blister Size No. 2



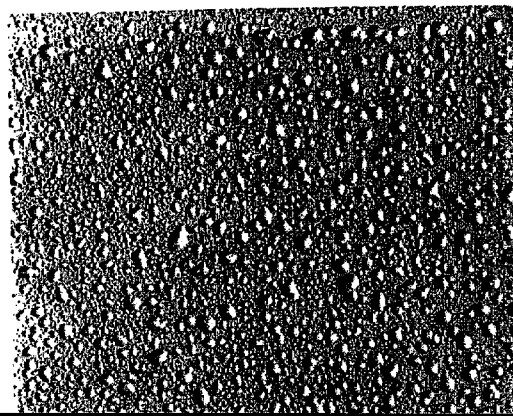
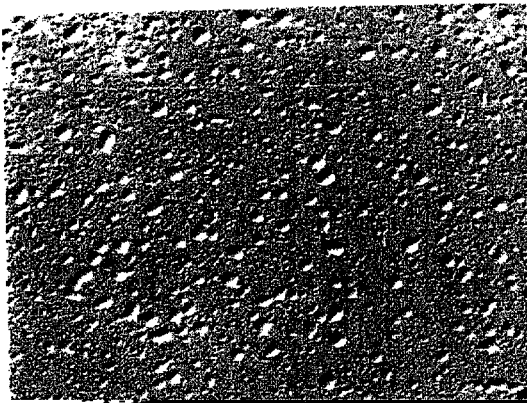


Few



Medium

FIG. 2 Blister Size No. 4



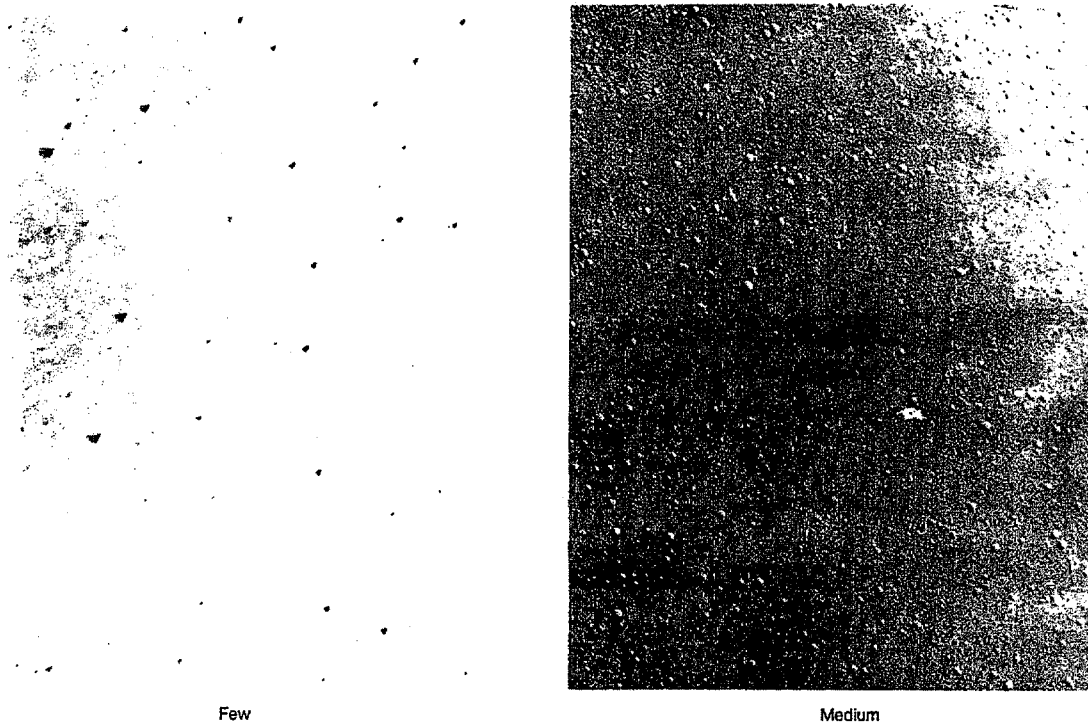


FIG. 3 Blister Size No. 6

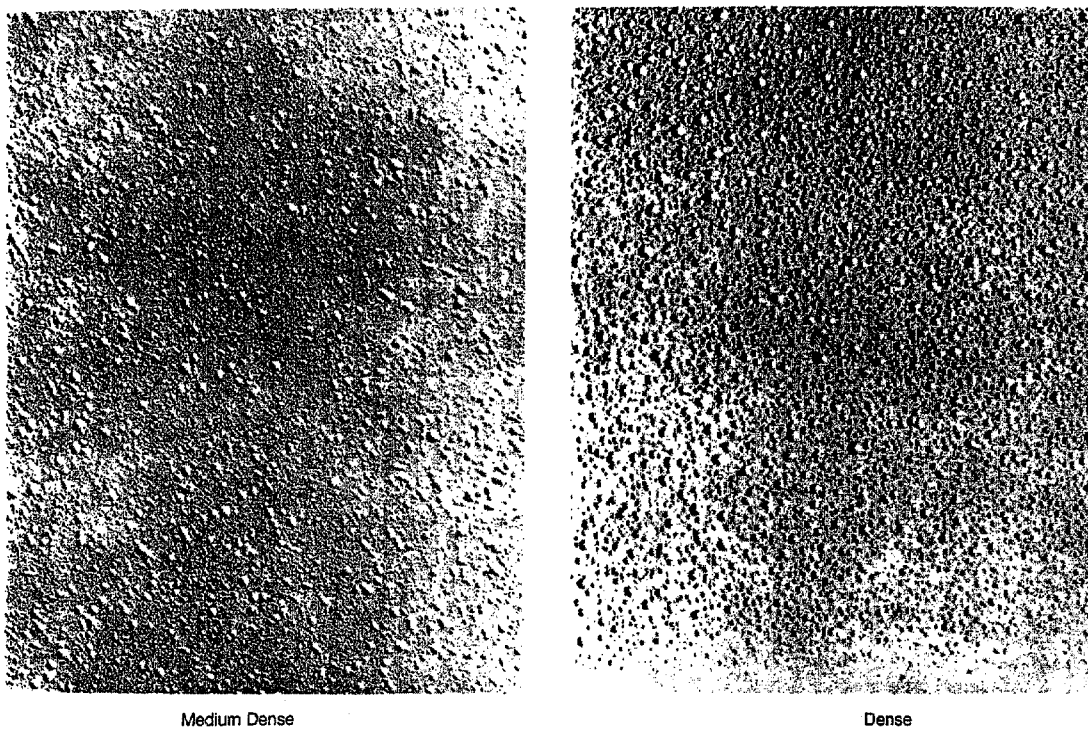
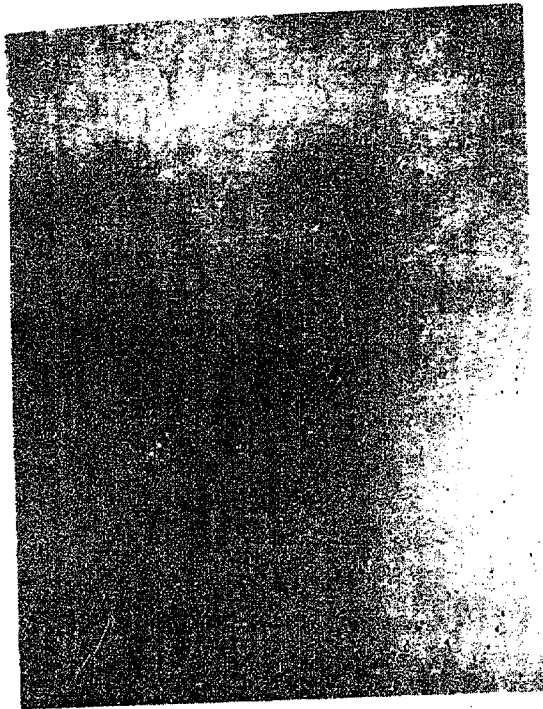
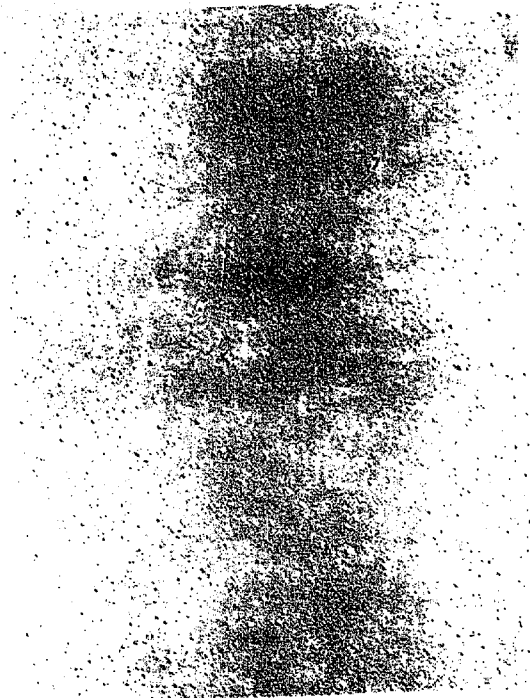


FIG. 3 Continued

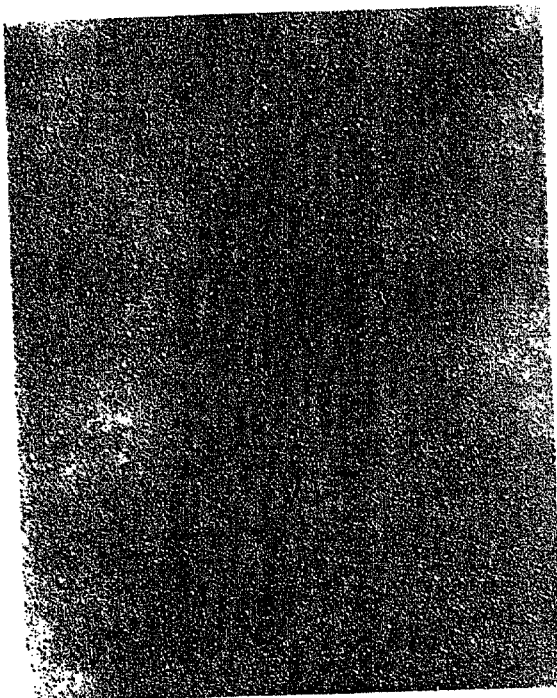


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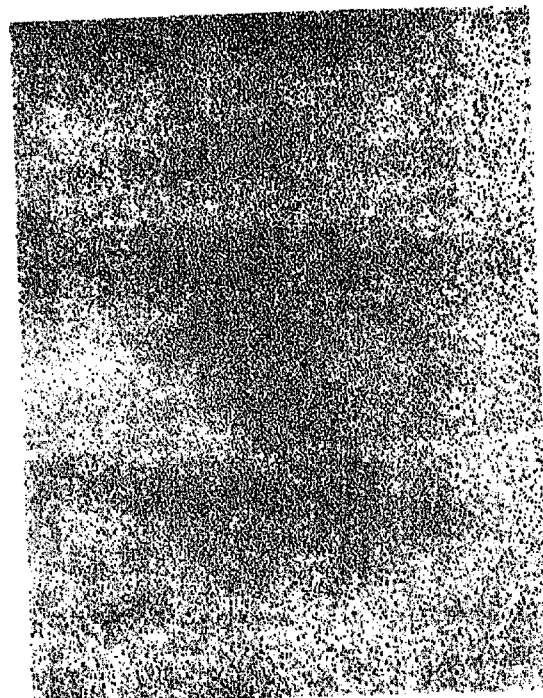


Medium

FIG. 4 Blister size No. 8



Medium Dense



Dense

FIG. 4 Continued

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Standard Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints¹

This standard is issued under the fixed designation D 772; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 6441 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers the evaluation of the degree of flaking (scaling) of exterior paints by comparison with photographic standards.

2. Referenced Documents

2.1 ASTM Standard:

D 1150 Single and Multi-Panel Forms for Recording Results of Exposure Tests of Paints²

2.2 Other Standards:

*Pictorial Standards of Coating Defects Handbook*³

3. Definition

3.1 *flaking (scaling)*—that phenomenon manifested in paint films by the actual detachment of pieces of the film itself either from its substrate or from paint previously applied. Flaking (scaling) is generally preceded by cracking or blistering, and is the result of loss of adhesion, usually due to stress-strain factors coming into play.

4. Significance and Use

4.1 Flaking (scaling) failure of paint films can occur in use. This test method provides a means of evaluating the degree of failure by comparing to pictorial standards.

5. Type of Flaking (Scaling)

5.1 Only one type of flaking (scaling) is recognized, as defined in Section 3.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Aug. 29, 1986. Published October 1986. Originally published as D 772 - 44 T. Last previous edition D 772 - 47 (1981)^{ε1}.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Copies of the pictorial photographic reference standards are contained in the publication *Pictorial Standards of Coating Defects* and may be obtained from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422. The silver halide-gelatin photographs are intended to be the only primary reference standards for this method. The reproductions of them in this test method are for the purpose of illustration only.

6. Use of Photographic Reference Standards

6.1 The photographic reference standards that are part of this test method and are provided in the *Pictorial Standards of Coating Defects Handbook* are representative of the degree of cracking of exterior paint films. Figure 1 is for illustration purposes only and should not be used for evaluation.

6.2 The use of the photographic reference standards² illustrated in Fig. 1 requires the following precautions:

6.2.1 Care must be taken not to confuse various types of failure that may be present on the same surface.

6.2.2 It must be realized that degree of failure will vary over any given area. Therefore, an average portion of the film should be used for comparison. On larger surfaces it is recommended that ratings be made at several locations and the mean and range reported.

6.2.3 In technical literature, a distinction is sometimes made between flaking and scaling. In most cases, however, flaking and scaling refer to the same phenomenon. In some instances, the term flaking is used to describe the detachment of pieces of film less than 1/4 in. (6.4 mm) in size, and scaling, the detachment of pieces over 1/4 in. in size. In other instances, the term flaking is used to describe the detachment of pieces of film from the immediate undercoat (intercoat failure) and scaling the detachment of pieces from the base (complete failure). It should be kept in mind that the flakes may vary widely in size and shape from those illustrated by the reference standards in Fig. 1, varying from a fraction of an inch to several inches in size.

6.2.4 Peeling is frequently due to a moisture condition and when this is evident it should be taken into consideration in any evaluation.

6.2.5 For convenience in recording the data obtained, the records may be kept on forms such as Standard D 1150.⁴

7. Precision and Bias

7.1 No precision or bias statement has been established for this test method.

⁴ These record sheets may be obtained from ASTM Headquarters (order Adjunct No. 12-41150-11 and 12-41150-21) and from the Federation of Societies for Coatings Technology.



No. 2



No. 4

FIG. 1 Degrees of Flaking (Scaling)

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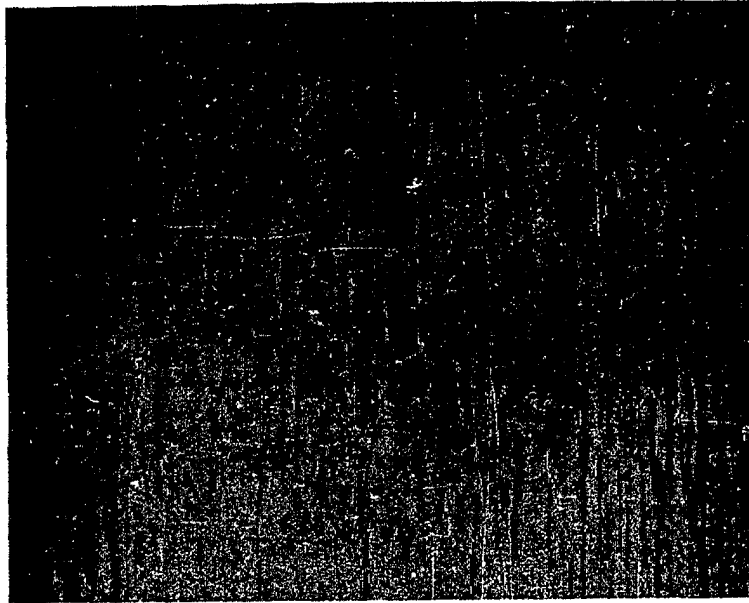
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No. 6



No. 8

FIG. 1 Degrees of Flaking (Scaling) (Concluded)

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Standard Practice for Conducting Tests on Paint and Related Coatings and Materials using Filtered Open-Flame Carbon-Arc Light and Water Exposure Apparatus¹

This standard is issued under the fixed designation D 822; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice covers accelerated exposure testing of paint, varnish, lacquer, and related products using filtered open-flame carbon-arc devices as described in Practice G 23. Two basic apparatus types are available for coating evaluation since open-flame carbon-arc devices can be manufactured with or without automatic humidity control. These are Types E and EH as described in Practice G 23.² Each type of device can be operated with different types of glass filters that filter various amounts of ultraviolet (UV) radiation. *All references to use of this practice must include a description of the filter used.* Table 1 describes commonly used test conditions. Interlaboratory comparisons *must* be made only with devices using the same filter type and test conditions.

1.2 Previous versions of this practice described exposures using either open-flame carbon arcs or enclosed carbon arcs.

NOTE 1—A new practice using enclosed carbon-arc exposure apparatus is under development. Another procedure for exposing these products is covered by Practice D 3361 in which the specimens are subjected to radiation from an unfiltered open-flame carbon arc that produces much higher levels of short wavelength radiation than filtered open flame or enclosed carbon arcs. Only automatic humidity controlled open-flame carbon-arc apparatus (Type EH) is applicable to Practice D 3361.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 4.*

2. Referenced Documents

2.1 ASTM Standards:

- D 358 Specification for Wood to Be Used as Panels in Weathering Tests of Coatings³
- D 523 Test Method for Specular Gloss³
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products³

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Tests for Protective Coatings.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 822 - 45. Last previous edition D 822 - 86.

² Apparatus and carbon arcs manufactured by Atlas Electric Devices Company, 4114 N. Ravenswood Ave., Chicago, IL 60613, have been found satisfactory for this purpose.

³ Annual Book of ASTM Standards, Vol 06.01.

- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces³
- D 659 Method of Evaluating Degree of Chalking of Exterior Paints³
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints³
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints³
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints³
- D 714 Test Method for Evaluating Degree of Blistering of Paints³
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints³
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers³
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base³
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base³
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁴
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁵
- D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates³
- D 2616 Test Method for Evaluation of Visual Color Difference with a Gray Scale⁴
- D 3361 Practice for Operating Light- and Water-Exposure Apparatus (Unfiltered Open-Flame Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products Using the Dew Cycle³
- D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films³
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁶
- G 23 Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Expo-

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁶ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

TABLE 1 Test Cycles Commonly Used for Carbon-Arc Exposure Testing of Paints, Varnishes, Lacquers and Related Coatings

Cycle Description	Black Panel Temp, ^a °F (°C)	Typical Uses
102 min light	145 ± 5	general coatings
18 min light and water spray	(63 ± 2.5)	
18 h using:		general coatings
102 min light	145 ± 5	
18 min light and water spray	(63 ± 2.5)	
6 h at 95 ± 4 % relative humidity with no water spray	75 ± 3 (24 ± 1.5)	
48 min light	145 ± 5	original equipment manufactured coatings
12 min light and water spray	(63 ± 2.5)	
4 h light	145 ± 5	exterior pigmented paints
4 h water spray	(63 ± 2.5)	
12 h light	145 ± 5	exterior wood stains and clears
12 h water spray	(63 ± 2.5)	
8 h light	145 ± 5	marine enamels
10 h light and water spray	(63 ± 2.5)	
6 h water spray		

^a Unless otherwise indicated, black panel temperature during light only portion of the cycle.

^b Historical convention has established this as a very commonly used test cycle.

sure of Nonmetallic Materials⁴

3. Significance and Use

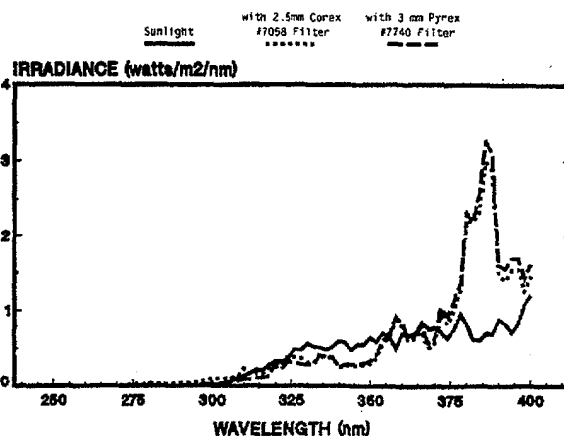
3.1 This practice is intended to evaluate coating films for their stability in apparatus that expose ultraviolet (UV) light and moisture. If the spectral power distribution of the light source used for exposure tests does not adequately simulate that of terrestrial solar radiation, it may produce a different type of degradation and distort the ranking of materials obtained in outdoor exposures. Figures 1 and 2 compare representative spectral power distributions of the open-flame carbon arc (with two different types of glass filters) with that of terrestrial sunlight. Soda borosilicate glasses remove more short wavelength UV radiation than potash lithia glasses⁷ and their use can provide carbon-arc exposures that are a better approximation of terrestrial solar UV. Exposures using soda borosilicate glass filters will typically take longer to produce photochemical degradation.

3.2 No single operating procedure for light-exposure apparatus with or without water can be specified as a direct simulation of natural exposure. This practice does not express, or imply, a specific correlation with outdoor exposure.

3.3 Since natural environments vary with respect to climate, geography, and topography, it may be expected that the effects of natural exposures will vary accordingly. Furthermore, all materials are not affected equally by the same environment. Therefore, results obtained by use of this practice should not be represented as equivalent to those of any natural weathering test until the degree of correlation has been empirically established for the material in question.

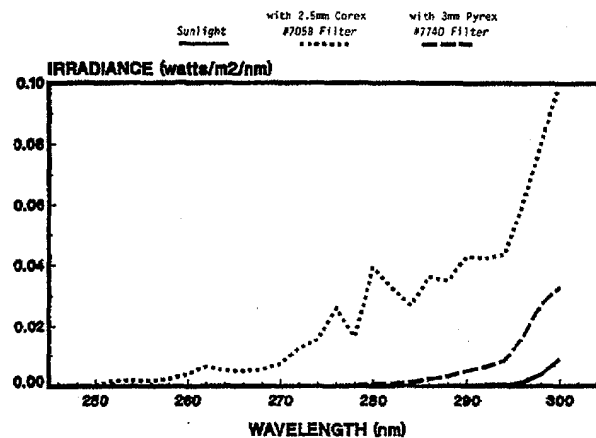
3.4 Variations in results are possible between instruments of the same type operating within the accepted limits of this practice. It is recommended that results obtained using this practice be compared with those of a control or reference

⁷ Pyrex No. 7740 and Correx No. 7058 Glass Filters, manufactured by Corning Glass Works, Corning, NY 14831, or equivalent, have been found satisfactory for this purpose.



NOTE—Sunlight was measured in Phoenix, AZ, at the summer solstice with clear sky at solar noon using a double grating monochromator (1-nm bandpass) with a quartz cosine receptor on an equatorial follow-the-sun mount. The carbon arc irradiance was measured at the sample plane centered within the allowed sample area. Because of momentary fluctuations in intensity due to flickering of the carbon arc flame, the spectral power distributions shown in this figure are representative and are not meant to be used to calculate or estimate total radiant exposure for tests in carbon-arc devices.

FIG. 1 Representative Spectral Power Distributions (250 to 400 nm) for Terrestrial Sunlight and Open Flame Carbon Arc Using Two Types of Glass Filters



NOTE—Measurements made as described in Fig. 1. Because of momentary fluctuations in intensity due to flickering of the carbon arc flame, the spectral power distributions shown in this figure are representative and are not meant to be used to calculate or estimate total radiant exposure for tests in carbon-arc devices.

FIG. 2 Representative Spectral Power Distributions (250 to 300 nm) for Terrestrial Sunlight and Open Flame Carbon Arc Using Two Types of Glass Filters

material that is mutually agreed upon between the interested parties.

3.5 All references to exposures in accordance with this practice must include a complete description of the filter glass and test cycle used.

4. Hazards

4.1 Precaution—In addition to other precautions, never look directly at the carbon arc because UV radiation can damage the eye. Most carbon-arc machines are equipped

with door safety switches, but users of old equipment must be certain to turn the OPERATE switch to OFF before opening the test-chamber door.

4.2 The burning carbon rods used in these devices become very hot during use. Make sure to allow at least 15 min for the arcs to cool after the device is turned off before attempting to change the carbon rods. Avoid inhaling ash dust when changing carbon rods.

5. Test Specimens

5.1 Apply the coating to flat (plane) panels with the substrate, method of preparation, method of application, coating system, film thickness, and method of drying consistent with the anticipated end use, or as mutually agreed upon between the producer and user.

5.2 Panel specifications and methods of preparation include but are not limited to Methods D 609, Specification D 358, or Practice D 1730. Select panel sizes suitable for use with the exposure apparatus.

5.3 Coat test panels in accordance with Test Methods D 823 and measure the film thickness in accordance with an appropriate procedure selected from Test Methods D 1005, D 1186, or D 1400. Nondestructive methods are preferred because panels so measured need not be repaired.

5.4 Prior to exposing coated panels in the apparatus, condition them at $73 \pm 3^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity for one of the following periods in accordance with the type of coating:

Baked coatings	24 h
Radiation-cured coatings	24 h
All other coatings	7 days

NOTE 2—The procedures and specifications described in 5.2 through 5.4 are recommended but others may be used if agreed upon between all interested parties.

6. Procedure

6.1 Mount the test specimens vertically both above and below the horizontal center line of the source of radiation. Rotate the specimens from the upper to lower portion of the revolving sample rack to provide more uniform exposure conditions over their surface. Samples may also be inverted during rotation. When the exposure interval does not exceed 24 h, locate each specimen equidistant from the horizontal axis of the arc. For exposure intervals not exceeding 100 h, rotate the specimens daily. For longer exposures, expose specimens for approximately as many hours in the top portion of the specimen rack as in the bottom portion. Other methods of achieving uniform total irradiation may be employed if mutually agreed upon between the concerned parties.

6.2 Table 1 lists test-cycle conditions commonly used for evaluation of paints, varnishes, lacquers, and related coatings. As discussed in 3.1, each of the test cycles can be run using different types of glass filters. The filter glass used must

be agreed upon between all interested parties.

6.3 It is recommended that the temperature of the water used for specimen spray be $60 \pm 9^\circ\text{F}$ ($16 \pm 5^\circ\text{C}$). Water used for specimen spray must meet the purity levels specified in Practice G 23 in order to avoid unrealistic water spotting.

6.4 When mutually agreed upon, cycles other than those listed in Table 1 may be used. The term "cycle" is defined as the set of exposure conditions (light, light plus water spray, dark periods) that are repeated.

7. Periods of Exposure

7.1 Use one of the following methods to determine the duration of the exposure under this practice:

7.1.1 A mutually agreed upon specified number of total hours.

7.1.2 The number of total hours of exposure required to produce a mutually agreed upon amount of change in either the test specimen or an agreed upon control or reference material.

8. Evaluation of Specimens After Exposure

8.1 Determine the changes in exposed test specimens in accordance with ASTM Test Methods D 523, D 610, D 659, D 660, D 661, D 662, D 714, D 772, D 2244, D 2616, D 4214, E 97 and Practice D 1729. Consider product use requirements when selecting appropriate methods.

8.2 Evaluate test specimens by ranking their performance relative to a control or reference material exposed at the same time.

8.3 Plot properties of test specimens and controls as a function of exposure time and compare rate of change with that of the control or reference material. When this method of evaluation is used, the control or reference material must be exposed at the same time and in the same device as the test specimen.

8.4 Other methods for evaluating test specimens may be used if mutually agreed upon between all interested parties.

9. Report

9.1 Report the following information:

9.1.1 Complete description of exposure procedure used including the following:

9.1.1.1 Light/light plus water/dark cycle used,

9.1.1.2 Operating black-panel temperature during all portions of the exposure cycle,

9.1.1.3 Operating relative humidity,

9.1.1.4 Type and thickness of filter glass used, and

9.1.1.5 Temperature of water used for water spray.

9.1.2 Total hours of test.

9.1.3 Test specimen preparation.

9.1.4 Identification of controls used.

9.1.5 Results of evaluation test or tests performed on specimens and control or standard samples.

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Standard Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels¹

This standard is issued under the fixed designation D 823; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace Method 2121 of Federal Test Method Standard No. 141A. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 Five test methods are given for preparing films of uniform thickness of coatings on test panels. These test methods are:

- Test Method A—Automatic Spray Machine Application
- Test Method B—Motor-Driven Dip Coater Application
- Test Method C—Motor-Driven Blade Film Application
- Test Method D—Hand-Held Spray Gun Application
- Test Method E—Hand-Held Blade Film Application

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 1005 Test Methods for Measurement of Dry Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer and Related Materials²

TEST METHOD A—AUTOMATIC SPRAY MACHINE APPLICATION

3. Summary of Test Method

3.1 A liquid material is applied to a test panel by means of an automatic spray machine consisting of a mounted spray gun and a panel holder. This machine can (1) move the

panel holder, with test panel, at a uniform speed through the atomized spray produced by a fixed spray gun, or (2) it can move the gun, with atomized spray, at a uniform speed past the test panel mounted on a fixed panel holder. A machine equipped with a programmable system can index the spray gun vertically for multiple passes and for multiple coats with selective time delay.

3.2 The thickness of coating applied is controlled by the traverse speed of the panel or gun, the fluid delivery rate of the gun, the viscosity of the material, and the amount of nonvolatile matter in the material.

4. Significance and Use

4.1 This test method should be used for those coatings that are designed for spray applications of objects in the factory or in the field. It is particularly important that it be used in the evaluation of metallic coatings for appearance properties, such as gloss and color.

4.2 Coatings applied by this test method may exhibit a slight orange-peel or spray wave.

5. Apparatus

5.1 *Test Panels*, of any smooth, planar material of a size that can be accommodated by the panel holder of the automatic spray machine.

5.1.1 When steel panels are used, they should be prepared in accordance with the appropriate method in Methods D 609.

5.2 *Automatic Spray Machine*,³ equipped with a panel holder and a mounting for a spray gun. The machine shall be designed to move the panel holder at a uniform speed past the fixed gun mount, or designed to move the gun mount at a uniform speed past the fixed panel holder. The panel holder or the gun mount traverse speed shall be adjustable from 25 to 100 ft (7.5 to 30 m)/min. Typical machines are shown in Fig. 1.

NOTE 1—Some automatic spray machines provide additional features that can improve the uniformity of film preparation. Some examples are: a z-bar panel holder; indexing of the panel holder at right angles to the gun to provide uniform lapping; and automatic control of number of passes, time between passes, and lapping distance.

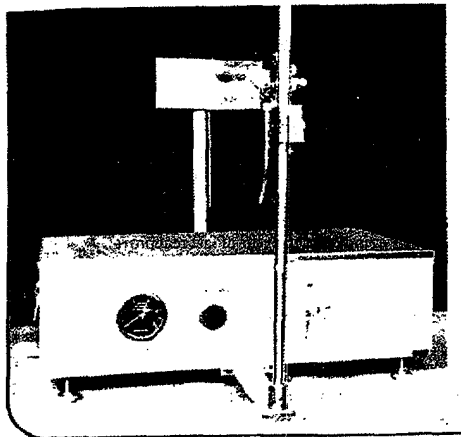
5.3 *Spray Gun*, any that will provide a uniform fan-type

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

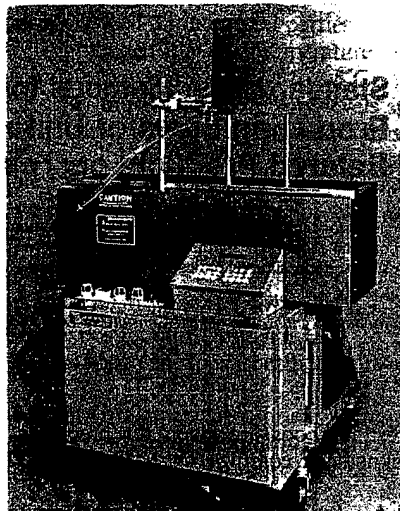
Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 823 - 45 T. Last previous edition D 823 - 87.

² *Annual Book of ASTM Standards*, Vol 06.01.

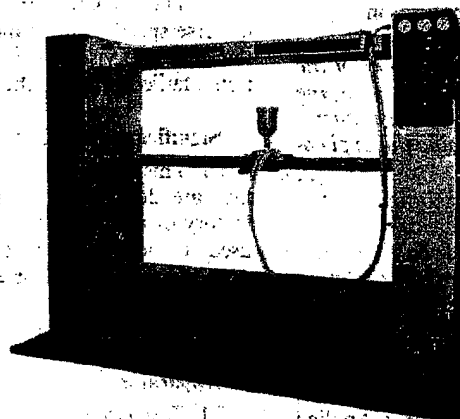
³ Machines suitable for this purpose are manufactured by Eclipse Systems, Inc., 12 Cork Hill Rd., Franklin, NJ 07416; Spraymation, Inc., 5320 N.W. 35th Ave., Ft. Lauderdale, FL 33309-6314.



(a) Fixed Gun, Travelling Panel Machine



(b) Fixed Panel, Travelling Gun Machine



(c) Fixed Panel Programmable Indexing Travelling Gun Machine

FIG. 1. Automatic Spray Machines, Method A

spray pattern at least 6 in. (150 mm) in width is satisfactory. The gun may be triggered manually or automatically.

5.4 *Pressure Gage*, covering the range of 0 to 100 psi (0 to 690 kPa).

5.5 *Air Pressure Regulator*.

5.6 *Air Supply*, oil-free, under pressure.

6. Preparation of Apparatus

6.1 Mount the spray gun on the automatic spray machine. Connect the air line hose from the regulator to the air pressure gage which in turn is connected to the air inlet of the spray gun.

6.2 Set the gun so that its tip is at the desired distance from the test panel surface, usually in the range from 8 to 12 in. (200 to 300 mm).

6.3 With the gun trigger fully open, adjust the air regulator to provide the desired reading on the air pressure gage.

NOTE 2—A suitable air pressure is usually from 40 to 75 psi (275 to 520 kPa).

6.4 Set the automatic spray machine controls to provide the desired traverse speed of the panel holder or the gun mount, whichever is pertinent to the type of machine being used.

NOTE 3—Suitable traverse speeds for automotive coatings usually range from 700 to 900 in./min (17.5 to 22.5 m/min).

7. Procedure

7.1 Strain the material to be sprayed into the container to be used with the spray gun. Reduce the material to a viscosity suitable for spraying.

7.2 Connect the container to the gun and test the spray gun operation while stationary, for correct spray pattern and uniformity by allowing a momentary spray to be deposited on a piece of paper placed in the panel position. Adjust the air pressure material flow, and spray fan width controls until the desired pattern and uniformity are obtained. Further refinements may be made in the spray pattern by modifying

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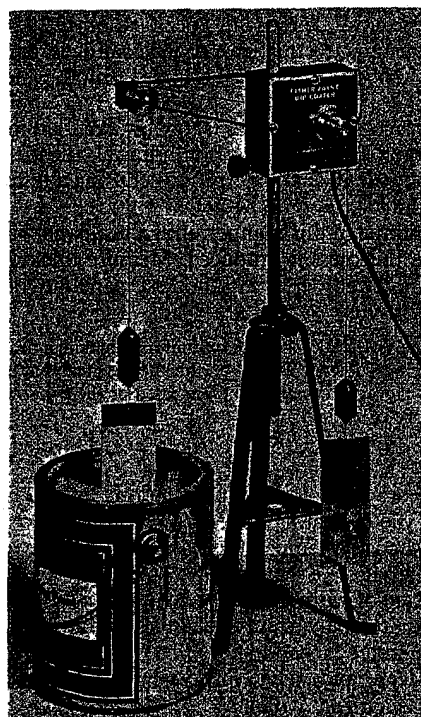
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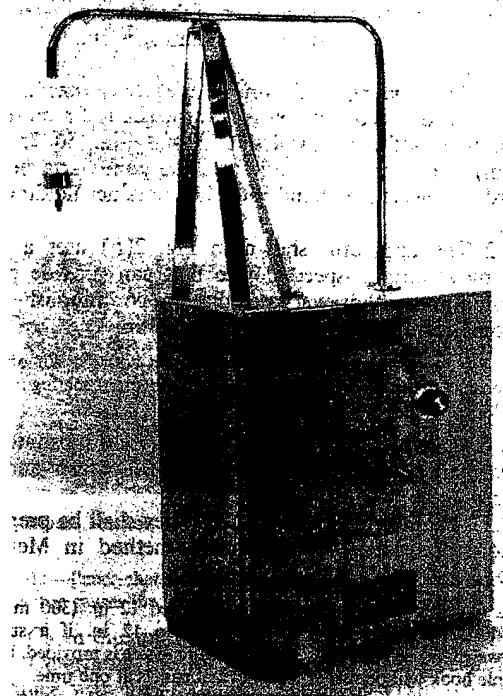
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(a) Dip-Coater With Motor-Driven Step-Cone Pulley



(b) Dip-Coater With Continuously Variable Speed Drive

FIG. 2 Dip-Coater

the air pressure, the type of thinning agent, and the consistency of the material.

NOTE 4—The width of the spray pattern should be considerably wider than the width of the test panel to assure spray uniformity on the test panel.

7.3 Place a test panel on the panel holder and start the machine. Operate the spray gun so that it will begin spraying a few seconds before the test panel enters the spray pattern and continue spraying a few seconds after the test panel leaves the spray pattern.

7.4 Remove the coated panel and bake, force-dry, or air-dry it, in accordance with its type, in a vertical position in a dust-free atmosphere, as described in Specification D 3924.

7.5 Determine the thickness of the coating in accordance with Test Methods D 1005, D 1186, D 1212, or D 1400, whichever is appropriate.

8. Report

8.1 Report the following information:

8.1.1 Type of coating material,

8.1.2 Viscosity and percent of nonvolatile coating material,

8.1.3 Distance of test panel from gun tip,

8.1.4 Air pressure,

8.1.5 Number of spray passes,

8.1.6 Traverse speed,

8.1.7 Temperature and relative humidity at time of application, and

8.1.8 Film thickness values obtained for applied coating.

9. Precision

9.1 The precision of preparing coated panels by this test method depends on the type of coating applied and its thickness.

9.2 For most types of coatings, individual thickness readings taken over the surface of the applied coating can be expected to deviate from the mean by no more than $\pm 5\%$.

METHOD B—MOTOR-DRIVEN DIP COATER APPLICATION

10. Summary of Test Method

10.1 A motor-driven device is employed to withdraw the test panel from a container of the coating material at a desired uniform rate.

10.2 The thickness of coating applied is controlled by the speed of panel withdrawal, the viscosity of the material, and the percent of solids in the material.

11. Significance and Use

11.1 This test method is limited to those materials that flow out to smooth films when test panels are dipped into the material and withdrawn.

12. Apparatus

12.1 *Dip Coater*,⁴ consisting of a mechanism that will withdraw a panel from a container of the coating material at a predetermined rate. Suitable apparatus, is shown in Fig. 2(a) and 2(b):

12.1.1 The apparatus shown in Fig. 2(a) uses a cord wound around a step-cone pulley on the shaft of a motor to provide panel withdrawal rate of 2, 3, and 4-in. (50, 75, and 100-mm)/min. Prior to withdrawal, the panel, attached to the cord, is lowered by hand into the container holding the material.

12.1.2 The apparatus shown in Fig. 2(b) uses a cord driven by a variable-speed device that can provide panel immersion and withdrawal rates that are continuously variable from 2.5 to 20 in. (65 to 510 mm)/min.

NOTE 5—Rectangular containers (F-style can with lid cut off) are useful because the smaller exposed surfaces of the liquid coating reduces volatile loss.

12.2 *Test Panels*, of any clean, smooth, rigid substrate of a size that can be accommodated by the dip coater and the container.

12.2.1 When steel panels are used they shall be prepared in accordance with the appropriate method in Methods D 609.

NOTE 6—The test panels should not exceed 12 in. (300 mm) in length, but the width may be varied up to 12 in. if a suitable counterweight is used and a dip tank of adequate size is provided. Use of a multiple hook will permit dipping several panels at one time.

13. Procedure

13.1 Adjust the coating material to the proper percentage of solids and viscosity. Measure the temperature of the material in the container at the time of application.

NOTE 7—The operating conditions (viscosity, percent of nonvolatile matter, and rate of withdrawal) are specific for a given coating material and film thickness and need to be determined by trial. Subsequent reproduction of the same operating conditions should give the same film thickness. Data are available⁵ on a variety of materials and film thickness to indicate the range required. The viscosity range for normal film thickness of 0.5 to 2.0 mil (13 to 50 mm) has been shown to be 1 to 2.5 P.

13.2 Place the prepared test panel on the hook attached to the cord and lower it into the container holding the coating material. Wind the cord once completely around the pulley of the correct size to give the desired rate of withdrawal.

13.2.1 For the stepped-cone pulley apparatus, wind the cord once completely around the pulley of the correct size to give the desired weight of withdrawal.

13.2.2 For the continuously variable speed apparatus set the desired panel immersion and withdrawal rates on the control panel.

13.3 Start the motor and withdraw the panel at the desired rate, with a smooth movement entirely free of

vibration. Bake, force-dry, or air-dry the coated panel, in accordance with its type, in a vertical position in a dust-free atmosphere in accordance with Specification D 3924.

13.4 Determine the thickness of the coating in accordance with Test Methods D 1005, D 1186, or D 1400, whichever is appropriate.

13.5 If the coating thickness is too low, coat another panel using a slower rate of panel withdrawal. If the coating thickness is too high, coat another panel using a faster rate of panel withdraw.

13.6 Continue in this manner until a test panel having the desired film thickness is produced. Measure thickness on at least three different areas of the test panel to determine coating uniformity.

NOTE 8—With the dip coater, non-uniform thickness on a panel is frequently obtained. Hence, if the film thickness is greater at the bottom than the top, the viscosity should be increased or the panel withdrawal speed should be reduced, or both.

14. Report

14.1 Report the following information:

14.1.1 Type of coating material,

14.1.2 Viscosity, temperature, and percent nonvolatile of coating material,

14.1.3 Rate of withdrawal,

14.1.4 Air temperature and relative humidity at time of application, and

14.1.5 Mean and range of dry film thickness values obtained.

15. Precision

15.1 The precision of preparing coated panels by this test method depends on the type of coating applied and its thickness.

15.2 For most types of coatings, individual thickness readings taken over the surface of the applied coating can be expected to deviate from the mean by no more than $\pm 5\%$.

METHOD C—MOTOR-DRIVEN BLADE FILM APPLICATION

16. Summary of Test Method

16.1 A uniform film is produced by an applicator blade that is pushed across the test panel at a uniform speed by a motor-driven device.

16.2 The thickness of coating applied is controlled by the clearance of the applicator blade and the viscosity and percentage of solids of the material.

17. Significance and Use

17.1 This test method is applicable to substrates consisting of smooth rigid materials, such as metal or glass, and of non-rigid materials, such as paper charts. It is more reliable for producing uniform films than is the use of hand-held draw-down applicators.

18. Apparatus

18.1 *Motor-Driven Blade Film Applicator*,⁶ consisting of a

⁶ Suitable apparatus may be obtained from several suppliers of paint testing equipment.

⁴ Suitable dip coaters are the Garard Dip Coater obtainable from Paul N. Gardner Co., Inc., 316 N.E. First St., P.O. Box 10688, Pompano Beach, FL 33061-6688 and the Dipcoater obtainable from Technical Equipment Co., P.O. Box 208, Willoughby, OH 44094.

⁵ Information covering viscosity, percent of solids, rates of withdrawal and film thickness for a variety of finishing materials is given in the paper by Payne, H. F., "The Dip Coater, An Instrument For Making Uniform Films by the Dip Method," *Industrial and Engineering Chemistry, Analytical Edition*, Vol 15, 1943, p. 48.

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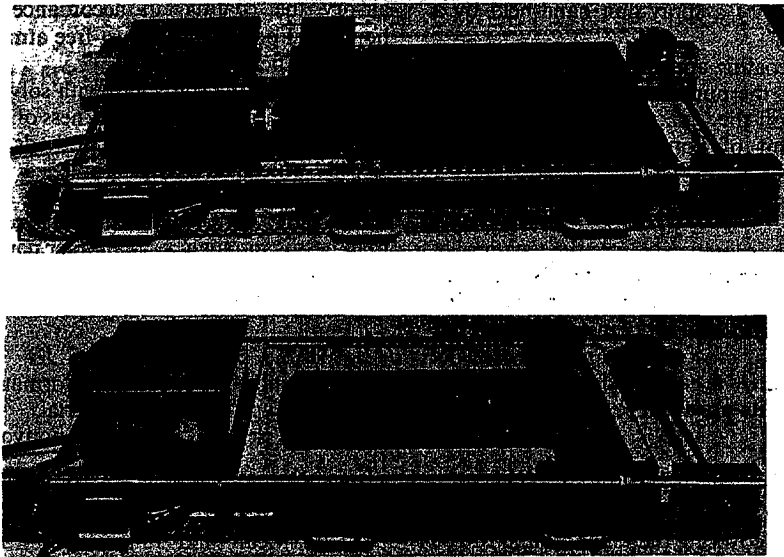


FIG. 3 Blade Film Applicator, Motor-Driven, Method C

base plate, a bar for holding an applicator blade, and a driving mechanism. The base plate shall hold paper charts flat by means of a vacuum. The blade holder shall be designed to accommodate common types of applicator blades and to accept weights for loading the applicator blade. A mechanism shall be provided to stop the blade movement automatically at the end of the draw-down. A suitable apparatus is shown in Fig. 3.

18.2 *Vacuum Source*, a vacuum pump or a water aspirator.

18.3 *Applicator Blade*, any common type, either with adjustable or fixed clearances.

18.4 *Test Panels*, any clean, smooth, rigid substrate or may be paper charts or similar materials.

NOTE 9—Rigid panels shall be cleaned in an approved manner. Steel panels shall be prepared in accordance with the appropriate method in Test Method D 609.

19. Procedure

19.1 Clean the base plate and place the test panel on it.

19.2 If a vacuum is needed to hold the test panel flat, connect the vacuum source to the base plate and turn it on.

NOTE 10—When films are being applied to paper charts or tin foil, a sheet of paper should first be placed on the vacuum plate to prevent formation of dimples at the plate perforations.

19.3 Select an applicator blade having a clearance that should provide a wet film thickness that will give the desired dry film thickness; or, if specified, the required wet film thickness. Insert the blade in the blade holder and load the holder with weights if needed.

19.4 Place a suitable amount of the coating material on the test panel in front of the blade. Start the motor-drive and coat the test panel.

19.5 Remove the coated panel and bake, force-dry, or air-dry the coating, in accordance with its type, in a horizontal position in a dust-free atmosphere in accordance with Specification D 3924.

NOTE 11—Paper charts should be taped down to prevent curling of the edges that causes the wet film to flow towards the center.

19.6 Clean the applicator blade.

19.7 Determine the thickness of the applied coating in accordance with Test Method D 1005, D 1186, or D 1400 whichever is appropriate.

19.8 If the coating thickness is too low, coat another panel using a larger blade clearance. If the coating thickness is too high, coat another panel using a smaller blade clearance.

19.9 Continue in this manner until a test panel having the desired dry film thickness is produced. Measure thickness on at least three different areas of the test panel to determine coating uniformity.

20. Report

20.1 Report the following information:

20.1.1 Type of coating material,

20.1.2 Viscosity and percentage of solids of coating material;

20.1.3 Clearance of applicator blade used,

20.1.4 Air temperature and relative humidity at time of application, and

20.1.5 Mean and range of the film thickness values obtained.

21. Precision

21.1 The precision of preparing coated panels by this test method depends on the type of coating applied and its thickness.

21.2 For most types of coatings, individual thickness readings taken over the surface of the applied coating can be expected to deviate no more than $\pm 5\%$ from the mean.

METHOD D—HAND-HELD SPRAY GUN APPLICATION

22. Summary of Test Method

22.1 A uniform film of a coating material is produced on

a test panel by the means of a spray gun hand-held by a person skilled in its use.

22.2 The thickness of coating applied is controlled by the traverse speed of the gun, the number of passes of the gun, the fluid delivery rate of the gun, the viscosity of the material, and the amount of nonvolatile matter in the material.

23. Significance and Use

23.1 This test method is applicable to the coating of substrates consisting of smooth, rigid materials, such as metal or glass. It is usually less reliable for producing uniform films than is the automatic spray method. However, films sufficiently uniform for most physical property tests of materials can be produced by a hand-held spray gun operated by a person skilled in its use.

24. Apparatus

24.1 *Spray Gun*, any that will provide a uniform fan-type spray pattern at least 6 in. (150 mm) in width is satisfactory.

24.2 *Air Pressure Gage*, covering the range from 0 to 100 psi (0 to 690 kPa).

24.3 *Air Pressure Regulator*.

24.4 *Air Supply*, oil-free and under pressure.

24.5 *Panel Holder*.

25. Preparation of Apparatus

25.1 Connect the air line hose from the regulator to the air pressure gage which in turn is connected to the air inlet of the spray gun.

25.2 With the trigger fully open, adjust the air regulator to provide the desired reading on the air pressure gage.

NOTE 12—A suitable air pressure is usually from 40 to 75 psi (275 to 520 kPa).

26. Procedure

26.1 Strain the material to be sprayed into the container to be used with the spray gun. Reduce the material to a viscosity suitable for spraying.

26.2 Connect the container to the gun and test the spray gun operation for correct spray pattern and uniformity by allowing a momentary spray to be deposited on a piece of paper placed in the panel position. Adjust the air pressure, material flow, and spray width controls until the desired pattern and uniformity are obtained. Further refinements may be made in the spray pattern by modifying the air pressure, the type of thinning agent, and the consistency of the material.

NOTE 13—The width of the spray pattern should be considerably wider than the width of the test panel to assure spray uniformity on the test panel.

26.3 Place a test panel on the panel holder. Hold the spray gun tip 10 to 12 in. (250 to 300 mm) from the test panel surface and trigger the spray gun. Begin by aiming the gun at right angles to the panel so that the spray pattern is just beyond the edge of the panel. Gradually make the spray pattern move across the panel at a traverse speed of 10 to 15 in. (250 to 400 mm)/s.

26.4 Reverse the traverse direction of the spray gun and make the spray pattern move across the panel again.

26.5 Remove the panel from its holder and bake or

air-dry the coating, in accordance with its type, in a horizontal position in a dust-free atmosphere in accordance with Specification D 3924.

26.6 Clean the spray gun with solvent.

26.7 Measure the film thickness of the dry applied coating in accordance with Test Methods D 1005, D 1186, or D 1400 whichever is appropriate.

26.8 If the coating thickness is too low, coat another panel using more passes of the spray pattern. If the coating thickness is too high, coat another panel with fewer passes of the spray pattern or, if this is not feasible, reduce the material further with thinner.

27. Report

27.1 Report the following information:

27.1.1 Type of coating material,

27.1.2 Viscosity and percent nonvolatile of coating material,

27.1.3 Temperature and relative humidity at time of application, and

27.1.4 Mean and range of dry film thickness values obtained.

28. Precision

28.1 The precision of preparing coated panels by this test method depends on the type of coating applied, and its thickness and the skill of the operator.

28.2 For most types of coatings, individual thickness readings taken over the surface of the applied coating can be expected to deviate from the mean by no more than $\pm 10\%$ if the coating has been applied by a skilled operator.

METHOD E—HAND-HELD BLADE FILM APPLICATION

29. Summary of Test Method

29.1 A uniform film of a coating material is produced on a test panel by the means of a hand-held applicator blade.

29.2 The thickness of coating applied is controlled by the rate at which the applicator blade is drawn across the test panel, the viscosity of the material, the amount of nonvolatile matter in the material, and the clearance of the blade.

30. Significance and Use

30.1 This test method is applicable to the coating of substrates consisting of smooth, rigid materials such as metal or glass. It is applicable to the coating of smooth cardboard and paper charts if some means is used to assure that these substrates are held flat.

30.2 This test method is usually less reliable for producing uniform films than is the motor-driven applicator blade method. However, films sufficiently uniform for most physical property tests of materials can be produced by a hand-held applicator blade operated by a person skilled in its use.

31. Apparatus

31.1 *Film Applicator Blade*, any common type, either with adjustable or fixed clearances.

31.2 *Auxiliary Flattening Bar*, precision ground.

31.3 *Test Panels*, any clean, smooth, rigid substrates or may be paper charts or other similar materials.

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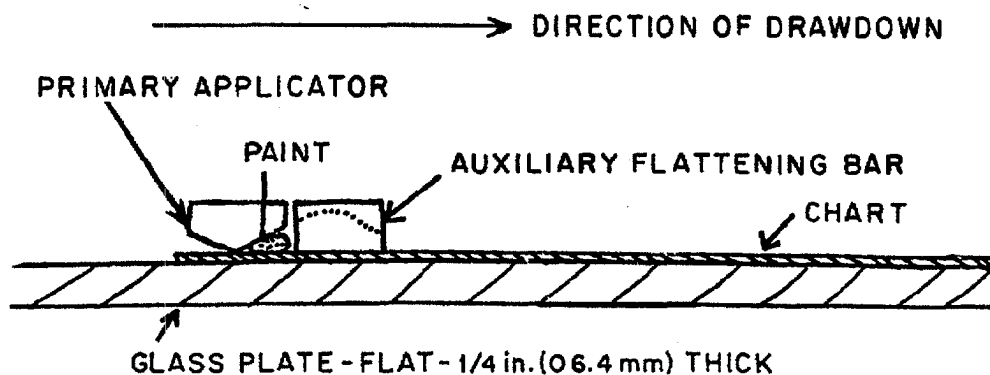


FIG. 4 Use of Auxiliary Flattening Bar

32. Procedure

32.1 Select an applicator blade that has a clearance that will provide a wet film thickness that should result in the desired film thickness.

32.2 For coating rigid substrates:

32.2.1 Position the applicator blade near the edge of the panel and place a pool of the liquid material in front of it.

32.2.2 Grasp the sides of the applicator with the fingers and pull it across the panel at a speed of about 10 to 12 in. (250 to 300 mm)/s.

32.3 For coating non-rigid substrates:

32.3.1 Position the applicator blade near the edge of the panel and place a pool of the liquid material in front of it. Place the auxiliary bar in front of the pool of material (see Fig. 4).

32.3.2 Grasp the sides of the applicator with the fingers and pull it across the panel at a speed of about 10 to 20 in. (250 to 500 mm)/s. As the auxiliary bar is pushed by the applicator, it should press the substrate flat adjacent to the advancing edge of the applicator.

32.4 Bake or air-dry the applied coating, in accordance with its type in a horizontal position in a dust free atmosphere in accordance with Specification D 3924.

32.5 Clean the applicator bar with solvent.

32.6 Measure the film thickness of the dry-applied coating in accordance with Test Methods D 1005, D 1186, or D 1400 whichever is appropriate.

32.7 If the coating thickness is too low, select an applicator blade with a greater clearance and coat another panel. If the coating thickness is too high, select an applicator blade with a smaller clearance and coat another panel.

33. Report

33.1 Report the following information:

33.1.1 Type of coating material,

33.1.2 Viscosity and percent nonvolatile of coating material,

33.1.3 Clearance of applicator blade used,

33.1.4 Air temperature and relative humidity at time of application, and

33.1.5 Mean and range of the film thickness values obtained.

34. Precision

34.1 The precision of preparing coated panels by this test method depends on the type of coating applied, its thickness and the skill of the operator.

34.2 For most types of coatings, individual thickness readings taken over the surface of the applied coating can be expected to deviate from the mean by no more than $\pm 10\%$ if the coating has been applied by a skilled operator.

35. Keywords

35.1 automatic spray; blade applicator; dip coater; organic coatings

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Standard Test Method for Evaluating Degree of Bleeding of Traffic Paint¹

This standard is issued under the fixed designation D 868; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout, including the title, in October 1989.

1. Scope

1.1 The photographic reference standards that are part of this test method² are representative of the degrees of bleeding of traffic or pavement marking paints. The standards are intended primarily for comparative evaluation in the laboratory.

1.2 Bleeding as defined is the only type of discoloration in this test method.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standard:

D 969 Test Method for Laboratory Determination of Degree of Bleeding of Traffic Paint³

2.2 Adjunct:

D 868 Bleeding resistance of paint (one photo)²

3. Terminology

3.1 Description of Term Specific to This Standard:

3.1.1 *bleeding characteristic*—that condition of discoloration manifested in traffic paint when applied to tar or asphaltic-type substrates. The number assigned to evaluate the degree of bleeding failure represents in these reference standards a measure of the contrast between the color of the

dry film on a nonbleeding surface and the color of the dry film on the test surface.

4. Significance and Use

4.1 Solvents in a traffic paint may cause bleeding of pavement constituents into the traffic marking, thereby rendering the traffic marking less effective as a lane or directional indicator. This test method in conjunction with the method for panel preparation in Test Method D 969 is used to evaluate such bleeding properties. The evaluation is very subjective and raises questions as to the usefulness of the results for specification compliance.

5. Use of Photographic Reference Standards

5.1 The photographic reference standards that are part of this test method² are representative of the degrees of bleeding of traffic or pavement marking paints. The photograph in Fig. 1 is for illustration purposes only and should not be used for evaluation.

5.2 The use of the photographic reference standards shown in Fig. 1 should be as follows:

5.3 In preparing films to compare with the reference standards for evaluating the extent of bleeding, the paint shall be applied to:

5.3.1 A nonbleeding reference surface, and

5.3.2 The test surface.

5.4 The paint under test shall be applied at a film thickness sufficient to ensure complete hiding.

5.5 The contrast in color between the films over the nonbleeding reference surface and the test surface shall be compared with the reference standards in Fig. 1 for a numerical rating.

NOTE—The degree of bleeding must necessarily be a function of both the bleeding surface agreed upon and the initial color of the paint. For instance, a yellow paint in general would be expected to show less bleeding than a white paint since its initial color is darker and as a consequence the discoloration effect of the bleeding surface upon this darker color will be minimized.

6. Precision and Bias

6.1 *Precision*—Due to the poor precision of this test method, if it is used in a specification, the permissible deviation from the maximum specified value should be agreed upon between the purchaser and the seller.

6.2 *Bias*—Bias cannot be determined.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved Nov. 29, 1985. Published January 1986. Originally published as D 868 - 46 T. Last previous edition D 868 - 48 (1981)^{e1}.

² Copies of the pictorial photographic reference standards are contained in the publication *Pictorial Standards of Coating Defects*, and may be obtained from ASTM Headquarters (request Adjunct PCN 12-408680-00) or the Federation of Societies for Coating Technology, 492 Norristown Rd., Blue Bell, PA 19422. The original source of the photographic reference standards illustrated in Fig. 1 is the Federation.

³ *Annual Book of ASTM Standards*, Vol. 06.01.

TRAFFIC PAINT COLOR BLEEDING STANDARDS

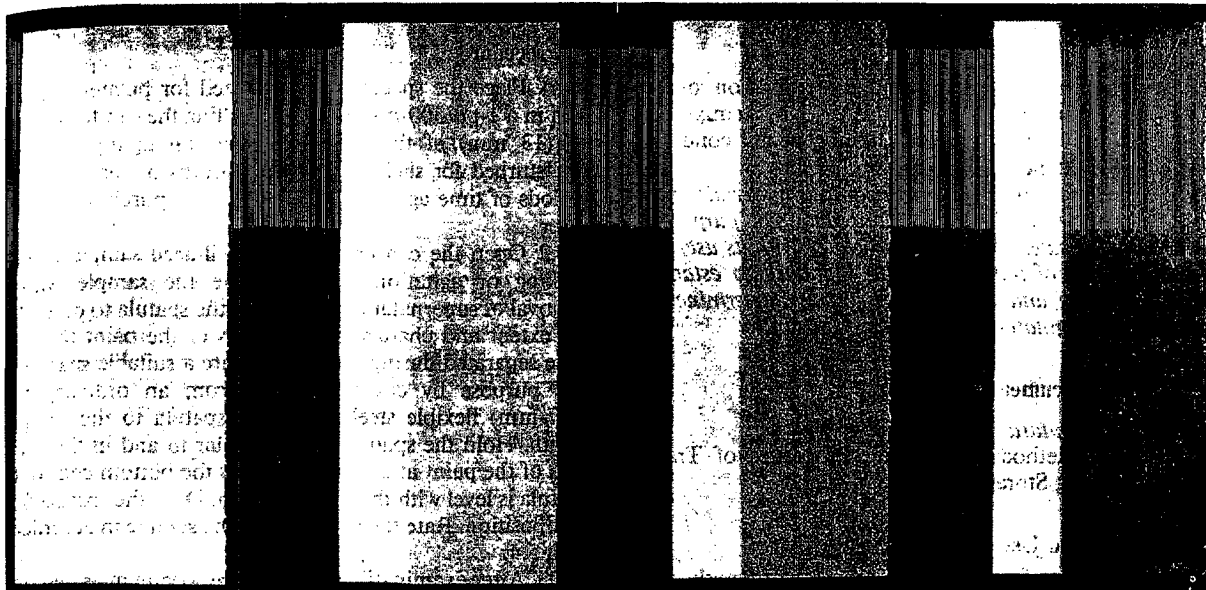


FIG. 1 Degrees of Bleeding

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Standard Test Method for Evaluating Degree of Settling of Paint¹

This standard is issued under the fixed designation D 869; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense to replace Method 4208 of Federal Test Method Standard No. 141A. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Editorial changes were made throughout, including the title, in October 1989.

1. Scope

1.1 This test method covers the determination of the degree of pigment suspension and ease of remixing a shelf-aged sample of paint to a homogeneous condition suitable for the intended use.

1.2 *This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

- 2.1 *ASTM Standard:*
D1309 Test Method for Settling Properties of Traffic Paints During Storage²

3. Significance and Use

3.1 Paints, if not formulated or processed properly may settle excessively. Paint that settles excessively is difficult to reincorporate into the paint system causing time delays or valuable pigment being left in the drum. This test method is an attempt to evaluate the degree of settling caused by the accelerated Test Method D 1309. This very subjective method of evaluation in conjunction with the variables of Test Method D 1309 raises questions as to the usefulness of the results for specification compliance.

4. Apparatus

4.1 *Container*, standard 1-pt (500-mL) friction-top can paint container, $3\frac{3}{8} \pm \frac{1}{16}$ in. (85.5 $\pm$ 1.5 mm) in diameter, and $3\frac{7}{8} \pm \frac{1}{16}$ in. (98.5 $\pm$ 1.5 mm) in height.

4.2 *Spatula*, weighing 45 ± 1 g with square-end blade $4\frac{3}{4}$ in. (125 mm) in length and approximately $1\frac{3}{16}$ in. (20 mm) in width, shall be used to examine the paint for pigment settling and reincorporation characteristics.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved Sept. 27, 1985. Published November 1985. Originally published as D 869 - 46 T. Last previous edition D 869 - 78.

² *Annual Book of ASTM Standards*, Vol 06.01.

5. Procedure

5.1 Place the specimen to be tested for pigment suspension in a 1-pt (500-mL) container, filling the can to within $\frac{1}{2}$ in. (13 mm) of the top. Close the can tightly and hold undisturbed for shelf aging for 6 months or for such other periods of time agreed upon between the purchaser and the seller.

5.2 Open the can holding the shelf-aged sample without shaking or agitation, and examine the sample without removal of supernatant vehicle. Use the spatula to determine the extent and character of portions of the paint that may have separated during storage. Prepare a suitable spatula for this purpose by cutting the tip from an ordinary 5-in. (127-mm) flexible steel laboratory spatula to the specified length. Hold the spatula perpendicular to and in the center area of the paint at a height whereby the bottom edge of the spatula is level with the top of the can. Drop the spatula from that position. Rate the condition of the sample in accordance with 5.4.

5.3 After examination of the entire specimen as described in 5.2, if a portion of the pigment has separated out to form a firm cake at the bottom of the container pour the supernatant portion of the liquid off into a clean container and hold for subsequent use. Reincorporate the separate cake by hand stirring with the spatula, adding back the liquid in small amounts until the pigment has been reincorporated to form a homogeneous paint suitable for the intended use or until it is determined that the pigment cake cannot be reincorporated by hand stirring. Rate the condition of the specimen in accordance with 5.4.

5.4 Rate the sample for degree of settling on a scale from 10 to 0 in accordance with the following. Give intermediate conditions the appropriate odd number.

Rating	Description of Paint Condition
10	Perfect suspension. No change from the original condition of the paint.
8	A definite feel of settling and a slight deposit brought up on spatula. No significant resistance to sidewise movement of spatula.
6	Definite cake of settled pigment. Spatula drops through cake to bottom of container under its own weight. Definite resistance to sidewise motion of spatula. Coherent portions of cake may be removed on spatula.
4	Spatula does not fall to bottom of container under its own weight. Difficult to move spatula through cake sidewise and slight edgewise resistance. Paint can be remixed readily to a homogeneous state.
2	When spatula has been forced through the settled layer it is very difficult to move spatula sidewise. Definite edgewise resistance to movement of spatula. Paint can be remixed to a homogeneous state.

Rating	Description of Paint Condition
0	Very firm cake that cannot be reincorporated with the liquid to form a smooth paint by stirring manually.

method, if it is used in a specification, the maximum deviation from the settling limits specified should be agreed upon between the purchaser and the seller.

6.2 *Bias*—Bias cannot be determined.

6. Precision and Bias

6.1 *Precision*—Due to the poor precision of this test

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Standard Practice for Testing Water Resistance of Coatings Using Water Immersion¹

This standard is issued under the fixed designation D 870; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the basic principles and operating procedures for testing water resistance of coatings by the partial or complete immersion of coated specimens in distilled or de-mineralized water at ambient or elevated temperatures. Although the apparatus and procedure could be employed in immersion tests using solutions of various materials in water, this practice is limited to tests in water alone.

1.2 This practice is limited to the methods of obtaining, measuring, and controlling the conditions and procedures of water immersion tests. It does not specify specimen preparation, specific test conditions, or evaluation of results.

NOTE 1—Alternative practices for testing the water resistance of coatings include Practices D 1735, D 2247, and D 4585.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, Lacquer, and Related Products on Test Panels²
- D 1193 Specification for Reagent Water³
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴
- D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus²
- D 2247 Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity²

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Nov. 27, 1987. Published January 1988. Originally published as D 870 - 46 T. Last previous edition D 870 - 86a.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁴ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

D 2616 Test Method for Evaluation of Visual Color Difference with a Gray Scale⁵

D 3359 Test Methods for Measuring Adhesion by Tape Test²

D 3363 Test Method for Film Hardness by Pencil Test²

D 4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation²

3. Summary of Practice

3.1 Coated specimens are partially or wholly immersed in water in a container that is resistant to corrosion. The exposure conditions are varied by selecting: (a) the temperature of the water, and (b) the duration of the test. Water permeates the coating at rates that are dependent upon the characteristics of the coating and upon the temperature of the water. Any effects such as color change, blistering, loss of adhesion, softening, or embrittlement are observed and reported.

4. Significance and Use

4.1 Immersion in water can cause the degradation of coatings. Knowledge on how a coating resists water immersion is helpful in predicting its service life. Failure in a water immersion test may be caused by a number of factors including a deficiency in the coating itself, contamination of the substrate, or inadequate surface preparation. The test is therefore useful for evaluating coatings alone or complete coating systems.

4.2 Water immersion tests are used for research and development of coatings and substrate treatments, specification acceptance, and quality control in manufacturing. These tests typically result in a pass or fail determination, but the degree of failure may also be measured. A coating system is considered to pass if there is no evidence of water-related failure after a specified period of time.

4.3 Results obtained from water immersion tests in accordance with this practice should not be represented as being equivalent to a period of exposure to water in the natural environment, until the degree of quantitative correlation has been established for the coating or coating system.

4.4 Corrosion tests of a scribed coating on a ferrous substrate are impractical in water immersion tests as the corrosion products tend to contaminate the water bath. Continuous overflow of the test tank is sometimes required to maintain consistent water quality.

4.5 The apparatus for the test is relatively inexpensive and can be as simple as a glass beaker with a stirrer. It is also

⁵ Annual Book of ASTM Standards, Vol 14.02.

possible to use very large tanks for testing coated pipe or other large coated products.

5. Apparatus

5.1 *Tank*, constructed of corrosion-resistant materials. Heating units, if required, should preferably be located outside the tank.

5.2 *System*, for circulation of the water with pressurized air, a stirrer, or a pump.

5.3 *Supports for the Test Specimens*, constructed of non-conductive materials to hold the specimens 30 mm apart and 30 mm from the bottom and sidewalls of the tank.

NOTE 2—The circulating system is for the purpose of exposing all the water in the tank to room air so the water does not become stratified and oxygen-depleted at the lower levels.

6. Test Specimens

6.1 This practice does not cover the preparation of test specimens. The substrate composition and surface preparation, specimen preparation, and the number of specimens should be agreed upon prior to testing.

NOTE 3—Applicable methods for the preparation of test panels and substrates are given in Method D 609 and Practice D 1730. Test Methods D 823 covers application techniques for the production of uniform films.

7. Procedure

7.1 Fill the tank with water to a depth where the test specimens are immersed for approximately three-quarters of their length. Reagent water conforming to Type IV of Specification D 1193 is suitable for use.

7.2 Heat the water to the desired temperature with the circulating system in operation. If no temperature is specified, heat the water to $100 \pm 2^\circ\text{F}$ ($38 \pm 1^\circ\text{C}$). Maintain the temperature throughout the test.

7.3 Place the test specimens in the tank so that the plane of the specimens is parallel to the flow of water in the tank. Protect the edges and backs of the specimens from corrosion

if these surfaces are not to be tested.

7.4 Replace the water if it becomes cloudy or colored. Continuous replacement of the water is permitted.

7.5 Conclude the test after a specified period of time or after effects from water immersion have been observed.

7.6 Wipe the test specimens dry. Rate specimens for changes in color, blistering, etc. Evaluate specimens no less than 5 min and no more than 10 min after removal from test, as the effects from water exposure can change within a short time. Remove only as many specimens as can be rated within the specified time.

NOTE 4—The 0 to 10 scale described in ASTM STP 500⁶ is preferred for rating. Relevant procedures for evaluating water effects are described in Test Methods D 610, D 714, D 2616, D 3359, and D 3363.

7.6.1 If possible, rate the specimens again after they have been removed from the test for a recovery period long enough that moisture absorbed within the specimens dries out and the specimens reach moisture equilibrium with room air. A recovery period from 12 to 24 h is generally sufficient. The post-recovery rating allows evaluation of the permanent effects of the exposure as distinct from the transient effects, and is especially important for evaluation of color and gloss.

8. Report

8.1 Report the following information:

8.1.1 Sample identification.

8.1.2 Results of the evaluation(s).

8.1.3 Reference to Practice D 870.

8.1.4 Hours of test duration.

8.1.5 Test temperature.

8.1.6 Brief description of water quality and water replacement procedure.

8.1.7 Special conditions of test or any deviations in test procedure.

⁶ *Paint Testing Manual, ASTM STP 500, ASTM, 1972.*

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Standard Test Method for Evaluating Degree of Resistance to Wear of Traffic Paint¹

This standard is issued under the fixed designation D 913; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the evaluation of degree of resistance to wear that may occur with traffic paints in road tests (see Practice D 713) or in actual service, using photographic standards for comparative evaluation.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 713 Practice for Conducting Road Service Tests on Fluid Traffic Marking Materials²

3. Description of Term Specific to This Standard

3.1 *failure, described by these reference standards*—that condition manifested in traffic paint by actual detachment of entire sections of the film from its substrate or from paint previously applied. The degree of resistance to failure is judged by the amount of substrate that is covered.

4. Significance and Use

4.1 This test method is designed to evaluate the resistance to wear of a traffic paint. It must be remembered that a high degree of performance of paint applied to a bare road surface may not guarantee similar results when the same paint is applied over old paint lines.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved March 25, 1988. Published May 1988. Originally published as D 913 - 47 T. Last previous edition D 913 - 83.

² *Annual Book of ASTM Standards*, Vol 06.01.

5. Type of Failure

5.1 The failure as described in Section 3 does not presume any specific mechanism, and all areas where the substrate is visible shall be considered a failure.

6. Use of Photographic Reference Standards

6.1 The photographic reference standards³ that are part of this test method are representative of the degrees of resistance to wear of stripes of traffic paint. The examples shown in Fig. 1 are for illustration purposes only and should not be used for evaluation.

6.1.1 The degree of resistance to wear is likely to vary over any given area. It is therefore necessary to use one of the following grading methods:

6.1.1.1 Select an area as representative and base the rating of the stripe on this area or

6.1.1.2 Grade segments of the stripe and average these gradings.

6.1.2 The photographic reference standards (Note) represent four degrees (97 %, 92 %, 77 %, 60 %) of resistance to wear. Substrate revealed by failure is readily discernible with the naked eye.

NOTE—The reference standards are representative of stripes of traffic paints. The percentage of surface covered is shown on each reference standard.

7. Procedure

7.1 Compare the representative areas of the traffic paint stripes with the photographic reference standards and estimate the percent of intact film.

8. Report

8.1 Report the mean and range of the substrate coverage estimations, if appropriate.

³ Full-size (8 by 10-in. (203 by 254-mm)) glossy prints of the photographic reference standards showing degrees of chipping are available from ASTM Headquarters. Order PCN 12-409130-00.

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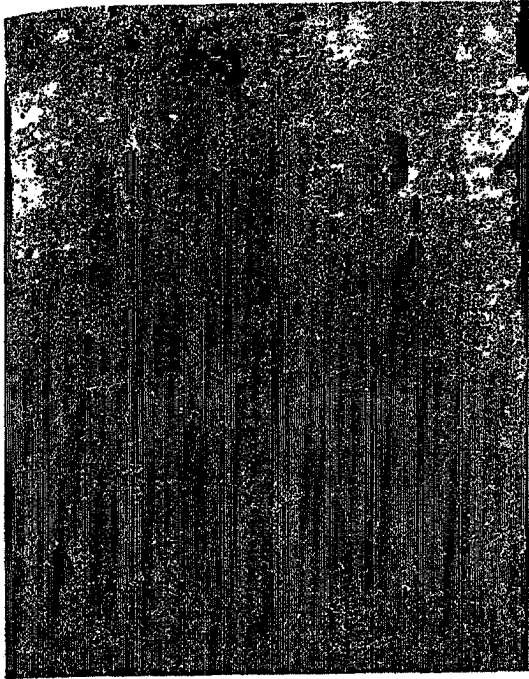
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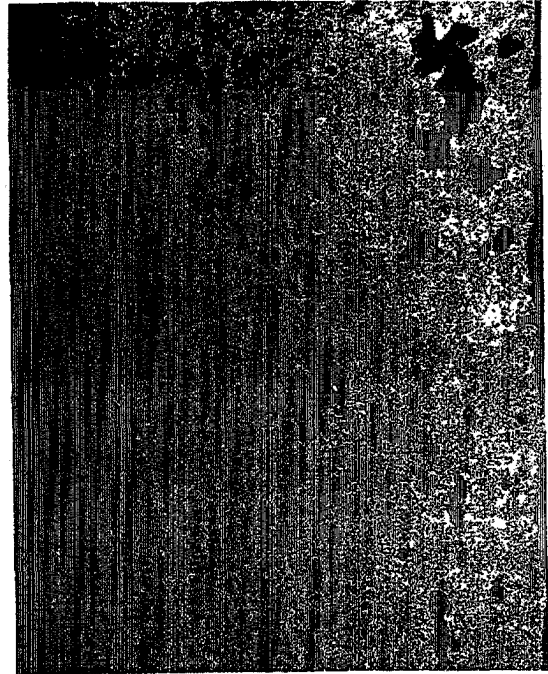
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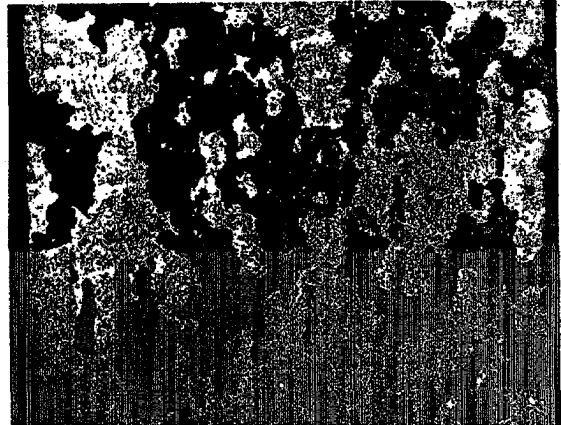
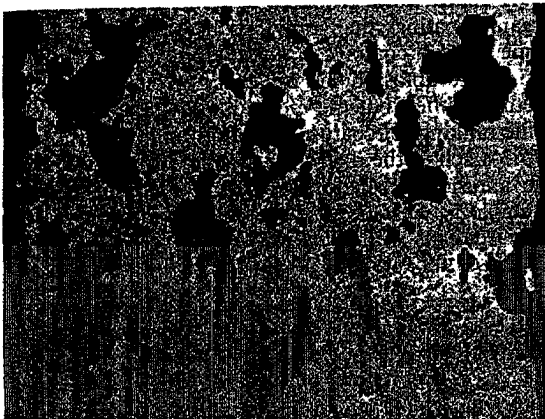
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(Film 92 % Intact)





Standard Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive¹

This standard is issued under the fixed designation D 968; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

¹ NOTE—Keywords were added editorially in January 1991.

1. Scope

1.1 These test methods cover the determination of the resistance of organic coatings to abrasion produced by abrasive falling onto coatings applied to a plane rigid surface, such as a metal or glass panel.

1.2 Two test methods based on different abrasives are covered as follows:

	Sections
Method A—Falling Sand Abrasion Test	1-12
Method B—Falling Silicon Carbide Abrasion Test	13-19

1.3 These methods should be restricted to testing in only one laboratory when numerical values are used because of the poor reproducibility of the methods (see 13.1.2 and 21.1.2). Interlaboratory agreement is improved significantly when ranking is used in place of numerical values.

1.4 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 968 - 48 T. Last previous edition D 968 - 51 (1972).

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.02.

3. Terminology

3.1 Description of Term Specific to This Standard:

3.1.1 *abrasion resistance*—the amount of abrasive required to wear through a unit film thickness of the coating.

4. Summary of Test Methods

4.1 Abrasive is allowed to fall from a specified height through a guide tube onto a coated panel until the substrate becomes visible. The amount of abrasive per unit film thickness is reported as the abrasion resistance of the coating on the panel. Silica sand or silicon carbide may be used, as specified.

5. Significance and Use

5.1 Silica sand produces a slower rate of abrasion for organic coatings than provided by silicon carbide but, for some types of coatings, it provides greater discrimination.

5.2 The abrasion resistance scales produced by the two methods differ, but the methods provide approximately the same rankings of coatings for abrasion resistance.

5.3 Each of the methods has been found useful for rating the abrasion resistance of specific types of coatings. For example Method A (falling sand) has been used for rating floor coatings while Method B (falling silicon carbide) has been used for rating coatings for ship decks.

METHOD A—FALLING SAND ABRASION TEST⁴

6. Apparatus and Materials

6.1 *Abrasion Tester*, as illustrated in Figs. 1 and 2.⁵ A gate for starting the flow of abrasive is located near the top of the guide tube. It consists of a metal disk inserted into a slit in the side of the guide tube with a collar covering the slit. The guide tube shall be firmly supported in a vertical position over a suitable receptacle, which shall contain a support for holding the coated panel at an angle of 45° to the vertical, so that the opening of the tube is directly above the area to be abraded and the distance from the tube to the coated surface face at the nearest point is 1 in. (25 mm) when measured in the vertical direction. The base of the apparatus shall be fitted with adjusting screws for properly aligning the equipment.

⁴ Hipkins, C. C., and Phain, R. J., "The Falling Sand Abrasion Tester," *ASTM Bulletin*, No. 143, December 1946, pp. 18-22.

⁵ A suitable abrasion tester may be obtained from Gardner/BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

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accordance with Test Methods D 1005, D 1400 or D 1186.

8. Standardization

8.1 Pour a quantity of standard sand into the funnel. Examine the sand stream falling from the lower end of the guide tube and align the apparatus by means of the adjusting screws in the base until the inner concentrated core of the sand stream falls in the center of the flow when viewed at two positions at 90° to each other. Introduce a measured volume of sand (2000 ± 10 mL is a convenient amount) and determine the time of efflux. The rate of flow shall be 2 L of sand in 21 to 23.5 s.

8.2 Secure a trial panel in the testing position, as described in 6.1, and introduce the sand in increments until a spot $\frac{5}{32}$ in. (4 mm) in diameter is worn through to the base material. The overall abraded area shall be elliptical in shape, about 1 in. (25 mm) in width and $1\frac{1}{4}$ in. (30 mm) in length. The center of the area of maximum abrasion shall be on the center line through the longer axis of the abraded pattern and within $\frac{9}{16}$ to $1\frac{1}{16}$ in. (14 to 17 mm) of the top edge. Slight final adjustment of the instrument may be required to center the abrasion spot in the pattern. A final check on alignment is made by determining the amount of sand that passes through a $\frac{5}{32}$ -in. (4-mm) hole in a metal panel placed directly under the tube. Place a container under the hole in the panel and allow a weighed amount of sand to pass through the tube onto the panel. Weigh the amount of sand that passed through the hole into the container. The apparatus can be considered to be in calibration if the amount of sand that passed through the hole is 90 to 93 % of the

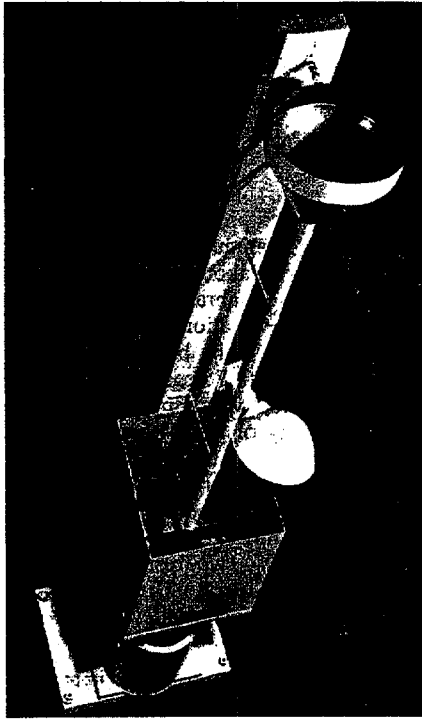


FIG. 1 Apparatus for Falling Sand Abrasion Test

6.2 *Standard Abrasive*—Natural silica sand from the St. Peters or Jordan sandstone deposits (located in the central United States) shall be considered standard when not more than 15 % of the grains in a sample are retained on a No. 20 (850 μ m) sieve and not more than 5% of the grains pass a No. 30 (600 μ m) sieve after 5 min of continuous sieving. The sand is characterized by its roundness of grains and its exceptionally high silicon dioxide content. Use the sieves described in Specification E 11.

NOTE 1—The abrading qualities of sand obtained from different sources may differ slightly, even though the sand meets the sieve requirements. Therefore, for maximum precision of test results, purchaser and seller should use sand from the same source.⁶

7. Test Specimens

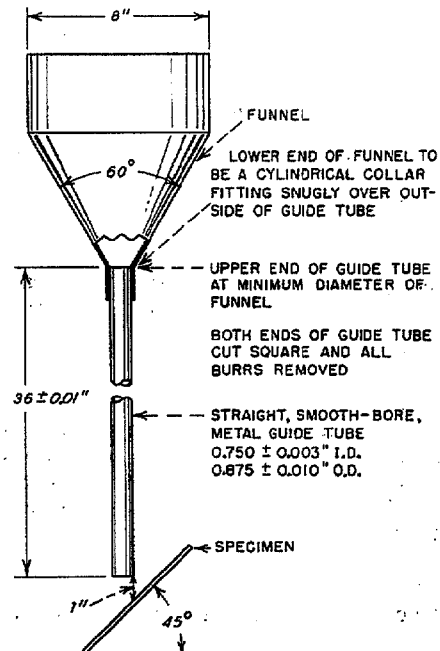
7.1 Apply uniform coatings of the material to be tested to a plane, rigid surface such as a metal or glass panel. Prepare a minimum of two coated panels for the material.

7.2 Cure the coated panels under the conditions of humidity and temperature agreed upon between the purchaser and seller.

NOTE 2—The coatings should be applied in accordance with Test Methods D 823, or as agreed upon between the purchaser and the seller.

NOTE 3—The thickness of the dry coatings should be measured in

⁶Sources of acceptable sand are the Ottawa Silica Co., P.O. Box 577, Ottawa, IL 61350; the Quackenbush Co., 3 West College Dr., Arlington Heights, IL 60004; and the Unimin Corp., 50 Locust Ave., New Canaan, CT. 06840.



NOTE—All dimensions are given in inches. 1 in. = 25.4 mm.

FIG. 2 Design Details of Abrasion Test Apparatus

amount of sand that impinged on the panel.

9. Conditioning

9.1 Unless otherwise agreed upon between purchaser and seller, condition the coated test panels for at least 24 h at $23 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ relative humidity. Conduct the test in the same environment or immediately on removal.

10. Procedure

10.1 Mark off on each coated panel three circular areas, each approximately 1 in. (25 mm) in diameter, and so arranged that each can be properly positioned in the panel support of the abrasion tester. Measure the thickness of the coating by Test Methods D 1005, D 1186, or D 1400 in at least three locations in each area. Record the mean of each set of measurements as the thickness of the coating over the respective area.

10.2 After conditioning, secure the coated panel in the tester as described in 6.1. Adjust the panel so that one of the marked areas will be centered under the guide tube. Pour standard sand, measured volumetrically, into the funnel. Withdraw the gate and allow the sand to flow through the guide tube and impinge on the coated panel. Collect the sand in a container located at the bottom of the tester. Repeat this operation until a $\frac{5}{32}$ -in. (4-mm) diameter area of the coating has worn through to the substrate. A convenient increment of sand to employ during the test is 2000 ± 10 mL. As the end-point is approached, increments of 200 ± 2 mL may be introduced into the funnel.

NOTE 4—When the gate is withdrawn from the guide tube, make certain that a collar covers the slit opening in the tube.

10.3 Abrade each of the remaining marked-off areas of the coated panel as outlined in 9.2.

NOTE 5—Check the alignment of the guide tube at frequent intervals to ensure that the concentrated inner core of the sand stream is falling in the center of the flow.

NOTE 6—After 25 passes through the apparatus, resieve the sand with a No. 30 sieve to remove fines. Replace the sand after 50 passes.

10.4 Repeat 10.1 to 10.3 on at least one additional panel coated with the material under test.

11. Calculation

11.1 For each area of the coated panel tested, calculate the abrasion resistance, A , in litres per mil from the following equation:

$$A = V/T$$

where:

V = volume of abrasive used, L (to one decimal place) and
 T = thickness of coating, mils (to one decimal place).

11.2 Calculate the mean of the abrasion resistance values obtained for different areas of the coated panel and the mean value of the replicate panels.

12. Report

12.1 Report the following information for each coated panel tested:

12.1.1 Temperature and humidity during curing and at the time of testing,

12.1.2 Type and source of abrasive,

12.1.3 Litres of abrasive used for each area tested,

12.1.4 Coating thickness in mils for each area tested,

12.1.5 Abrasion resistance values for each area tested,

12.1.6 Mean abrasion resistance for each coated panel tested, and

12.1.7 Mean abrasion resistance and range of the replicate coated panels.

13. Precision⁷

13.1 On the basis of an interlaboratory test of this test method in which three laboratories tested four types of coatings differing in their abrasion resistance, the within-laboratory coefficient of variation was found to be 9 % with 22 degrees of freedom and the between-laboratories coefficient of variation 35 % with 7 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

13.1.1 *Repeatability*—Two results, each the mean of three runs, obtained by the same operator should be considered suspect if they differ by more than 25 % of their mean value.

13.1.2 *Reproducibility*—Two results, each the mean of three runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 118 % of their mean value.

NOTE 7—The reproducibility of this test is improved substantially when rankings of the coatings by magnitude of abrasion resistance are used. In the interlaboratory test for evaluating precision, all laboratories ranked the coatings in the same order.

METHOD B—FALLING SILICON CARBIDE TEST

14. Apparatus and Materials

14.1 *Abrasion Tester*, as described in 6.1, with two exceptions:

14.1.1 A metal washer with an opening of 8.5 ± 0.1 mm is centered in the bottom opening of the funnel to restrict the flow of the abrasive.

14.1.2 The disk gate installed in a slit at the top of the guide tube may be replaced by a gate in the bottom of the funnel. This gate consists of a solid metal disk attached to a long vertical screw and mounted above the washer.

14.2 *Standard Abrasive*⁸—Silicon carbide grain shall be considered standard when graded as follows after a suitable period of continuous sieving:

Less than 1 % retained on a No. 10 (2.00 mm) sieve
Maximum of 20 % retained on a No. 14 (1.40 mm) sieve
0 % pass a No. 20 (0.85 μm) sieve

14.2.1 Use the sieves described in Specification E 11.

15. Test Specimens

15.1 Prepare the coated panels as outlined in 7.1 and 7.2.

16. Standardization

16.1 Standardize the abrasion tester by the procedures

⁷ Supporting data are available from ASTM Headquarters. Request RR-D01-1037.

⁸ An acceptable silicon carbide grain is #16 grade Carborundum, obtainable from the Carborundum Co., P.O. Box 423, Electro Minerals Div., Niagara Falls, NY 14302. A suitable equivalent may be used.

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given in 8.1 and 8.2, with the following exceptions:

- 16.1.1 Use silicon carbide where sand is specified.
- 16.1.2 Weigh the volume of silicon carbide to be introduced into the tester. Determine the efflux time for this volume. The rate of flow shall be 10 ± 1 g/s.

17. Conditioning

17.1 Unless otherwise agreed upon between purchaser and seller, condition the coated panels for at least 24 h at $23 \pm 2^\circ\text{C}$ and 50 ± 5 % relative humidity. Conduct the test in the same environment or immediately on removal therefrom.

18. Procedure

18.1 Measure the thickness of the coating by the procedures given in 10.1.

18.2 Abrade the coated panel by the procedures given in 10.2 and 10.3 using silicon carbide as the abrasive. Determine the volume or weight, or both, of abrasive used to reach the end point.

18.3 Repeat 18.1 and 18.2 on at least one additional coated panel of the material under test.

19. Calculation

19.1 For each area of the coated panel tested, calculate the abrasion resistance in litres per mil from the equation given in 10.1 or, calculate the abrasion resistance, A , in kilograms per mil from the equation:

$$A = W/T$$

where:

W = weight of abrasive used, kg (to one decimal place) and T = thickness of coating, mils (to one decimal place).

19.2 Calculate the mean of the abrasion resistance values

obtained at different locations on the test specimen and the mean values of the replicate panels.

20. Report

20.1 Report the information specified in Section 12. Abrasion resistance may be reported as litres per mil or as kilograms per mil.

21. Precision⁷

21.1 On the basis of an interlaboratory test of this test method in which operators in three laboratories tested four coatings having a broad range of abrasion resistance, the within-laboratory coefficient of variation was found to be 19 % with 16 degrees of freedom and the between-laboratories coefficient of variation 45 % with 8 degrees of freedom. Based upon these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

21.1.1 *Repeatability*—Two results, each the mean of three runs, obtained by the same operator should be considered suspect if they differ by more than 56 % of their mean value.

21.1.2 *Reproducibility*—Two results, each the mean of three runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 147 % of their mean value.

NOTE 8—The reproducibility of this test is improved substantially when rankings of the coatings by magnitude of abrasion resistance are used. In the interlaboratory test for evaluating precision, all laboratories ranked the coatings in the same order.

22. Keywords

22.1 abrasion (of paints/related coatings); falling abrasive tester; falling sand abrasion test; falling silicon carbide abrasion test; resistance, abrasion

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Standard Test Method for Laboratory Determination of Degree of Bleeding of Traffic Paint¹

This standard is issued under the fixed designation D 969; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Editorial changes were made throughout, including the title, in October 1989.

1. Scope

1.1 This test method covers a laboratory test procedure for determining the degree of bleeding of traffic or pavement marking paints.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 476 Specification for Titanium Dioxide Pigments²

D 867 Specification for Pumice Pigment²

D 868 Test Method for Evaluating Degree of Bleeding of Traffic Paint³

D 1199 Specification for Calcium Carbonate Pigments²

2.2 Adjunct:

D 868 Bleeding resistance of paint (one photo)⁴

3. Significance and Use

3.1 Solvents in a traffic paint may cause bleeding of pavement constituents into the traffic marking, thereby rendering the traffic marking less effective as a lane or directional indicator. This test method describes how to prepare a panel for evaluation. The very subjective method of evaluating the degree of bleeding raises questions as to the usefulness of the result for specification compliance.

4. Apparatus

4.1 Film Applicator—A conventional type drawdown

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved Nov. 29, 1985. Published January 1986. Originally published as D 969 - 48 T. Last previous edition D 969 - 54 (1981)^{ε1}.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Copies of the pictorial photographic reference standards are contained in the publication *Pictorial Standards of Coating Defects*, and may be obtained from ASTM Headquarters (request Adjunct PCN 12-408680-00) or the Federation of Societies for Coating Technology, 492 Norristown Rd., Blue Bell, PA 19422. The original source of the photographic reference standards illustrated in Fig. 1 is the Federation.

gage⁵ that casts a film having a minimum width of 2 in. (50 mm) and an approximate wet thickness of 15 mils (380 μ m) (applicator clearance of approximately 30 mils (760 μ m)).

4.2 *Test Panel for Coal-Tar Substrate*—A 5 by 10-in. (127 by 254-mm) piece cut from a standard roll of so-called "15-lb (6.8-kg) coal-tar saturated asbestos or rag felt" (Note).

4.3 *Test Panel for Asphalt Substrate*—A 5 by 10-in. (127 by 254-mm) piece cut from a standard roll of 15-lb (6.8-kg) asphalt-saturated felt.

NOTE—The following paint composition, when used on 15-lb (6.8-kg) felts from various sources, gave the severe bleeding necessary for a rating of between 4 and 2 on the coal tar, and the lesser bleeding necessary for a rating between 9 and 7 on asphalt; when compared to the photographic reference standards of Test Method D 868.⁴ This control paint is suggested only for use in standardizing the test panels.

	Pounds (kg) ^A	Gallons (L) ^B
Titanium dioxide TiO ₂ (Specification D 476, Type II, Class II)	219.3	6.25
Calcium carbonate/CaCO ₃ /ASTM Specification D 1199	525.7	23.29
Aluminum stearate (a 5% mixture in toluol shall gel below 120°F)	2.1	0.23
Pumice, No. 100 (Specification D 867)	87.8	4.48
Alkyd resin solution, pure oxidizing 52 % soya oil modified, 50 % solution in 38 K.B. mineral spirits	425.7	55.98
V.M. & P. naphtha	53.9	8.78
Lead naphthenate, 24 %	5.7	0.59
Cobalt naphthenate, 6 %	1.3	0.16
Chemical ASA (anti-skin agent)	1.8	0.24
Totals	1323.3	100.00

^A 1 lb = 0.454 kg.

^B 1 gal = 3.7854 L.

5. Preparation of Test Panel

5.1 Cut the 5 by 10-in. (125 by 255-mm) specimen panel from that portion of the roll 5 in. in from the edges.

5.2 Provide a nonbleeding contrast surface by affixing a 3/4-in. (20-mm) cellophane tape, with firm pressure, to the entire length of the panel so that the outside edge of the tape is at least 1 in. (25 mm) from the edge of the panel and parallel to the edge of the panel.

6. Procedure

6.1 Place the test panel on a smooth flat surface (such as a

⁵ The "Bradley Blade," also known as the "Bird Film Applicator," available from any coating materials supply house, has been found satisfactory for this purpose.

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glass panel), with the tape side up on the left, and hold in a flat position by weighting down the edges.

6.2 Draw the paint under test down over the specimen panel in such a manner that the entire width of the tape is covered, leaving the remainder of the film to the right of the tape in direct contact with the test panels.

6.3 Keep the coated panel in a flat position until the film is set (after which the weights may be removed) and allow the coated panel to dry for 48 h at 70 to 80°F (21 to 27°C).

6.4 Immediately after completion of 48-h drying, observe the contrast in color between the portion of the film over the tape and that portion that is in direct contact with the test panels. Rate the degree of bleeding numerically in accordance with the nearest photographic reference standard in Test Method D 868.⁴

7. Precision and Bias

7.1 Precision and bias cannot be determined.

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Standard Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers¹

This standard is issued under the fixed designation D 1005; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

⁶¹ NOTE—Figure 1 which was inadvertently deleted was editorially included in May 1990.

1. Scope

1.1 This test method covers the measurement of film thickness of dried films of paint, varnish, lacquer, and related products using micrometers. Procedures A and B utilize stationary micrometers and Procedures C and D, hand-held micrometers. Procedures A and C are not recommended for films less than 0.5 mils (12.5 μm) in thickness. The minimum thickness required for Procedures B and D is a function of that required to enable removal of the sample as a free film.

1.2 The procedures appear as follows:

1.2.1 *Procedure A*—Stationary micrometer for measuring coatings applied to plane rigid surfaces.

1.2.2 *Procedure B*—Stationary micrometer for measuring free films.

1.2.3 *Procedure C*—Hand-held micrometer for measuring coatings applied to plane rigid surfaces.

1.2.4 *Procedure D*—Hand-held micrometer for measuring free films.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

D823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D2370 Test Method for Tensile Properties of Organic Coatings²

3. Significance and Use

3.1 This test method is particularly applicable to the measurement of free films and is also satisfactory for the

measurement of films on laboratory test panels.

3.2 The accuracy and precision of the thickness measurements may be influenced by the deformability of the coating. This test method is not applicable to coatings that are readily deformable under the load of the measuring instrument.

3.3 The accuracy and precision of the thickness measurements are also influenced by the uniformity of the substrate when the coatings are applied to laboratory test panels.

4. Apparatus

4.1 Procedures A and B:

4.1.1 The apparatus shall consist of a dial comparator, dial indicator, or micrometer. A rigid base is required for mounting the dial comparator or dial indicator gages. The presser foot of the micrometer or dial indicator shall be circular, from $\frac{1}{16}$ to $\frac{1}{8}$ in. (1.5 to 3.0 mm) in diameter, and shall be flat on the bottom. The presser foot shall be fixed to an indicator that reads to 0.1 mil (2.5 μm). The load on the presser foot shall be between 20 and 40 psi (140 and 275 kPa). For Procedure B, a smooth uncoated test plate is also required.

4.1.2 Verify the accuracy of instrument calibration by setting to zero with the anvils closed followed by measuring shims of known thicknesses or standards specifically designed for this purpose. Record the standard thickness gage measurement and the micrometer reading. Use these results to construct a calibration curve.

4.2 *Procedures C and D*—The apparatus shall consist of a hand-held micrometer. The anvils of the micrometer shall be circular, from $\frac{1}{16}$ to $\frac{1}{8}$ in. (1.5 to 3.0 mm) in diameter, with flat bottoms. Verify the accuracy of instrument calibration by setting to zero with the anvils closed followed by measuring shims of known thicknesses or standards specifically designed for this purpose. Record the standard thickness gage measurement and the micrometer reading. Use these results to construct a calibration curve.

5. Test Specimens

5.1 *Procedures A and C*—Apply test films to a suitable plane, rigid base material from which the dried film may be satisfactorily removed. The panels shall be of sufficient size to permit film thickness measurements to be made 1 in. (25 mm) from any edge.

5.1.1 Coatings should be applied in accordance with Test

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Aug. 31, 1984. Published December 1984. Originally published 1949. Last previous edition D 1005 - 51(1979).

² Annual Book of ASTM Standards, Vol 06.01.

Methods D 823 or as agreed upon between the purchaser and the seller.

5.2 *Procedures B and D*—Free films of the test material are required. Alternatively, the test materials can be applied to an appropriate substrate in order that they can be removed as free films without deformation. If the method of specimen preparation affects the film forming properties of the test material or requires cutting or scraping to remove the free film, use Procedures A or C instead.

5.2.1 Free films may be prepared in accordance with Test Method D 2370.

6. Procedure

6.1 Procedure A:

6.1.1 Mount the test panel rigidly on a suitable base. Clamp or hold it to the base in such a way that there will be no movement or spring of the panel during the film thickness measurement.

6.1.2 Close the gage slowly until contact is made, but without visible distortion of the film. Read the gage, estimating to 0.1 mil (2.5 μm), and record the reading.

6.1.3 Open the gage and remove the film carefully from the area where the measurement was taken. Any suitable means, chemical or mechanical, may be used to remove the film, taking care not to distort the panel. Close the gage, slowly on the area from which the film was removed, and take a reading, estimating to 0.1 mil (2.5 μm).

6.1.4 The difference in the gage readings before and after the removal of the film is the thickness of the film. Record to 0.1 mil (2.5 μm).

6.1.5 As an alternative to the above, the panels may be measured in distinct locations prior to the application of the coating, and again in the identical locations after the coating has cured. The difference represents the coating thickness.

6.1.6 Take a sufficient number of readings to characterize the panel. A recommended minimum is three determinations for a 3 by 6-in. (75 by 150-mm) panel and more in proportion to size.

6.2 Procedure B:

6.2.1 Mount a smooth uncoated test panel rigidly on a suitable base. Clamp or hold it to the base in such a way that there will be no movement or spring of the panel during the film thickness measurement. Figure 1 illustrates one satisfactory way of rigidly mounting the panel for measurement.

6.2.2 Close the gage slowly until contact is made. Read the gage, estimating to 0.1 mil (2.5 μm), and record the reading.

6.2.3 Open the gage and lay a free film of the test material on the panel in the same area where the measurement was taken. Close the gage slowly with care not to distort the film and take a reading, estimating to 0.1 mil (2.5 μm).

6.2.4 The difference in the gage readings is the thickness of the film. Record to 0.1 mil (2.5 μm).

6.2.5 When conditions permit, perform a minimum of three determinations adjacent to one another on each film.

6.3 Procedure C:

6.3.1 Hold the hand-held micrometer in such a manner that the micrometer can be steadied against a film surface. Separate the micrometer anvils to a distance at least twice that of the film thickness to be measured.

6.3.2 Place the coated base material between anvil con-

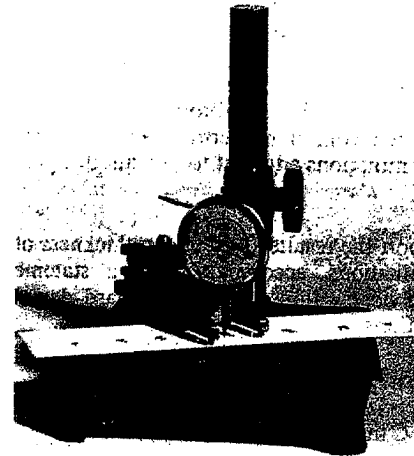


FIG. 1

tacts. Be sure to align the panel so that it is perpendicular to the contact points. Carefully bring the micrometer anvil into contact with the film by releasing the spring tension or rotating the adjustment barrel. Do not compress the film.

6.3.3 Record the film thickness to 0.1 mil (2.5 μm). For spring-loaded micrometers, record the value directly from the dial indicator. For barrel micrometers, record the value using the instrument in the friction mode. The friction mode allows the thimble sleeve to slip without further turning the measuring screw. The friction mode provides for a consistent measuring pressure from panel to panel.

6.3.4 Open the gage and remove the film carefully from the area where the measurement was taken. Any suitable means, chemical or mechanical, may be used to remove the film, taking care not to distort the panel. Close the gage, slowly on the area from which the film was removed, and take a reading, estimating to 0.1 mil (2.5 μm).

6.3.5 The difference in the gage readings before and after the removal of the film is the thickness of the film. Record to 0.1 mil (2.5 μm).

6.3.6 As an alternative to the procedures in 6.3.2 through 6.3.5, the panels may be measured in distinct locations prior to the application of the coating, and again in the identical locations after the coating has cured. The difference represents the coating thickness.

6.3.7 Take a sufficient number of readings to characterize the panel. A recommended minimum is three determinations for a 3 by 6-in. (75 by 150-mm) panel and more in proportion to size.

6.4 Procedure D:

6.4.1 Hold the hand-held micrometer in such a manner that the micrometer can be steadied against a film surface. Separate the micrometer anvils to a distance at least twice that of the film thickness to be measured.

6.4.2 Place the free film to be measured between anvil contacts. Be sure to align the film so that it is perpendicular to the contact points. Carefully bring the micrometer anvil into contact with the film by releasing the spring tension or rotating the adjustment barrel. Do not compress the film.

6.4.3 Record the film thickness to 0.1 mil (2.5 μm). For spring-loaded micrometers, record the value directly from

the dial indicator. For barrel micrometers, record the value using the instrument in the friction mode. The friction mode allows the thimble sleeve to slip without further turning the measuring screw. The friction mode provides for a consistent measuring pressure from film to film.

6.4.4 When conditions permit, perform a minimum of three determinations adjacent to one another on each film.

7. Report

7.1 Report the results as the mean thickness of a number of determinations, accompanied by a statement of the number of observations and the standard deviation of the determinations.

8. Precision and Bias

8.1 *Precision*—On the basis of an interlaboratory study of Procedure C in which operators in two laboratories on two or three days made four replicate measurements on each of three coated panels differing in film thickness, the within-laboratory standard deviations were found to be 0.17 mil at the 1-mil thickness level, 0.32 mils at the 4-mil thickness level, and 0.28 mil at the 8-mil thickness level. On the basis of the same interlaboratory study of Procedure C in which operators in seven laboratories made four replicate measure-

ments on each of three coated panels differing in film thickness, the between-laboratories standard deviations were found to be 0.09 mil at the 1-mil thickness level, 0.29 mil at the 4-mil thickness level, and 0.33 mil at the 8-mil thickness level. Based on these standard deviations, the following criteria should be used to judge the precision of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two measurements, each the mean of four replicates, obtained by the same operator should be considered suspect if they differ by more than 0.7 mil at the 1-mil thickness level and by more than 1.2 mils at the 4 to 8-mil thickness level.

8.1.2 *Reproducibility*—Two measurements, each the mean of four replicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.3 mils at the 1-mil thickness level and by more than 1.1 mils at the 4 to 8-mil thickness level,

8.2 *Bias*—Bias has not been determined.

9. Keywords

9.1 dial comparator; dial indicator; dial indicator gage; film—dry film thickness; measurement of organic coatings; micrometer

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Practice for Conducting Exterior Exposure Tests of Paints on Wood¹

This standard is issued under the fixed designation D 1006; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This practice has been approved for use by agencies of the Department of Defense to replace Method 6161.1 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

^{e1} NOTE—Editorial changes were made throughout in April 1986.

1. Scope

1.1 This practice deals only with the testing of house paints and trim paints on new, previously unpainted wood.

1.2 This practice describes a test procedure that embodies the principles considered necessary for reliable results. Variations necessitated by circumstances may be introduced by agreement provided they do not violate these principles. One procedure embodying the principles is described in the Annex for use by those who find it convenient.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 358 Specification for Wood to Be Used As Panels in Weathering Tests of Coatings²

D 1150 Single and Multi-Panel Forms for Recording Results of Exposure Tests of Paints²

2.2 U.S. Federal Standard:

TT-W-571b Federal Specification for Wood-Preservative, Recommended Treating Practice³

3. Significance and Use

3.1 The procedure described in this practice is intended to aid in evaluating the performance of house and trim paints applied to new, previously unpainted wood.

3.2 Since natural environment varies with respect to season, geography, and topography, test results can vary in accordance with location and may not correlate to actual in-service performance (5.1).

4. Extent of Test Program

4.1 The extent of the exterior exposure test program must be governed by the breadth of the conclusions desired. The

types of paints to be tested, the range in climatic conditions to be met, also the types of woods and structures on which the paints are to be used are important factors to be considered in establishing the exposure program.

5. Location of Test Stations

5.1 The climatic conditions of the test sites should be representative of those of the area in which the paints are to be used. The type and rate of failure of a paint film will vary when exposed to different combinations of climatic and atmospheric conditions. For reliable results, exposure sites should be selected that are representative geographically, climatically, and in atmospheric contaminations with those of the locality in which the paint will be used. To obtain conclusions that are valid for paints with national distribution requires exposure at several sites, selected to cover a wide range in climatic conditions.⁴

6. Exposure Positions

6.1 Panels for testing house paints and trim paints should be exposed on vertical test fences facing both south and north. In comparisons where dirt collection and mildew resistance are not pertinent, north vertical exposures may be omitted. There should be no obstructions close enough to shade test panels from the sun more than 2 h after sunrise, or 2 h before sunset.

6.2 In the case where it is desirable to expose coated panels in a sheltered area, such as under eaves, a suitable test fence with a sheltered or eave arrangement can be used (see Annex).

7. Construction of Test Fences

7.1 Test fences should be durable and rigid enough to remain upright under the action of prevailing winds and frost throughout the contemplated period of testing.⁵

7.2 Lower edges of test panels, when mounted on test fences, should be at least 18 in. (460 mm) above ground level to avoid dampness and mud splash. Backs of painted boards or plywood should be protected against direct exposure to the weather by methods such as, (1) having panels on both

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Oct. 29, 1973. Published December 1973. Originally published as D 1006 - 51 T. Last previous edition D 1006 - 56 (1970).

² Annual Book of ASTM Standards, Vol 06.01.

³ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁴ Suggested sites include the Great Lakes region, Florida, extreme southern Louisiana, the southwest region, and northeast region.

⁵ Fences, such as presented by W. A. Southard in the May 1959 issue of the *Official Digest*, are acceptable.

sides of the fence, (2) mounting the panels on sheathing, and (3) closing the opposite side of the fence.

7.3 Fences should have watertight caps to keep water from getting behind test panels.

8. Selection of Woods for Test Panels

8.1 Paint need be tested only on woods on which it is likely to be used in practice. Conclusions drawn from tests made on a limited variety of woods, however, should not be generalized for woods of other kinds.

NOTE—See Specification D 358.

8.2 Prior to use, test lumber and panels should be stored under such conditions that the moisture content of the wood will be maintained within the normal range for exterior woodwork in the region in which the tests are made.

9. Construction of Test Panels

9.1 For house paints, unless the pattern of the siding requires some other choice, test panels should be made of one or the other of two patterns of siding, namely, $\frac{1}{2}$ or $\frac{3}{4}$ by 6-in. (13 or 20 by 150-mm) bevel siding or 1 by 6-in. (25 by 150-mm) drop siding.

9.2 If the panels in the house paint test are not subdivided, one 3-ft (900-mm) length of 6-in. (150-mm) siding will be acceptable. If the panels are subdivided, two 18-in. (460-mm) lengths are sufficient. Exposures on wood panels should preferably be carried out on three panels to allow for variations in the wood.

9.3 For trim paints, the test panel should carry 1 by 4-in. (25 by 100-mm) pieces of trim lumber at each end.

9.4 A test panel of 1.5 ft² (1400 cm²) or more in area, as provided in 9.2, may be subdivided into two or more test areas each not less than 10 in. (250 mm) long and 0.75 ft² (700 cm²) in area. Each test area is for painting with a different paint. Paints placed on the test areas of one panel furnish a comparison as to behavior.

9.5 When it will not interfere with the properties to be tested, all panels should be coated on the back to prevent warping.

10. Control or Comparison Paint for Extending the Comparisons

10.1 When several paints are to be compared, one paint should be selected as a standard of comparison or "control." The control paint should then be applied on one test area of each test panel. Variations caused by wood differences are revealed in the behavior of the control paint, and can be used to adjust the ratings of the other paints to a common basis.

For best results there should be two controls—one known to perform well and one known to perform poorly.

11. Application of Paints

11.1 All tests that are to be compared closely with one another should be placed on exposure as nearly simultaneously as possible. When a group of tests is too extensive for completion within a month, use a control paint or duplicate of at least 5 % of the test areas at successive exposure periods.

11.2 It is best in theory and practice to do the painting out-of-doors in proper weather for painting; however, indoor painting is permissible provided no more than 1 week⁶ elapses between successive coats and between applying the last coat and exposing on the test fence; and provided, further, that all painting is done under essentially the same drying conditions. It is necessary to allow each coat to cure sufficiently before top coating.

11.3 Preferred procedure is to apply paints with the test panel in a vertical position and kept vertical until the paint has set. If paint is spread on horizontal panels, the panels should be placed vertically immediately thereafter.

11.4 Records should be kept of the spreading rates at which paints are applied. When the purpose of the tests is to compare commercial paints, it may be appropriate to let the painter apply them at what seems to be their natural spreading rates. When the purpose is to study variation in paint composition, application should usually be at suitable predetermined spreading rates which can be controlled by applying a given weight or volume of coating to a measured area.

12. Inspections and Records

12.1 After panels have been exposed to the weather, inspections should be made after not more than 1 month, at 3 months, and at intervals of 3 months during the first 2 years, and every 6 months thereafter. Midwinter inspections, however, may be omitted in northern latitudes.⁷ Inspections may be made more frequently if desired. Usually the exposures should be continued for a considerable length of time after deterioration has reached the point at which best practice calls for repainting.

12.2 Records should be kept on forms such as Standard D 1150.⁸

⁶ Seventy-two hours is the preferred maximum.

⁷ Inspection should be made to assure that exposed panels are not covered by accumulated snow banks.

⁸ These record sheets may be obtained from ASTM Headquarters (order, Adjunct No. 12-411500-11 and 12-411500-21) and from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

ANNEX

(Mandatory Information)

A1. CONSTRUCTION OF TEST FENCE AND TEST PANELS

A1.1 The plan for test fence and panels described in this Annex conforms to the principles set forth in this practice. It represents only one of numerous possible embodiments of the principles recommended.

A1.2 Construction of Test Fence:

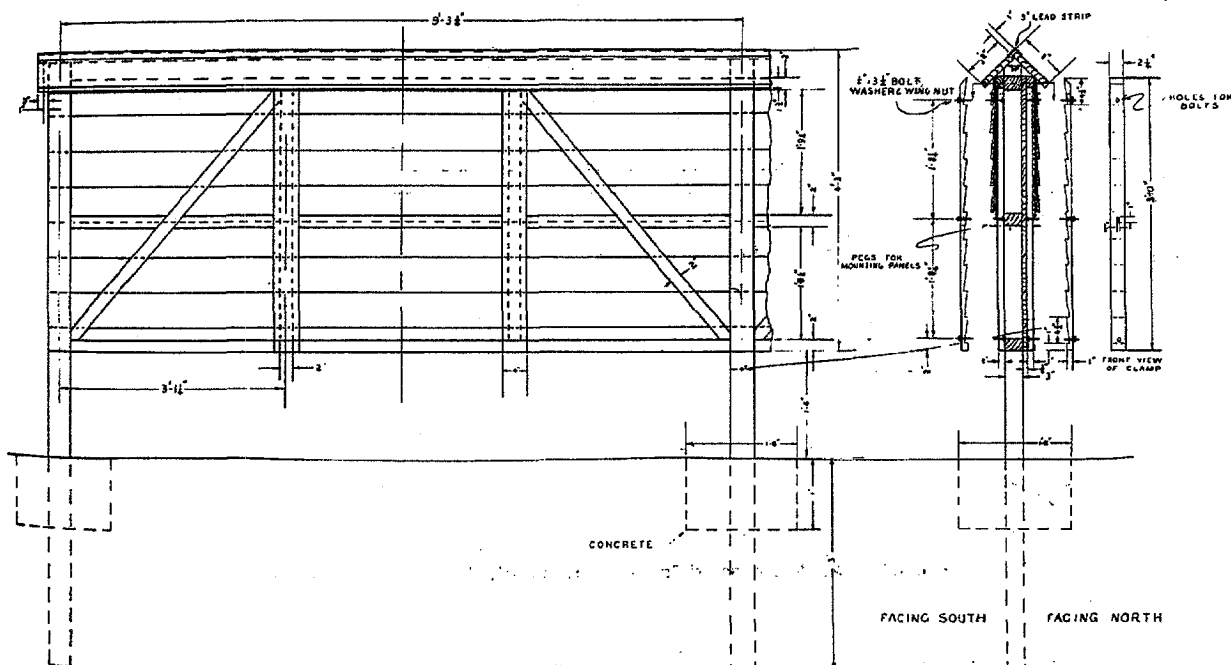
A1.2.1 The test fence, or test rack, runs east and west, and is constructed to hold test panels on both sides so that there are panels facing both north and south. There are two rows of panels, one above the other, on each side of the fence. A 90° cap is placed along the top of the fence, and projects approximately 1 in. (25 mm) beyond the face of the mounted panels. The fence must be sufficiently sturdy in construction to withstand strong winds. It is mounted on wood posts that are impregnated with creosote under pressure, in accordance with Fed. Spec. TT-W-571b.

A1.2.2 Figure A1.1 shows one span or unit. The fence can be extended to as many units as the site permits or as are needed for the number of exposure tests to be made. Additional fences may be built parallel to one another, but they should be spaced far enough apart to keep each fence from casting shadows on the adjacent fences during all but the first 2 h after sunrise and the last 2 h before sunset at the time of the winter solstice.

A1.2.3 When cleated panels of drop siding, which do not have backing, are used, there shall always be a pair of panels, one facing north and one facing south, to give the backs mutual protection from the weather. If for any reason there are panels on one side only of the fence, the other side shall be covered with roofing paper or other covering to protect the backs of the panels.

A1.3 Construction of Test Panels:

A1.3.1 House Paints—The boards of siding are assembled in a manner similar to house construction. Five pieces of ½ by 6-in. (13 by 150-mm) bevel siding are nailed securely on a backing of ¼-in. (6-mm) plywood exterior grade, as shown in Fig. A1.2. The top board is a blank connecting board, cut to narrower width and painted as hereinafter described. The other four boards are test boards: they may be all of one species or two each of two different species. The overlap between boards should be not less than 1 in. (25 mm). Cadmium- or zinc-coated nails, 1½ in. (28 mm) long, should be used and should be spaced as indicated, and clinched on the back. The lower edge of the bottom board is shimmed out from the plywood with a wood shim and the bottom board projects ¼ in. beyond the shim in order to permit insertion of a panel underneath. The top of the panel, which



NOTE—1 in. = 25.4 mm; 1 ft = 0.3048 m.

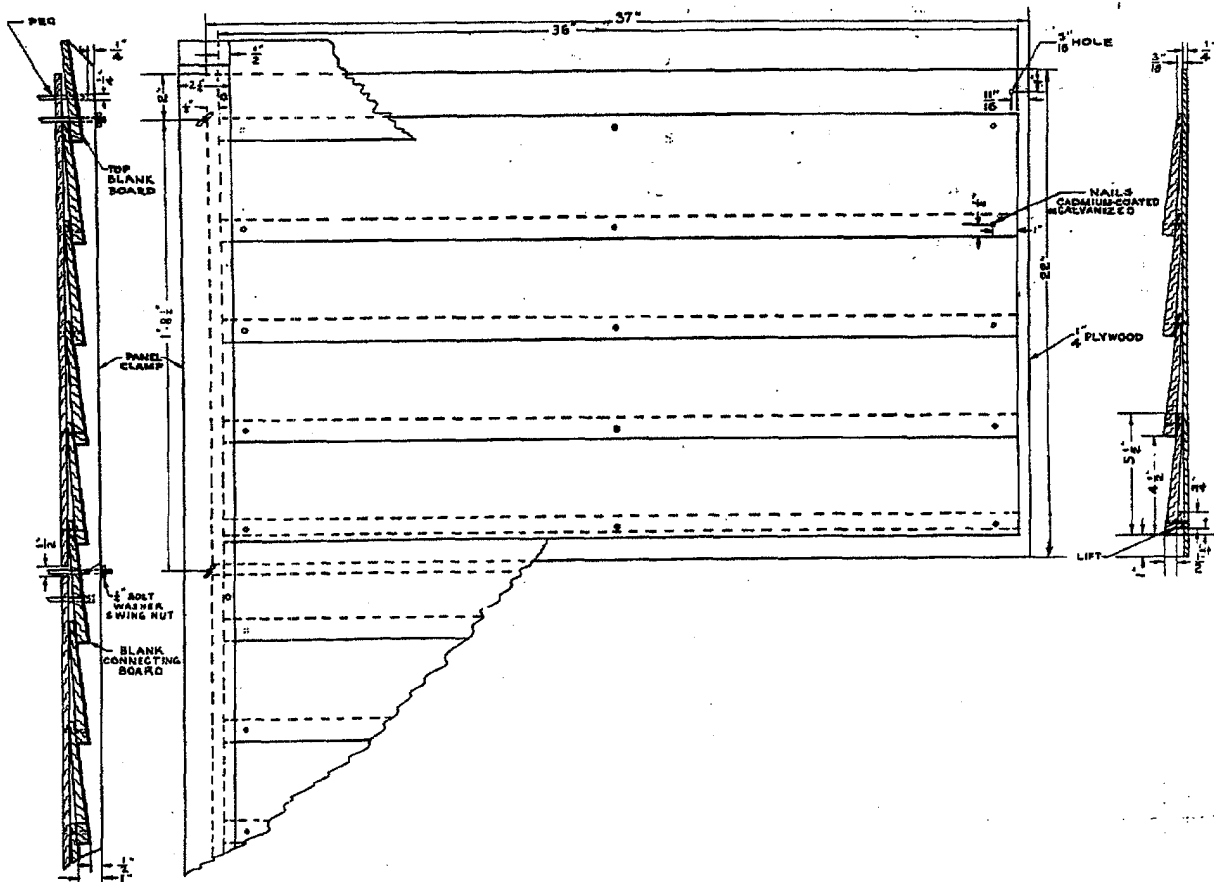
FIG. A1.1 ASTM Panel Rack for Exterior Exposure of Paints

is a blank board precoated with chalk-resisting exterior paint such as aluminum paint, is cut to a width of $4\frac{1}{2}$ in. (115 mm); the cutting makes a suitable shim for use under the bottom board. The plywood projects $\frac{1}{2}$ in. beyond the boards at the ends and 1 in. (25 mm) at the bottom; at the top the plywood projects 2 in. (50 mm) beyond the top of the second board and is overlapped by about $2\frac{1}{2}$ in. (65 mm) by the top blank board. Holes are bored through the top blank board as indicated to permit positioning of the panel on the fence by means of small pegs. Holes are bored through the projecting ends of the plywood to permit secure fastening of the panels to the fence by means of wood clamps held in position by bolts with wing nuts as indicated (see also Fig. A1.1).

A1.3.1.1 Panels made of 6-in (150-mm) drop siding should consist of four test boards 37 in. (940 mm) long, and a narrow blank connecting board 3 in. (75 mm) wide, fitted snugly together and held by three cleats, $\frac{3}{8}$ by 2 in. (16 by 50 mm) wide, nailed firmly on the backs. By adjusting the width

of the blank connecting board the dropsiding panels can be made to fit on fences designed as shown in Fig. A1.1, although it may be impracticable to mount both types of panel together on the same fence.

A1.3.2 *Trim Paints*—An example of a panel for testing trim paint is shown in Fig. A1.3. It is a modification of the panel shown in Fig. A1.2. The modification consists in shortening the pieces of siding to 27 in. (685 mm) to make room for two pieces of trim lumber, 1 by 4 in. (25 to 100 mm), at each end; and in narrowing the exposed width of the siding to 4 in. to make room for another piece of trim lumber, 1 by 6 in. (25 by 150 mm), across the top. This piece is undercut, as shown in the sketch, so as to fit over the siding. The cap may be made of galvanized iron, aluminum, or painted iron. No blank connecting boards between two panels are needed. The panel may be fastened to the fence in the same way as the body paint panel. A much less elaborate panel, satisfactory in many cases, is merely a plain board, approximately 1 by 6 by 36 in. (25 by 150 by 915 mm).

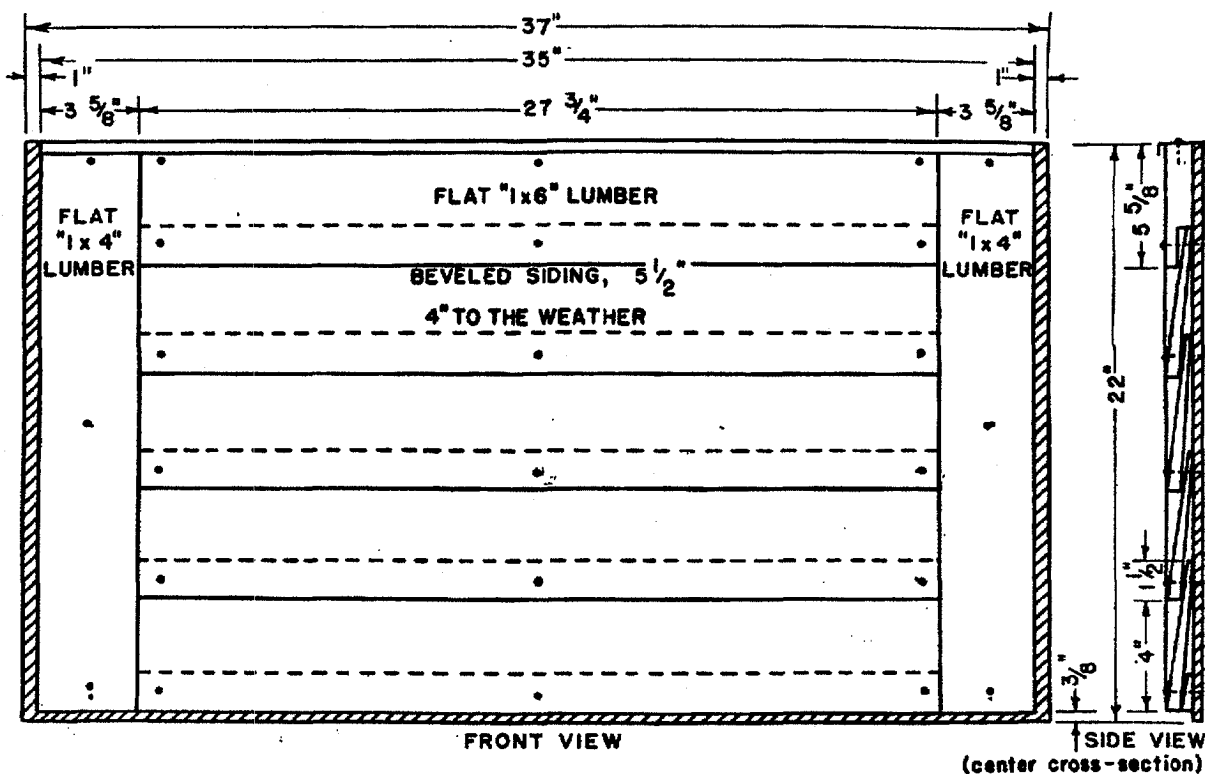


NOTE—1 in. = 25.4 mm.

FIG. A1.2 ASTM Clapboard Test Panel for Exterior Exposure of Paints

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NOTE—1 in. = 25.4 mm.

FIG. A1.3 ASTM Test Panel for Exterior Exposure of Trim Paints

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Conducting Exterior Exposure Tests of Paints on Steel¹

This standard is issued under the fixed designation D 1014; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 6160 of Fed. Test Method Std. No. 141 A and for listing in the DoD Index of Specifications and Standards.

¹ NOTE—Editorial changes were made throughout in March 1988.

1. Scope

1.1 This test method covers the determination of the relative service of exterior paints and other materials of similar purpose when applied on steel surfaces exposed out-of-doors.

1.2 Experience indicates that the steel used as a test surface has a marked bearing upon the weathering results. It is the purpose of this test method to minimize the influence of variation in steel surfaces on any series of tests by providing for uniformity in the selection of the steel surface, particularly in cooperative work. This test method also outlines uniform procedures for conducting the exposure tests and for evaluating and recording results.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- A 36 Specification for Structural Steel²
- A 283 Specification for Low and Intermediate Tensile Strength Carbon Steel Plates²
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products³
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces³
- D 659 Methods of Evaluating Degree of Chalking of Exterior Paints³
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints³
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints³
- D 714 Test Method for Evaluating Degree of Blistering of Paints³

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Painting.

Current edition approved Nov. 28, 1983. Published January 1984. Originally published as D 1014 - 49 T. Last previous edition D 1014 - 66 (1973).

² Annual Book of ASTM Standards, Vol 01.04.

³ Annual Book of ASTM Standards, Vol 06.01.

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base³

D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings³

D 2200 Pictorial Surface Preparation Standards for Painting Steel Surfaces³

G 7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials⁴

3. Significance and Use

3.1 The procedures described in this test method are intended to aid in evaluating performance of coatings on either new or previously rusted steel.

3.2 Since the natural environment varies with respect to season and geographic location, test results may not correlate with in-service performance.

4. Base Materials for Test Specimens

4.1 A minimum of two and preferably four test specimens shall be used to evaluate the performance of any paint system.

4.2 The surface preparation for the test panels should be that expected to be done in the field or in-service. The surface preparation shall be the same for all test panels in the test program unless surface preparation is one of the variables to be evaluated. Surface preparation must be essentially identical for all test panels, as the thoroughness of preparation may directly determine the performance life of the applied coating system.

4.3 The test panels should be fabricated from the same material over which the coating is expected to perform in-service. Any of the following surfaces may be used:

4.3.1 *Abrasive Blasted Steel Plate*—The steel plate shall conform to Specification A 36 or Specification A 283. The minimum thickness shall be $\frac{1}{16}$ in. (1.6 mm). The minimum size shall be 4 by 6 in. (100 by 150 mm). Burrs and sharp projections shall be removed from the edges by filing. The test panels shall be freed of oil by suitable grease-removing solvents in accordance with Methods B, C, or D of Methods D 609. The surface shall be blasted to meet the requirements

⁴ Annual Book of ASTM Standards, Vol 14.02.

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of Standard D 2200, $Sa\ 2\frac{1}{2}$, if another degree of surface preparation has not been agreed upon.

4.3.2 Rusted Surfaces—Hot rolled steel angle or plate, or both, are useful for determining the performance of paints applied to structures that cannot be thoroughly cleaned of rust and corrosion products. The steel angle and plate shall conform to Specification A 283. The steel angles shall be at least 4 by 4 by $\frac{1}{8}$ in. (100 by 100 by 3.2 mm) in cross section and 12 in. (305 mm) in length. The minimum size of the steel plate shall be 4 by 6 in. (100 by 150 mm) with a minimum thickness of $\frac{1}{16}$ in. (1.6 mm). Burrs and sharp projections shall be removed from the edges by filing. The test pieces shall be freed from oil by the use of suitable grease-removing solvents in accordance with Methods B, C, or D of Methods D 609. Those persons desiring to test coatings over rusty or slightly rusted surfaces (Note 1) should refer to Standard D 2200, select the degree of rusting desired from the rust grades given, and utilize the degree of surface preparation that can be accomplished in the field or on the job.

NOTE 1—When testing over rusty surfaces, the test specimens should preferably be pre-corroded (weathered) in the same environment in which they ultimately will be exposed. The environment in which the steel is rusted prior to painting has considerable influence on the performance of paint applied to such steel. Artificial rusting, however, is permitted but conditions must be stated in the test report.

4.3.3 Cold-Rolled Steel Strip—Cold-rolled steel strip has a slightly roughened surface free from mill scale and rust, and is useful for checking the relative performance of paints on a clean, uniform surface. The steel strip shall conform to one of the types described in Methods D 609. The panels shall be not less than 4 by 6 in. (102 by 152 mm) in size and all edges shall be smooth and uniformly rounded. The metal panels shall be prepared by the agreed upon method (A, B, C, or D) in Methods D 609.

4.4 After surface preparation, the panels shall be prime coated as soon as possible to prevent flash rusting or deposit of any foreign contaminant on the cleaned surface. After surface preparation, if the panels are stored prior to coating, precautions must be taken to preserve the clean surface.

5. Painting Test Specimens

5.1 Apply all coatings in strict accordance with the coating manufacturer's written recommendations. The method of application expected for the production work on the job should also be used for test panel application.

5.2 If the method of application is unknown select one of the following (Note 2):

- Method A—Automatic Spray Machine
- Method B—Automatic Dip Coater
- Method C—Manual Spray Application
- Method D—Motor Driven Blade Applicator
- Method E—Brush Application
- Method F—Roller Coating
- Method G—Curtain Coating

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NOTE 2—Details for the application of paint by Methods A, B, or D are given in Test Methods D 823.

5.3 Measure and record the film thickness of each coat in accordance with the methods recommended in Test Methods D 1186. If the test panel is covered by rust and mill scale, these methods will be less accurate, as they are influenced by the surface characteristics of this base metal. In such cases, approximations can be made by wet film thickness measurements in accordance with Methods D 1212, or the amount of paint applied to a known area can be weighed and the average dry film thickness computed. It should be noted that even though dry film thickness measurements in accordance with Test Methods D 1186 are less accurate on surfaces with rust and mill scale than on smooth steel surfaces, they are still more accurate than those obtained by Methods D 1212 or weighing.

5.4 Allow the proper drying time between coats for multiple paint systems and before exposure as required by the coatings manufacturer and include in the test record.

5.5 Paint the back and edges of all test specimens with the same systems as that being tested on the front of each panel. This painting provides considerable information on the behavior of the paint system on the reverse side.

5.6 The test specimens may be scribed to base metal prior to exposure. Rate any corrosion from this point of damage in accordance with Methods D 610.

6. Exposure

6.1 The type of exposure and the position of the painted specimens may be selected from the following:

- 5°, facing south
- 45°, facing south
- 45°, facing north
- Vertical, facing south
- Vertical, facing north

Insulated (backed or black box) in accordance with Practice G 7.

6.2 Mount the specimens so they do not cast shadows on each other, or contact each other or any metallic material, or any material capable of acting as a wick. Also, mount the specimens so that the products of weathering and rain water drippings do not flow from one to another.

NOTE 3—A suitable material for the construction of racks and supports is painted wood. Metal, such as aluminum, is also suitable if the test specimens are properly insulated as by the use of porcelain or suitably selected plastic.

7. Test Period and Rating of Paints

7.1 The test period shall be sufficient to evaluate the characteristics of the system under consideration.

7.2 Inspect at regular intervals.

7.3 Evaluate (resistance to or) degree of rusting, chalking, checking, cracking, and blistering using the following ASTM photographic reference standards: Test Methods D 610, D 659, D 660, D 661, and D 714.



Standard Single- and Multi-Panel Forms for Recording Results of Exposure Tests of Paints¹

This standard is issued under the fixed designation D 1150; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout in May 1987.

1. Single-Panel Paint Record Form

1.1 The single-panel form² provides on one sheet the complete exposure record of a paint test panel. It provides a record for a period of 60 months for 15 or more different types of failure. On the front side there are eight graphs upon which the record for practically every type of failure may be plotted. The type of failure is specified on six of the graphs as indicated and there are two additional graphs for special types of failure that may be required. In order to record three types of failure on a single graph the symbols X, O, and (dot) are used. The numerical system of grading is used. Ten indicates perfection or absence of failure while zero represents complete failure. Ratings are plotted along the vertical axis in steps of 2. Intermediate ratings in steps of 1 may be plotted between the lines. Time is plotted along the horizontal axis. To illustrate the use of the single-panel record form, Fig. 1 shows a section of one graph with gloss, chalking, and erosion ratings recorded.

1.2 On the reverse side of the single-panel form are columns for recording the composition, the reduction procedure, and miscellaneous information for a paint system for as many as four coats. Ample space is provided for additional remarks.

2. Referenced Documents

2.1 ASTM Standards:

D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces³

¹ This standard is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Sept. 12, 1955. Published November 1955. Originally published as D 1150 - 51 T. Last previous edition D 1150 - 55 (1982)^{e1}.

² Copies of the single-panel form and the multi-panel inspection sheet are available from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422, and from ASTM, 1916 Race St., Philadelphia, PA 19103. Request Adjunct No. 12-411500-11 (single-panel) or 12-411500-21 (multi-panel).

³ *Annual Book of ASTM Standards*, Vol 06.01.

D 659 Method of Evaluating Degree of Chalking of Exterior Paints³

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints³

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints³

D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints³

D 714 Test Method for Evaluating Degree of Blistering of Paints³

D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints³

2.2 Other Documents:

*Exposure Standards Manual*⁴

3. Multi-Panel Paint Inspection Sheet

3.1 Although the single-panel form may be used in the field to record results directly, it is recommended that observation in the field be recorded on the multi-panel paint inspection sheet² and later be transcribed to the single form. Provision is made on the multi-panel form for recording twenty different observations of twenty different panels. The type of failure is to be written in the spaces under the heading "Properties." The back of the sheet may be used for remarks or any additional data that need be recorded.

4. Photographic Reference Standards

4.1 The following ASTM test methods refer to photographic reference standards for use in evaluating exposure tests of paints:

4.2 *Chalking*—Method D 659.

4.3 *Checking*—Test Method D 660.

4.4 *Cracking*—Test Method D 661.

4.5 *Erosion*—Test Method D 662.

4.6 *Flaking*—Test Method D 772.

4.7 *Rusting*—Method D 610.

4.8 *Blistering*—Test Method D 714.¹

4.9 The *Exposure Standards Manual* also contains pictorial standards for evaluating paint failures.

⁴ Available from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

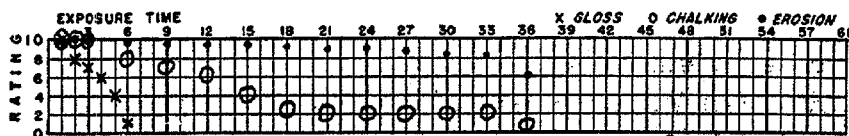


FIG. 1 Sample Section of Single Panel Form Showing Gloss, Chalking, and Erosion Ratings

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Standard Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base¹

This standard is issued under the fixed designation D 1186; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace Method 6181 of Federal Test Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These test methods cover the nondestructive measurement of the dry film thickness of nonmagnetic coatings applied over a ferrous base material using commercially available test instruments. The test methods cover the use of instruments based on magnetic measuring principles only. Test Method A provides for the measurement of films using magnetic pull-off gages and Test Method B provides for the measurement of films using magnetic flux gages.

1.2 These test methods are not applicable to coatings that will be readily deformable under the load of the measuring instrument.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

D823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

2.2 Steel Structures Painting Council Standard:

SSPC-PA2 Measurement of Dry Paint Thickness with Magnetic Gages³

TEST METHOD A—MAGNETIC PULL-OFF GAGES

3. Summary of Test Method

3.1 Instruments complying with this test method measure thickness by using a spring calibrated to determine the force required to pull a permanent magnet from a ferrous base coated with a nonmagnetic film. The instrument must be placed directly on the coating surface to take a reading.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 1186 - 51. Last previous edition D 1186 - 81.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

4. Significance and Use

4.1 The attractive force of the magnet to the substrate varies inversely with the thickness of the applied film. The spring tension required to overcome the attraction of the magnet to the substrate is shown on the instrument scale as the distance (in mils or micrometres) between the magnet and the substrate.

4.2 The accuracy of the measurements may be influenced when made closer than 1 in. (25 mm) to an edge.

5. Apparatus

5.1 *Permanent Magnet*, small, either attached directly to a coil spring ("pencil" gage)⁴ or to a horizontal lever arm which is attached to a helical spring ("banana" gage).⁵ Increasing force is applied to the magnet by extending the coil spring in the first case or turning a graduated dial that coils the helical spring in the second. The readings obtained are shown directly on the instrument scale or converted by reference to a calibration curve.

6. Test Specimens

6.1 When this test method is used in the field, the specimen is the coated structure or article on which the thickness is to be evaluated.

6.2 For laboratory use, apply the materials to be tested to panels of the composition and surface conditions on which it is desired to determine the thickness.

NOTE 1—Applicable test panel description and surface preparation methods are given in Methods D 609.

NOTE 2—Coatings should be applied in accordance with Test Methods D 823 or as agreed upon between the purchaser and the seller.

7. Calibration of Apparatus

7.1 Calibrate the instrument in an area free of stray magnetic fields, such as power lines, generators, or welding equipment. There shall be no vibration apparent on the test piece when the instrument is being calibrated.

7.2 Use a bare section of the substrate after the specified surface preparation method has been accomplished. If an uncoated section of the substrate is not available, uncoated test panels of a similar steel type on which the specified surface preparation has been performed, may be used.

⁴ Apparatus of the "pencil" type found to be suitable for this purpose includes Tinsley Gage, Elcometer Pencil Pull-Off Gage.

⁵ Apparatus of the "banana" type found to be suitable for this purpose includes Magne Gage, Mikrotest, Elcometer Inspector Thickness Gage.

7.3 Use nonmagnetic thickness shims for calibration. Most shims are non-precision and, therefore, thickness must be verified with a micrometer.

NOTE 3—Other thickness standards such as National Bureau of Standards certified thickness calibration standards may be used for the calibration of the magnetic pull-off gages. The use of these thickness standards requires a different calibration method as described in SSPC-PA2.

7.4 Select calibration shims in the expected thickness range to be measured. For example, if a coating is approximately 3 mils (75 μ m) in thickness, calibrate the instrument at 3 mils. Then check the calibration, using shims of both a lesser and greater thickness, to determine the thickness range over which the instrument will register accurately.

7.5 Lay a calibration shim on the bare, uncoated substrate and bring the instrument magnet in direct contact with the shim. Remove the magnet from the shim by slowly rotating the dial scale ring clockwise (for helical spring-type instruments) or lifting the entire instrument housing (for coil spring-type instruments). Hold the shim so that it will not flex during calibration, causing the magnet to lift prematurely. Observe the thickness shown on the instrument scale at the moment when the magnet breaks contact with the surface.

7.6 If the instrument scale reading does not agree with the shim thickness, calibration is required. This can be accomplished by physical calibration or by drafting a calibration curve. A calibration curve involves plotting a graph with the actual gage reading on one axis and the shim reading on the other. For physical calibration, consult the manufacturer's instructions.

NOTE 4—Instruments based on the scale dial ring/helical spring can be calibrated and used in any position, while those based on the coil spring must be calibrated and used in the vertical position only.

8. Procedure

8.1 Use the instrument only after it has been calibrated in accordance with Section 7.

8.2 Assure that the coating is dry prior to use of the instrument.

8.3 Inspect the magnet tip and surface to be measured to assure that they are clean. Adherent magnetic filings or other surface contaminants will affect gage readings.

8.4 Take readings in areas free of vibration, electrical, or magnetic fields.

8.5 If thickness readings are encountered outside the range of accuracy determined during calibration in 7.4, repeat the calibration procedure in that range. Check the calibration frequently during use to assure that the instrument continues to read properly.

8.6 Take a sufficient number of readings to characterize the sample.

8.6.1 For laboratory measurements, a recommended minimum is three for a 3 by 6-in. (75 by 150-mm) panel and more in proportion to size.

8.6.2 For field measurements, a recommended minimum is five determinations at random for every 100 ft² (10 m²) of surface area. Each of the five determinations should be the mean of three separate gage readings within the area of a 1/2-in. (12-mm) diameter circle.

8.7 Make measurements at least 1 in. (25 mm) away from any edge or corner of the specimen. If it is necessary to measure closer than 1 in., recheck the calibration in the specific area to determine the extent of the effect (if any) the edge has on the measurement.

8.8 Report the instrument used, serial number, range, and mean of the thickness readings found.

9. Precision

9.1 An interlaboratory test was conducted to determine the within-laboratory and between-laboratory precision of several types of instruments in measuring a wide range of coating thickness by this method. On the basis of this interlaboratory test, the within-laboratory coefficients of variation and between-laboratory coefficients of variation were found to be those shown in Table 1 for the instruments evaluated. Based upon these coefficients of variation, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results by the same operator with the same instrument should be considered suspect if they differ by more than the maximum allowable difference values shown in Table 1.

9.1.2 *Reproducibility*—Two results, each the mean of four measurements, obtained by operators in different laboratories should be suspect if they differ by more than the maximum allowable difference values shown in Table 1.

TEST METHOD B—MAGNETIC FLUX GAGES

10. Summary of Test Method

10.1 This test method covers instruments that measure coating thickness by changes in the magnetic flux within the instrument probe or the instrument itself. The instrument probe must remain in direct contact with the coating at all times during measurement.

11. Significance and Use

11.1 The magnitude of flux changes is an inverse (nonlinear) function of the distance between the probe and the ferrous substrate.

11.2 The accuracy of the measurements may be influenced when made closer than 1 in. (25 mm) to an edge or 3 in. (75 mm) to another mass of steel. The edge or mass of steel may cause flux leakage from the instrument, thus distorting the readings.

12. Apparatus

12.1 The testing apparatus shall be mechanically or electrically operated. The mechanically operated instruments house an integral horseshoe magnet, the contacts of which are placed directly on the coated substrate.⁶ The electrically operated instruments utilize a separate instrument probe that houses the magnet and must be placed directly on the surface.⁷ In both types, the coating thickness is shown on the instrument scale or meter.

⁶ Apparatus of the mechanical type found to be suitable for this purpose includes Elcometer 101.

⁷ Apparatus of the probe type found to be suitable for this purpose includes the Minitest, Minitecor, Verimeter, GE Gage, Ferascope, and Accuderm.

TABLE 1 Precision of Film Thickness Measurements

Within Laboratory							
Coating Thickness		0.5 to 1.5 mils			5 to 17 mils		
Test Method	Instrument	Degrees of Freedom (DF)	Coefficient of Variation (v_w), %	Maximum Allowable Difference (MAD), %	Degrees of Freedom (DF)	Coefficient of Variation (v_w), %	Maximum Allowable Difference (MAD), %
A	Coil Spring						
	Elcometer 157	12	9		12	5	
	Tinsley	12	8		12	4	
	Pooled	24	7.65	22.4	24	4.53	13.3
A	Helical spring						
	Elcometer 111	12	6		12	3	
	Microtest FIM	28	5		28	2	
	Magne Gage	4	6		8	2	
	Pooled	44	5.4	15.4	48	2.3	6.6
B	Mechanical						
	Elcometer 101	22	7	20.6	22	3	8.8
B	Probe						
	Elcometer 150	12	5		12	1	
	Minitest FN 250	6	5		6	2	
	GE Type B Gage	32	4		32	3	
	Permascope ES	12	4		12	2	
	Verimeter	2	6		3	2	
	Accuderm	4	1		6	2	
	Pooled	68	4.25	12.1	71	2.4	6.8
Between Laboratories							
Coating Thickness		0.5 to 1.5 mils			5 to 17 mils		
Test Method	Instrument	Degrees of Freedom (DF)	Coefficient of Variation (v_b), %	Maximum Allowable Difference (MAD), %	Degrees of Freedom (DF)	Coefficient of Variation (v_b), %	Maximum Allowable Difference (MAD), %
A	Coil Spring						
	Elcometer 157	2	22		2	12	
	Tinsley	2	10		2	7	
	Pooled	4	17.1	67.2	4	9.8	38.6
A	Helical Spring						
	Elcometer 111	2	5	30.5 ^A	2	2	12.2 ^A
	Microtest FIM	8	15	49	8	10	32.6
	Magne Gage	8	8		8	8	
B	Mechanical						
	Elcometer 101	4	11	43.5	4	2	8 ^C
B	Probe						
	Elcometer 150	4	7		4	4	
	Minitest FN 250	...	...		2	3	
	GE Type B	12	6		...	...	
	Permascope ES	6	6		6	3	
	Pooled	22	6.2	18.3 ^A	12	3.4	10.4
	Minitest FN 250	2	22		...	...	
	GE Type B	...	...		12	6	18.5
	Verimeter	8	8		8	8	
	Accuderm	8	8		8	8	

^A Insufficient results for reliable estimate.

^B No interlaboratory results provided.

^C Between-laboratories precision usually larger than within-laboratory precision.

13. Test Specimens

13.1 The test specimens described in Section 6 are suitable for the instruments of Test Method B.

14. Calibration of Apparatus

14.1 Follow the steps outlined in 7.1 through 7.4.

14.2 Hold the instrument contact or probe handle firmly on the surface and perpendicular to the measuring plane during calibration and use. Follow the manufacturer's instructions for the specific adjustment of the instrument.

NOTE 5—The probe-type instruments can be calibrated and used in any position, but the integral-magnet type must be recalibrated or calibration verified for each position of use.

15. Procedure

15.1 Follow the steps outlined in 8.1 through 8.7.

15.2 Take measurements no closer than 1 in. (25 mm) to an edge or 3 in. (75 mm) to another mass of steel. If such measurements are necessary, recheck the calibration in the specific area to determine the effect the edge or mass of steel has on the instrument reading.

15.3 Report the instrument used, serial number, range, and mean of the thickness readings found.

16. Precision

16.1 *Precision*—On the basis of an interlaboratory study of this test method in which several types of instruments

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were used in various laboratories to measure a wide range of dry-film thickness on the same set of panels, the within-laboratory and between-laboratories coefficients of variation were found to be as shown in Table 1. Based upon these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

16.1.1 *Repeatability*—Two results obtained by the same operator using instruments from the same category should be

considered suspect if they differ by more than the maximum allowable difference values given in Table 1 for the appropriate film thickness.

16.1.2 *Reproducibility*—Two results, each the mean of four measurements, obtained by operators in different laboratories using instruments from the same category should be considered suspect if they differ by more than the maximum allowable difference values given in Table 1 for the appropriate film thickness.

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Maximum
Allowable
Difference
(MAD), %

13.3

6.6

8.8

6.8

Maximum
Allowable
Difference
(MAD), %

38.6

12.2^a

32.6

8^c

10.4

18.5

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Standard Test Method for Viscosity by Ford Viscosity Cup¹

This standard is issued under the fixed designation D 1200; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the Department of Defense to replace Method 4282 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of the viscosity of Newtonian or near-Newtonian paints, varnishes, lacquers, and related liquid materials with the Ford-type efflux viscosity cup. If the material is non-Newtonian, that is, shear-thinning or thixotropic, Test Method D 2196 should be used.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer²

E 1 Specification for ASTM Thermometers³

3. Terminology

3.1 Definitions:

3.1.1 *Newtonian liquid*—a liquid in which the viscosity is independent of the shear stress or shear rate. If the ratio of shear stress to shear rate is not constant, the liquid is non-Newtonian.

3.1.2 *Near-Newtonian liquid*—a liquid in which the variation of viscosity with shear rate is small and the effect on viscosity of mechanical disturbances such as stirring is negligible.

4. Summary of Test Method

4.1 The Ford viscosity cup is filled level full with the liquid under test, and the time for the material to flow through one of the standard orifices is measured.

5. Significance and Use

5.1 This test method is useful for the determination of package and application viscosities of a number of paints

and other coatings and in the thinning of these materials, but is limited to Newtonian or near-Newtonian liquids.

5.2 There are other types of apparatus for measuring viscosity in the laboratory that produce more accurate results.

6. Apparatus

6.1 *Ford Viscosity Cups*—Nos. 1, 2, 3, 4, and 5 Ford viscosity cups made of corrosion- and solvent-resistant materials assembled as complete units (Note 1), and conforming to the dimensional requirements shown in Fig. 1. The orifice dimensions are considered as a guide only as the combination of cup and orifice dimensions must permit conformance to the flow formula for each cup as listed in the Appendix.

NOTE 1—If the orifice is removed from the cup for any reason the cup should be recalibrated before use as described in the Appendix.

6.2 *Thermometer*—Saybolt Viscosity Thermometer conforming to the requirements for Thermometer 17C (19 to 27°C) or 17F (66 to 80°F) as prescribed in Specification E 1.

6.3 *Timing Device*—Any timing device may be used providing that the readings can be taken with a discrimination of 0.2 s or better.

7. Test Specimen

7.1 The specimen of the material to be tested shall be visibly homogeneous and free of any foreign material or air bubbles.

8. Temperature of Testing

8.1 All measurements with the Ford viscosity cups shall be made at 77°F (25°C) or a temperature agreed upon between producer and user. Temperature drift during the test should be kept to a minimum and should not exceed $\pm 0.4^\circ\text{F}$ ($\pm 0.2^\circ\text{C}$).

NOTE 2—It is impossible to predict the effect of temperature change on each material with which the apparatus may be used. This factor may be less than 1 % per degree Celsius for some liquids whereas others may be as high as 8 to 10 % per degree Celsius.

9. Calibration

9.1 Cups should be calibrated in accordance with the procedure described in the Appendix. The frequency of this calibration check depends upon the amount of use and the care that the individual cup receives. If the cup varies more

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 1200 - 52 T. Last previous edition D 1200 - 82.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 14.03.

than 10 % from standard, it should not be used.

10. Conditioning

10.1 Bring the material to a temperature a few degrees below that desired and then agitate vigorously for 10 min on a reciprocating shaker in a pint can two-thirds full. Allow to stand undisturbed for 10 min while adjusting further to the desired temperature. Make the viscosity determination at the end of the 10-min period.

11. Procedure

11.1 Make viscosity determinations in a room free of drafts and rapid changes in temperature. For the highest degree of precision the room temperature should be between 72 and 82°F (22 and 28°C). Determinations should be made at a temperature above the dew point of the atmosphere surrounding the apparatus.

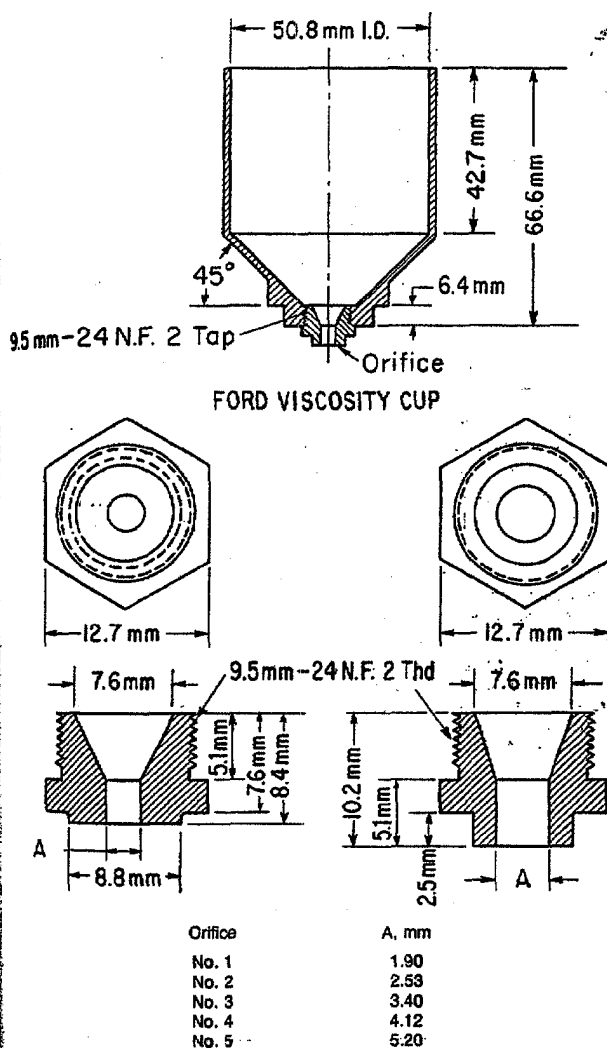


FIG. 1 Ford Viscosity Cup and Orifices

11.2 Choose the proper cup so that the time of efflux will be between 20 and 100 s (preferably between 30 and 100 s) for cup Nos. 3, 4, and 5; between 55 and 100 s for cup No. 1; and between 40 and 100 s for cup No. 2 (Fig. 2).

11.3 Level the instrument so that a cup may be filled level full without a meniscus or overflow at one side.

11.4 Determine the time in seconds of efflux as follows: Close the orifice, for example, by holding a rubber stopper against it. Fill the cup with the prepared specimen. The preferred method is to overfill the cup and scrape off the excess with a straightedge. Pull the stopper away and simultaneously start the timing device. Measure the time until the first break in the stream.

11.5 Measure the temperature of the fluid in the efflux stream.

11.6 If the cup has been established to be nonstandard when calibrated as described in the Appendix, apply the percent difference to the measured seconds to get the corrected viscosity in Ford-cup seconds.

12. Care of Cup

12.1 Following each determination, clean the cup by the use of a suitable solvent and a soft brush. Under no conditions should metal cleaning tools be brought into contact with the instrument. Particular care must be exercised in cleaning the orifice to avoid any film deposit or nicks on the inside walls.

13. Report

13.1 Report the following information:

13.1.1 The efflux time to the nearest 0.2 s for the cup orifice combination (for example, viscosity 33.2 s with No. 4 Ford-cup), the temperature of the test specimen (as in the efflux stream), and the immediate history of agitation and rest prior to the measurement.

14. Precision and Bias

14.1 On the basis of an interlaboratory test of this test method in which eight cooperators from four different laboratories made measurements on five different paints, the within-laboratory coefficient of variation was found to be 2.8 % with 35 df and the between-laboratory coefficient of variation was found to be 6.9% with 30 df. Based on these coefficients, the following criteria should be used for judging

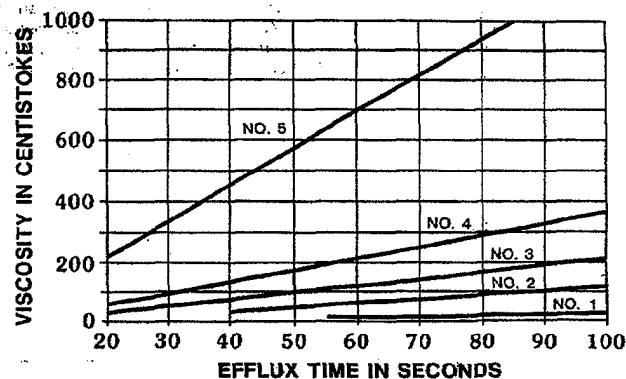


FIG. 2 Approximate Viscosity Curves for Ford Cups

the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Two results obtained by the same operator on different days should be considered suspect if they differ by more than 8 %.

14.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if

they differ by more than 20 %.

15. Index Terms

15.1 This test method is indexed under the following terms: flow cup; viscometers—Ford; Ford cup.

APPENDIX

X1. Calibration Procedure for Ford Cups

X1.1 The orifice of the Ford cup is commonly made of brass or some other corrosion-resistant material which is subject to wear with use and cleaning. A small change in diameter of the orifice becomes significant in the results obtained with the use of this type of viscosity-measuring apparatus.

X1.2 The viscosity standards⁴ are available only as 1-pt. samples.

X1.3 Select the appropriate liquid viscosity standard for the cup to be calibrated (see Table X1.1). Bring this cup and the liquid viscosity standard to a constant temperature as close as possible to 77.0°F (25.0°C) or to the operating temperature of the cup. Determine the time of efflux to the nearest 0.2 s using the procedure detailed in Section 11. Keep the temperature drift to within $\pm 0.4^\circ\text{F}$ ($\pm 0.2^\circ\text{C}$). If the temperature is not 77°F, the actual temperature must be noted and the viscosity of the standard oil corrected to this temperature.

X1.4 The following formulas are used to convert the time of flow in seconds, t , to kinematic viscosity V :

$$V_1 = 0.49 (t - 35.0)$$

$$V_2 = 1.44 (t - 18.0)$$

$$V_3 = 2.31 (t - 6.58)$$

$$V_4 = 3.85 (t - 4.49)$$

$$V_5 = 12.1 (t - 2.00)$$

where V_1 , V_2 , V_3 , V_4 , and V_5 = kinematic viscosity using orifice No. 1, 2, 3, 4, and 5, respectively, cSt.

TABLE X1.1 Viscosity Standards Recommended for Calibrating Ford Viscosity Cups

Cup Number	Approximate Cup Viscosity Range, cSt	Standard Oil Designation	Approximate Designated Viscosity at 77°F (25°C), cSt ⁴
1	10 to 35	S-10	20
2	25 to 120	S-20	35
3	49 to 220	S-60	120
4	70 to 370	S-60	120
5	200 to 1200	S-200	460

⁴ Exact viscosities are supplied with the oil samples.

X1.5 The difference between the certified viscosity and the determined viscosity, multiplied by 100 and divided by the certified viscosity, will give the percent variation of the cup from standard. A percent correction can be applied to the seconds flow when the cup is in normal use. If the cup varies more than 10 % from standard, it is recommended that the orifice be replaced and that the cup be recalibrated.

⁴ Certified kinematic viscosity standards are available from the Cannon Instrument Co., P.O. Box 16, State College, Pa. 16801. For particular oils applicable for use with the Ford Cups refer to Table X1.1. Oils available from other sources, having known kinematic viscosities, may also be used.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)¹

This standard is issued under the fixed designation D 1209; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 4243.1 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Paragraph 1.3 and Footnotes 6, 7, and 10 were added editorially and subsequent footnotes renumbered in March 1988.

1. Scope

1.1 This test method describes a procedure for the visual measurement of the color of essentially light colored liquids (Note 1). It is applicable only to materials in which the color-producing bodies present have light absorption characteristics nearly identical with those of the platinum-cobalt color standards used.

NOTE 1—A procedure for estimating color of darker liquids, described for soluble nitrocellulose base solutions, is given in Methods D 365.

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements see Section 6.

1.3 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

2. Referenced Documents

2.1 ASTM Standards:

- D 156 Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)²
- D 365 Test Methods for Soluble Nitrocellulose Base Solutions³
- D 1193 Specification for Reagent Water⁴
- E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁵
- E 202 Method for Analysis of Ethylene Glycols and Propylene Glycols⁵
- E 346 Method for Analysis of Methanol⁶

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates. Current edition approved April 27, 1984. Published August 1984. Originally published as D 1209 - 52. Last previous edition D 1209 - 79.

² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁵ Annual Book of ASTM Standards, Vol 15.05.

⁶ Annual Book of ASTM Standards, Vol 15.05.

3. Significance and Use

3.1 The property of color of a solvent varies in importance with the application for which it is intended, the amount of color that can be tolerated being dependent on the color characteristics of the material in which it is used. The paint, varnish, and lacquer solvents, or diluents commercially available on today's market normally have little or no color. The presence or absence of color in such material is an indication of the degree of refinement to which the solvent has been subjected or of the cleanliness of the shipping or storage container in which it is handled, or both.

3.2 For a number of years the term "water-white" was considered sufficient as a measurement of solvent color. Several expressions for defining "water-white" gradually appeared and it became evident that a more precise color standard was needed. This was accomplished in 1952 with the adoption of Test Method D 1209 using the platinum-cobalt scale. This test method is similar to the description given in *Standard Methods for the Examination of Water and Waste Water*⁷ and is referred to by many as "APHA Color." The preparation of these platinum-cobalt color standards was originally described by A. Hazen in the *American Chemical Journal*⁸ in which he assigned the number 5 (parts per ten thousand) to his platinum-cobalt stock solution. Subsequently, in their first edition (1905) of *Standard Methods for the Examination of Water*, the American Public Health Association, using exactly the same concentration of reagents, assigned the color designation 500 (parts per million) which is the same ratio. The parts per million nomenclature is not used since color is not referred directly to a weight relationship. It is therefore recommended that the incorrect term "Hazen Color" should not be used. Also, because it refers primarily to water, the term "APHA Color" is undesirable. The recommended nomenclature for referring to the color of organic liquids is "Platinum-Cobalt Color, Test Method D 1209."

3.3 The petroleum industry uses the Saybolt colorimeter Test Method D 156 for measuring and defining the color of hydrocarbon solvents; however, this system of color measurement is not commonly employed outside of the petro-

⁷ *Standard Methods for the Examination of Water and Waste Water*, M. Franson, Ed., American Public Health Assoc., 14th ed., 1975, p. 65.

⁸ Hazen, A., "New Color Standard for Natural Waters," *American Chemical Journal*, Vol XIV, 1892, p. 300-310.

TABLE 1 Absorbance Tolerance Limits For No. 500 Platinum-Cobalt Stock Solution

Wavelength, nm	Absorbance
430	0.110 to 0.120
455	0.130 to 0.145
480	0.105 to 0.120
510	0.055 to 0.065

leum industry. It has been reported by various sources that a Saybolt color of +25 is equivalent to 25 in the platinum-cobalt system or to colors produced by masses of potassium dichromate ranging between 4.8 and 5.6 mg dissolved in 1 L of distilled water. Because of the differences in the spectral characteristics of the several color systems being compared and the subjective manner in which the measurements are made, exact equivalencies are difficult to obtain.

4. Apparatus

4.1 *Spectrophotometer*, equipped for liquid samples and for measurements in the visible region.⁹

NOTE 2—The spectrophotometer used must be clean and in first-class operating condition. The instrument should be calibrated in accordance with the instructions given in the Standards for Checking the Calibration of Spectrophotometers (200 to 1000 nm).¹⁰

4.2 *Spectrophotometer Cells*, matched having a 10-mm light path.

4.3 *Color Comparison Tubes*—Matched 100-mL, tall-form Nessler tubes, provided with ground-on, optically clear, glass caps. Tubes should be selected so that the height of the 100-mL graduation mark is 275 to 295 mm above the bottom of the tube.

4.4 *Color Comparator*—A color comparator constructed to permit visual comparison of light transmitted through tall-form, 100-mL Nessler tubes in the direction of their longitudinal axes. The comparator should be constructed so that white light is passed through or reflected off a white glass plate and directed with equal intensity through the tubes, and should be shielded so that no light enters the tubes from the side.¹¹

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.¹² Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water

⁹The Beckman Model B and its equivalents have been found satisfactory for this purpose.

¹⁰See National Bureau of Standards Letter Circular LC-1017.

¹¹A unit available from Scientific Glass and Instruments, Inc., P.O. Box 6, Houston, TX 77001, has been found suitable for this purpose.

¹²"Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

TABLE 2 Platinum-Cobalt Color Standards

Color Standard Number	Stock Solution, mL	Color Standard Number	Stock Solution, mL
5	1	70	14
10	2	100	20
15	3	150	30
20	4	200	40
25	5	250	50
30	6	300	60
35	7	350	70
40	8	400	80
50	10	450	90
60	12	500	100 ^A

^A This is platinum-cobalt color No. 10 in Methods D 365.

conforming to Type IV of Specification D 1193.

5.3 *Cobalt Chloride* ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$).

5.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.5 *Potassium Chloroplatinate* (K_2PtCl_6).

6. Hazards

6.1 Concentrated hydrochloric acid is a corrosive chemical.

7. Platinum-Cobalt Reference Standards

7.1 *Platinum-Cobalt Stock Solution*—Dissolve 1.245 g of potassium chloroplatinate (K_2PtCl_6) and 1.00 g of cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) in water. Carefully add 100 mL of hydrochloric acid (HCl, sp gr 1.19) and dilute to 1 L with water. The absorbance of the 500 platinum-cobalt stock solution in a cell having a 10-mm light path, with reagent water in a matched cell as the reference solution,¹³ must fall within the limits given in Table 1.

7.2 *Platinum-Cobalt Standards*—From the stock solution, prepare color standards in accordance with Table 2 by diluting the required volumes to 100 mL with water in the Nessler tubes. Cap the tubes and seal the caps with shellac or a waterproof cement. When properly sealed and stored, these standards are stable for at least 1 year and do not degrade markedly for 2 years.¹⁴

7.2.1 For a more precise measurement of light colors below 15 platinum-cobalt, prepare color standards from the stock solution in accordance with Table 3 by diluting the required volumes to 100 mL with water in the Nessler tubes. Use a semi-microburet for measuring the required amount of stock solution.

8. Procedure

8.1 Introduce 100 mL of specimen into a Nessler tube, passing the specimen through a filter if it has any visible turbidity. Cap the tube, place in the comparator, and compare with the standards.

9. Report

9.1 Report as the color the number of the standard that

¹³See the manufacturer's instruction manual for complete details for operating the spectrophotometer.

¹⁴Scharf, W. W., Ferber, K. H., and White, R. G., "Stability of Platinum-Cobalt Color Standards," *Materials Research and Standards*, Vol 6, No. 6, June 1966, pp. 302-304.

TABLE 3 Platinum-Cobalt Color Standards for Very Light Colors

Color Standard Number	Stock Solution, mL	Color Standard Number	Stock Solution, mL
1	0.20	9	1.80
2	0.40	10	2.00
3	0.60	11	2.20
4	0.80	12	2.40
5	1.00	13	2.60
6	1.20	14	2.80
7	1.40	15	3.00
8	1.60		

most nearly matches the specimen. In the event that the color lies midway between two standards, report the darker of the two.

9.2 If, owing to differences in hue between the specimen and the standards, a definite match cannot be obtained, report the range over which an apparent match is obtained, and report the material as "off-hue."

10. Precision¹⁵

10.1 Color Standards:

10.1.1 These precision statements are based upon an interlaboratory study in which five platinum-cobalt standards having values of 25, 75, 170, 385, and 475 were prepared in accordance with the instructions given in Section 7 of this test method and were given coded labels. These solutions were tested by one analyst in each of ten different laboratories making a single observation on one day and then repeating the observation on a second day. The analysts were requested to estimate the color to the nearest one unit for solutions below 40 platinum-cobalt, to the nearest five units for solutions between 40 and 100 platinum-cobalt and to the nearest ten units for solutions above 100 platinum-

cobalt. In this interlaboratory study, the within-laboratory coefficient of variation was found to be 1.8 % with 60 degrees of freedom, and the between-laboratories coefficient of variation was found to be 5.3 % with 54 degrees of freedom. Based on these results, the following criteria, calculated in accordance with Practice E 180, should be used for judging the acceptability of results at the 95 % confidence level when the results are obtained under optimum conditions where the hue of the sample matches exactly the hue of the standards. Poorer precision will be obtained in varying degrees as the hue of the sample departs from that of the standards.

10.1.1.1 *Repeatability*—Two results, obtained by the same analyst on different days, should be considered suspect if they differ by more than 5.1 %.

10.1.1.2 *Reproducibility*—Two results, obtained by analysts in different laboratories, should be considered suspect if they differ by more than 15 %.

10.2 Specimen:¹⁶

10.2.1 In an interlaboratory study of this test method in which the standards described in Table 3 were used, the within-laboratory standard deviation was found to be one platinum-cobalt unit at 56 degrees of freedom and the between-laboratory standard deviation was found to be 2 platinum-cobalt units at 25 degrees of freedom. Based on these standard deviations, the following criteria should be used for judging, at the 95 % confidence level, the acceptability of results obtained on light colored samples.

10.2.1.1 *Repeatability*—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than two platinum-cobalt units.

10.2.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than seven platinum-cobalt units.

¹⁶ These precision statements are based on interlaboratory studies conducted by Committee E-15 on Industrial Chemicals on samples of ethylene glycol and methanol as reported in Method E 202, Method E 346, and research report RR: E15-28.

¹⁵ Supporting data are available from ASTM Headquarters. Request RR: D01-1024.

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Standard Test Method for Fineness of Dispersion of Pigment-Vehicle Systems¹

This standard is issued under the fixed designation D 1210; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Editorial changes were made throughout in June 1988.

1. Scope

1.1 This test method covers measurement of the degree of dispersion (commonly referred to as "fineness of grind") of the pigment in a pigment-vehicle system such as liquid coatings and their intermediates. It may also be used to assess the inclusion of particulates by a cleanliness rating.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 The product is spread by means of a scraper in a calibrated tapered path. At some point in this path, particles or agglomerates, or both, will become visible. A direct reading from the calibrated scale is then made at the point where the particles form a definite pattern. When the single path gage is used it is also possible to rate "cleanliness" (see 6.2).

3. Significance and Use

3.1 In making pigmented products, the pigment is usually dispersed in a portion of the vehicle in some sort of mill. At this stage, it is necessary to be able to judge if the pigment agglomerates have been sufficiently broken up so as not to interfere with the smoothness of the finished coating film. This test method describes a way of making this judgment.

4. Apparatus

4.1 **Gage**—A hardened steel, stainless steel, or chrome-plated steel block (Fig. 1) approximately 6.7 in. (170 mm) in length, and 0.6 in. (15 mm) in thickness. The top surface of the block shall be ground smooth and planar and shall contain one or two paths 5 in. (127 mm) in calibrated length. The path shall be tapered uniformly in depth lengthwise from 100 μ m (about 4 mils) at 10 mm from one end to zero depth at the other with intermediate calibrations in accordance with the depth at those points. Preferred calibrations are

Hegman units and micrometres (Note 1). Two path widths are covered by this test method:

4.1.1 Two parallel paths each 0.5 in. wide (12.5 mm) and spaced 0.5 in. apart centered in a block 2.5 in. (65 mm) wide.

4.1.2 One path 2 in. (50 mm) in width centered in a block 3.5 in. (90 mm) wide.

NOTE 1—Several arbitrary scales and modifications of the gage are used by industry. In order that readings obtained with these arbitrary scales and modifications can be reported in the preferred units, the approximate relationship of these scales to gage depth is shown in the following example:

Hegman Scale ^A	Depth, μ m ^B	Depth, mils ^B	PC or FSPT Scale ^C	NPIRI Scale ^D
0	100	4	0	40
1	90	3.5	1/4	35
2	75	3	2/4	30
3	65	2.5	3/4	25
4	50	2	5	20
5	40	1.5	6/4	15
6	25	1	7/4	10
7	15	0.5	8/4	5
8	0	0	10	0

^A Sometimes referred to in error as the North Standard scale.

^B Rounded to nearest 5 μ m or 0.5 mil.

^C Federation of Societies for Paint Technology scale.

^D National Printing Ink Research Institute scale, 0 to 10 on the NPIRI Production Grindometer, but extended on many gages to 20 or 30.

4.2 **Scraper**—A double-edged hardened steel, stainless steel, or chrome-plated steel blade (Fig. 2) 3.75 in. (95 mm) long, 1.5 in. (38 mm) wide, and 0.25 in. (6.4 mm) thick. The two edges on the 3.75-in. sides shall be rounded to a radius of 0.015 in. (0.38 mm).

5. Care of Gage

5.1 Clean the gage immediately after each use. Use a solvent and a soft cloth. Keep the gage covered or encased at all times when not in use. Protect gages that lie idle for extended periods of time from rust with an oil coating or oil-soaked wrap.

5.2 Do not allow any hard materials to come in contact with the gage surface or scraper in any manner that might result in scarring or nicking. Avoid tapping or scratching with other metal.

5.3 The scraper may be rendered unsatisfactory for use by wear or nicks of the contact edge or warpage (Note 2). Replace or recondition unsatisfactory blades.

NOTE 2—Wear or warpage of the scraper may be noted by facing the edge of the scraper down on the smooth level face of the gage, then

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials, and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paint and Paint Materials.

Current edition approved Nov. 30, 1979. Published January 1980. Originally published as D 1210 - 52. Last previous edition D 1210 - 78.

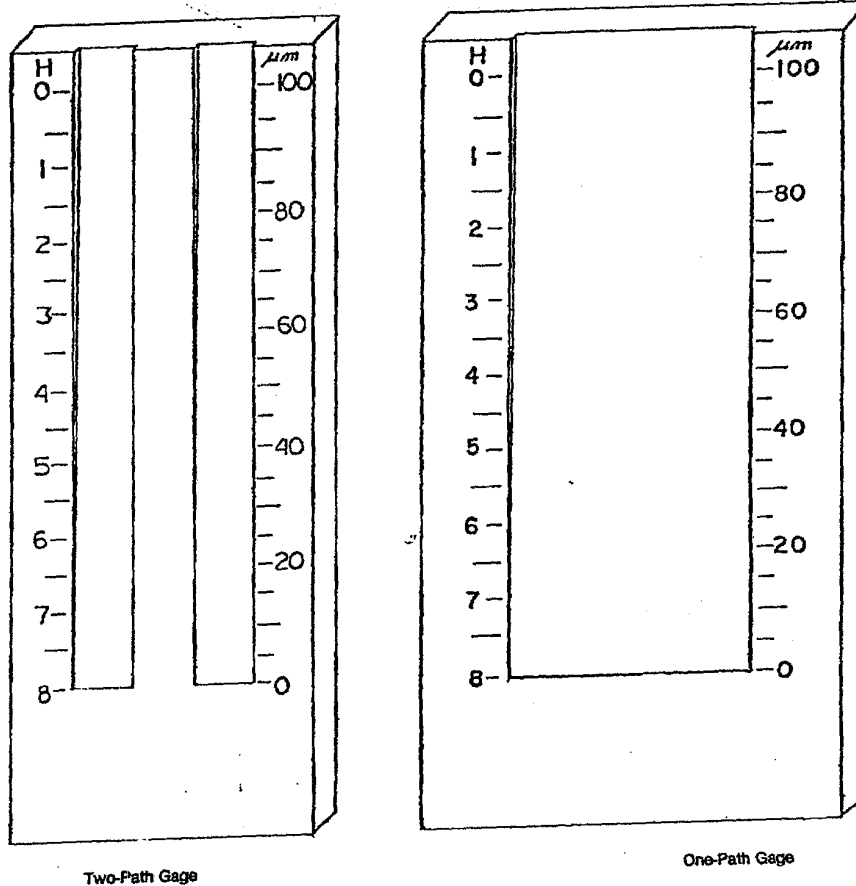


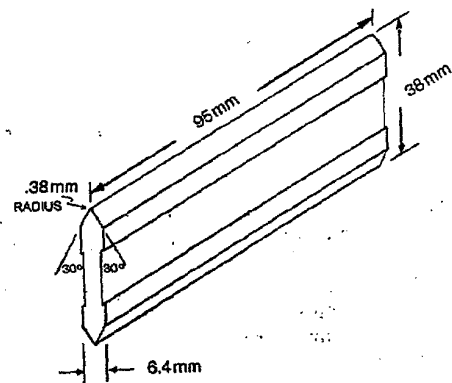
FIG. 1 Fineness Gages

inspecting the contact edge by means of a strong light, placed behind the gage. Rocking the scraper forward or back will reveal poor contact due to wear or warpage. Any light coming through between scraper and gage face shows that the scraper has been damaged and is not satisfactory for use.

6. Visual Standards

6.1 The diagrams in Fig. 3 are reproductions of six typical fineness gage patterns with the double-path gage in 4.1.1, and they should be viewed with the purpose of standardizing the relationship of particle distribution to fineness designation. The arrow in each drawing represents the end point (reading) for that distribution. These patterns are to be used for notation of frequency of particles and should not be interpreted according to the size of the dots. Although called "standards," they are really examples of fineness readings to be used as a guide, since no two particle distributions will be exactly the same.

6.2 Similarly, Fig. 4 exhibits typical fineness gage patterns for the 2-in. (51-mm) gage in 4.1.2. These diagrams are to be used like those for the double-path gage except that a "cleanliness" rating is also shown. "Cleanliness" is descrip-



NOTE—1 in. = 25.4 mm
FIG. 2 Scraper

tive of the number of particles that appear in the path above the fineness designation. Three ratings are indicated: A (0 to 8 specks), B (9 to 15 specks), and C (16 or more specks).

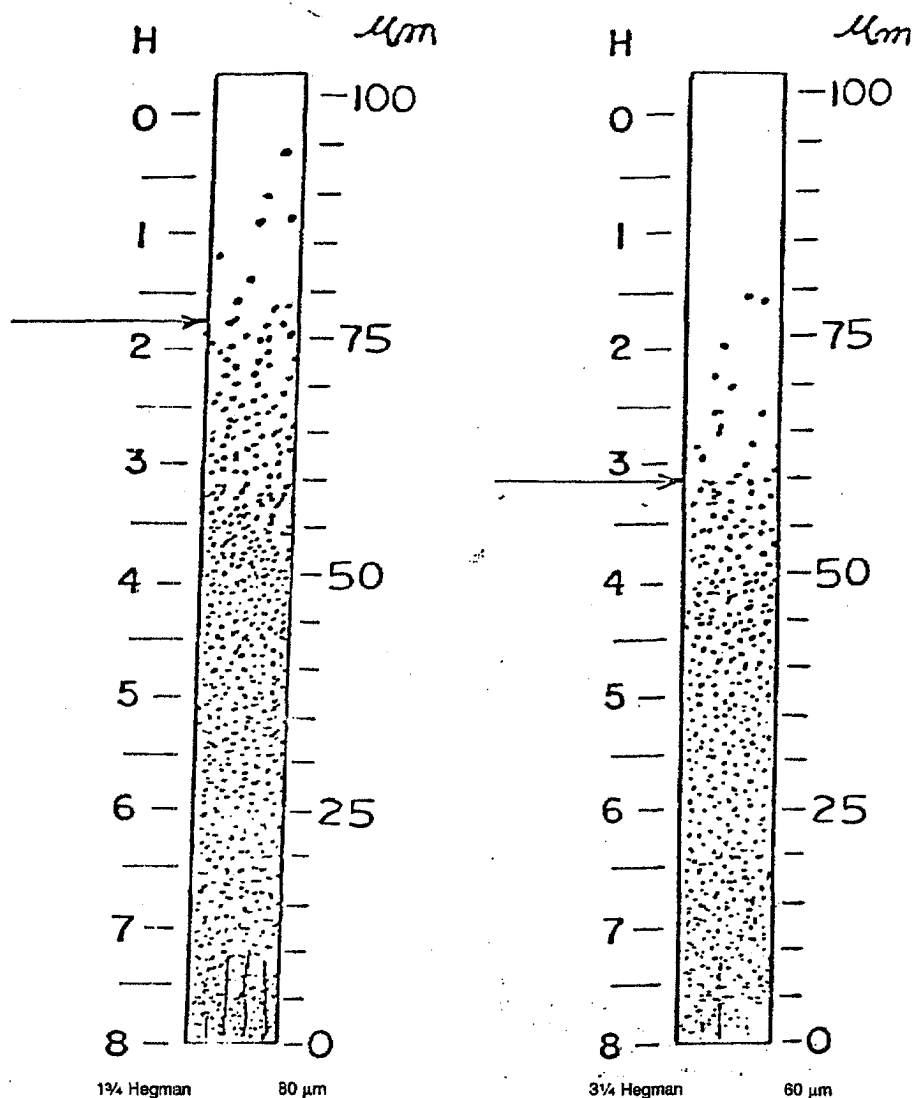


FIG. 3a Typical Fineness Gage Patterns

7. Procedure

7.1 Place the gage on a horizontal flat, nonslippery surface and wipe clean immediately before the test. Be sure the gage surface is free of lint.

7.2 Hand stir the specimen (Note 3) vigorously for 2 min, taking care that air bubbles are not whipped into the paint. To be sure of an accurate grind reading, specimens must be free of air bubbles.

NOTE 3—For this test method to function properly, the pigment particles in the specimens to be tested should be free to settle to the bottom of the gage channel after the drawdown. Therefore, before testing, high-viscosity intermediate specimens that have little ability to flow should be reduced with a compatible liquid. Reduction should be in approximately the same proportion as the intermediate will be reduced in practice.

7.3 Immediately place the material to be tested in the deep end of the path, or paths, so that it overflows the path

slightly. When using the double-path gage, place material in both paths.

7.4 Holding the scraper in both hands, nearly vertical but inclined slightly toward the operator, draw the material down the length of the path toward the shallow end of the gage with a uniform, brisk motion in approximately 1 to 2 s. Exert upon the scraper only sufficient pressure to clean excess material from the face of the gage. Within 10 s of placing the specimen on the gage, make a reading as follows:

7.4.1 View the gage from the side, perpendicular to the length of the path. Keeping the gage between the operator and the light source, make the angle between the face of the gage and the line of vision between 20 and 30°.

NOTE 4—Clear finishes may have to be viewed at a lower angle or they may have to be opacified with a finely ground colorant or dye in order to see better the particles of flattening pigment.

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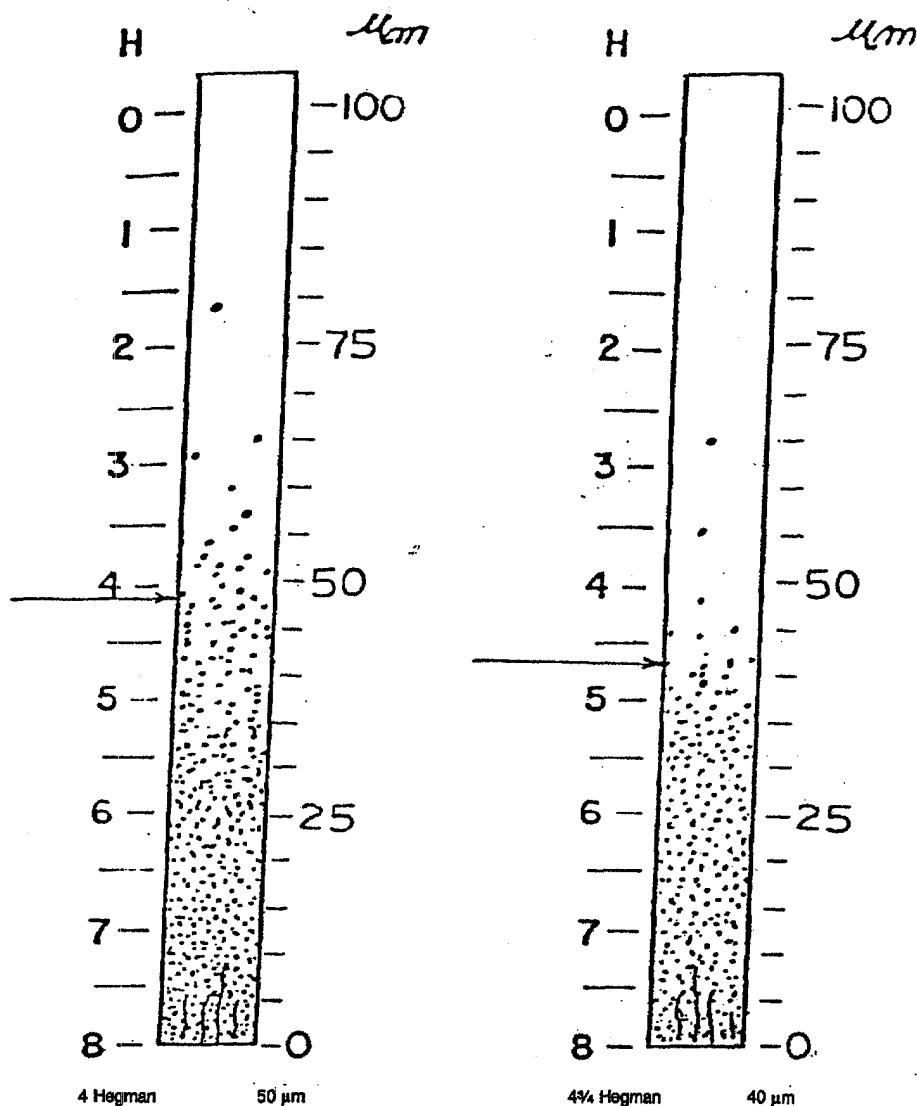


FIG. 3b Typical Fineness Gage Patterns

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7.4.2 Observe the point where the material first shows a definite speckled pattern, not just isolated specks (see Figs. 3 and 4). This is the fineness reading. When using the two-path gage, average the values in the two paths to the nearest 1/4 Hegman unit. This average is considered one reading.

7.5 After the first drawdown and reading, which are preliminary for establishing proper test conditions and locating the position of the fineness reading, repeat the procedure twice, beginning with 7.3, to obtain two test readings. This process allows the two test readings to be made with limited time lapses between completion of drawdown and reading. (Do not consider any reading for the reported fineness when the time lapse exceeds 10 s.) Average the two readings to the nearest 1/4 Hegman units (5 μ m).

7.6 Interpretation of Dispersion Pattern:

7.6.1 Inspect the initial drawdown for pattern and the

approximate fineness. Determine the point in the particle distribution that approximates a similar end point pattern to that of the pictorial standards.

7.6.2 Judge cleanliness on the one-path gage either by comparison to the typical fineness patterns (shown only at a 6 Hegman level but applicable by analogy to any fineness level) or by counting nibs coarser than the selected fineness level (see 6.2 for cleanliness ranges).

8: Report

8.1 Report the following information:

8.1.1 Whether the two-path gage in 4.1.1 or the one-path gage in 4.1.2 was used.

8.1.2 The average of two readings conforming to the conditions of 7.6. Report micrometre readings to the nearest

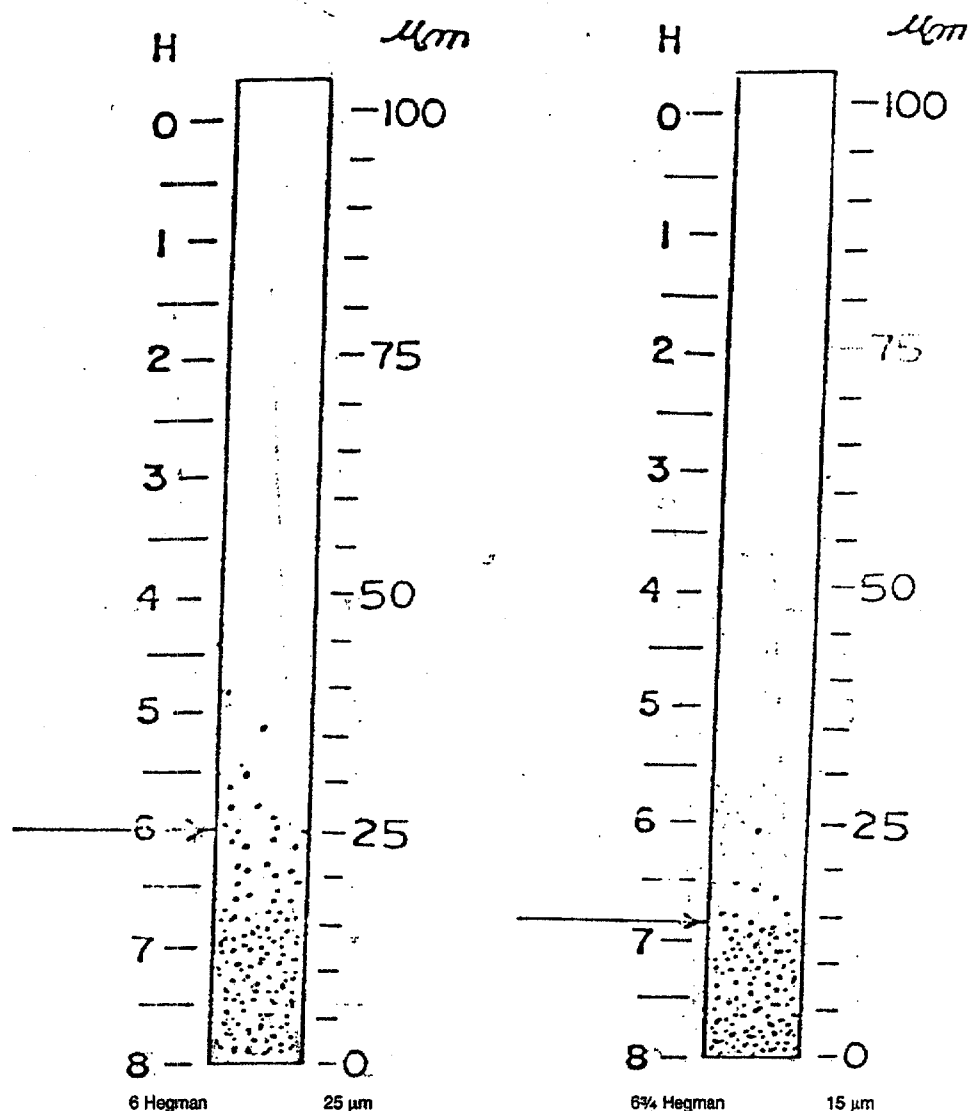


FIG. 3c Typical Finesse Gage Patterns

multiple of 5 μm and Hegman readings to the nearest $\frac{1}{4}$ unit.

8.1.3 Cleanliness may be reported also when using the two-path gage.

9. Precision and Bias²

9.1 On the basis of an interlaboratory test of this test method in which 23 operators in 7 laboratories tested 6 samples of paints covering a broad range of compositions and finenesses, the single-operator standard deviation was found to be 0.27 Hegman units (3.4 μm), the within-laboratory standard deviation was found to be 0.70 Hegman units (8.8 μm) and the between-laboratories standard deviation was found to be 0.74 Hegman units (9.3 μm). Based on

these standard deviations and the requirement that readings are to be reported to the nearest $\frac{1}{4}$ Hegman unit or multiple of 5 μm , the following criteria should be used for judging the precision of results at a 95% confidence level:

9.1.1 *Repeatability*—Two results obtained by a single operator should be considered suspect if they differ by more than $\frac{1}{4}$ Hegman unit (10 μm).

9.1.2 *Reproducibility*—Two results, each the mean of two readings, obtained by different operators in the same or different laboratories should be considered suspect if they differ by more than $2\frac{1}{4}$ Hegman units (25 μm).

10. Index Terms

10.1 This test method is indexed under the following terms: pigment dispersion (paint); fineness of grind (dispersion); Hegman scale.

² Supporting data available from ASTM Headquarters. Request RR: D 01 - 1017.

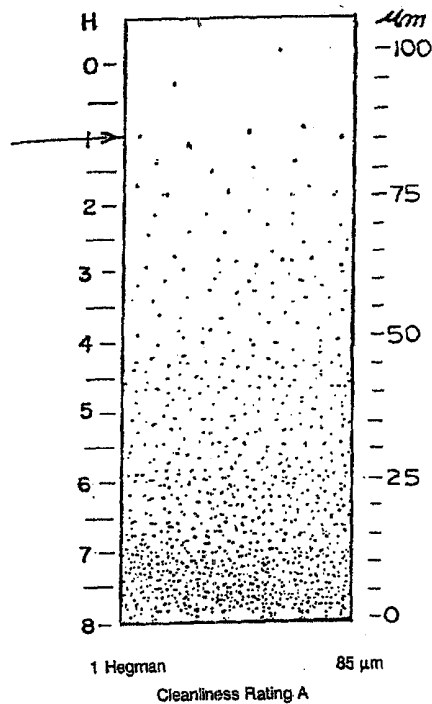


FIG. 4a Typical Fineness Gage Pattern

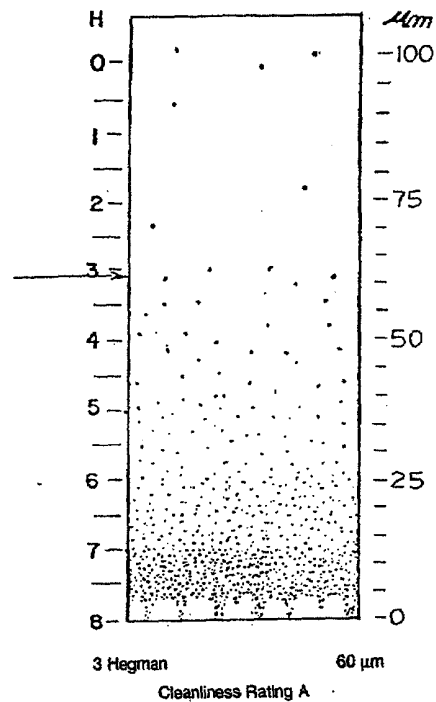


FIG. 4c Typical Fineness Gage Pattern

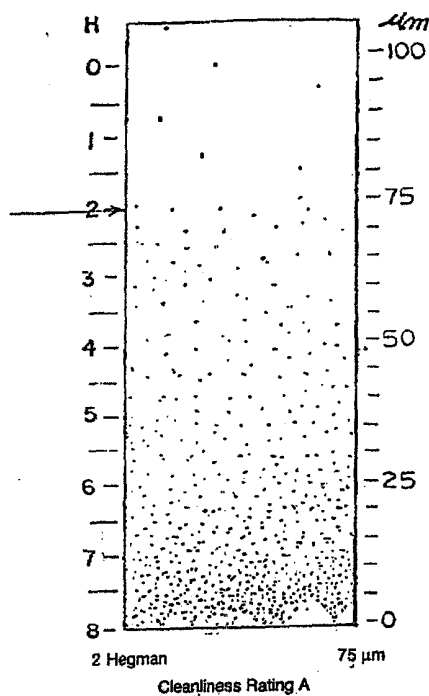


FIG. 4b Typical Fineness Gage Pattern

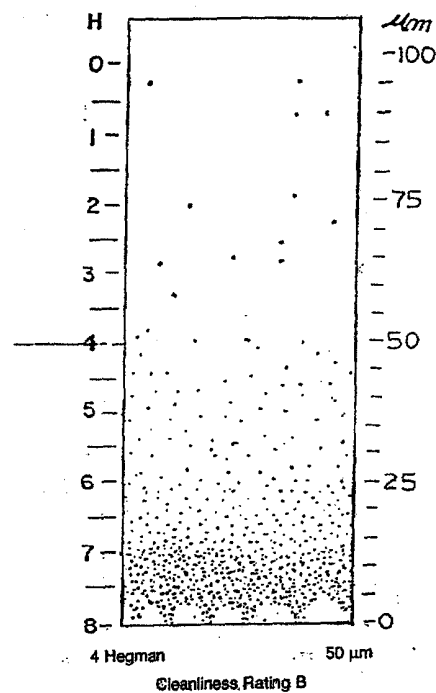


FIG. 4d Typical Fineness Gage Pattern

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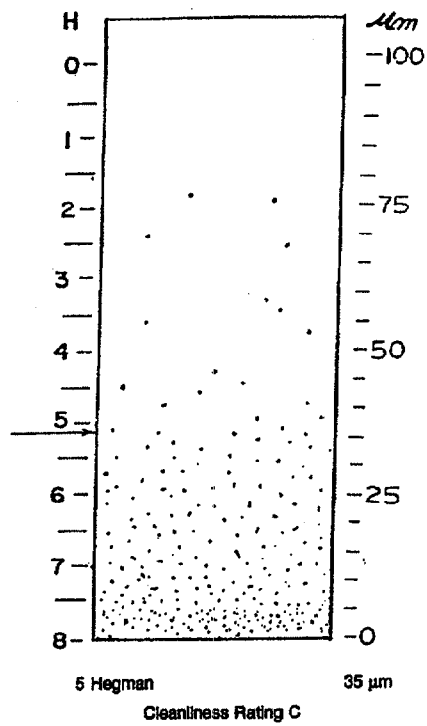


FIG. 4e Typical Fineness Gage Pattern

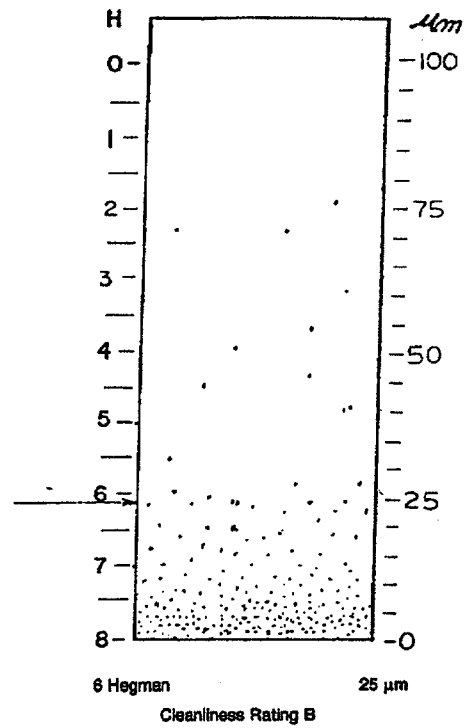


FIG. 4g Typical Fineness Gage Pattern

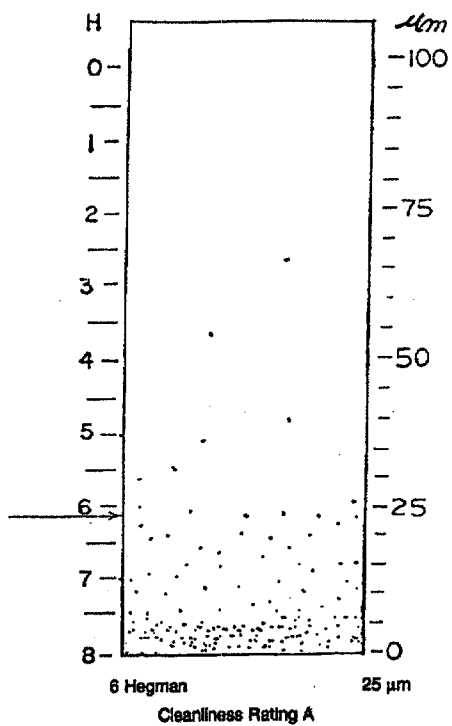


FIG. 4f Typical Fineness Gage Pattern

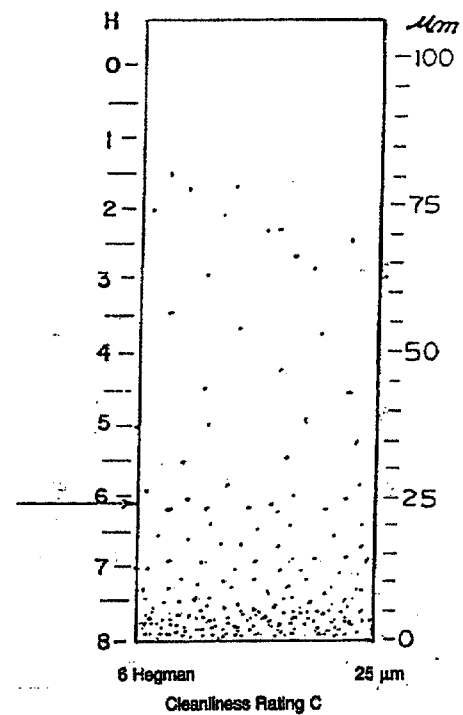


FIG. 4h Typical Fineness Gage Pattern

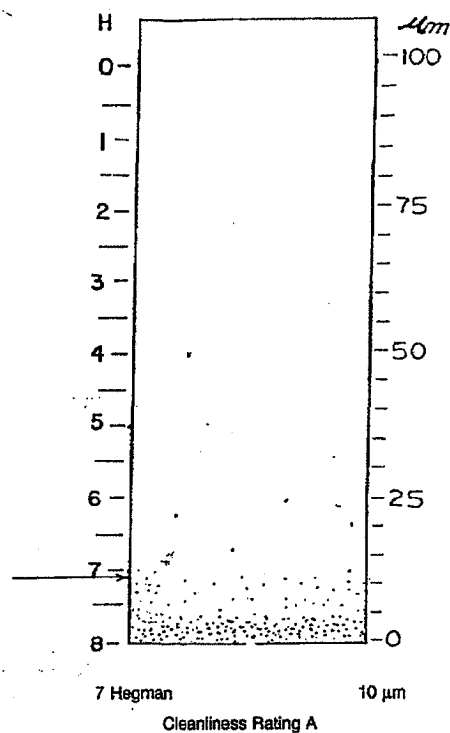


FIG. 41 Typical Fineness Gage Pattern

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood¹

This standard is issued under the fixed designation D 1211; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of the resistance to checking and cracking of clear nitrocellulose lacquer films applied to wood or plywood substrates when subjected to sudden changes from high to low temperatures.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D805 Testing Veneer, Plywood, and Other Glued Veneer Constructions²

D2571 Guide for Testing Wood Furniture Lacquers³

3. Description of Terms Specific to This Standard

3.1 True cold-checks on solid wood show as one or more straight cracks on the applied film. Cold-checks manifest themselves in either of two ways:

3.1.1 Long continuous wavy lines with the grain or at various directions at angles that can be perpendicular to the direction of the grain.

3.1.2 Innumerable fine lines erratic in direction and length forming a network over a portion or all of the panel. This effect may be likened to crazing of the lacquer film.

3.1.3 On plywood the direction of the cracks will often vary because of the stresses set up by other than the top stratum. Therefore, all checks may be considered as failures, and appropriate notations on the character of the cracks must be made to assist in the interpretation. While it is recognized that cracks in the substrate may occur (veneer checking), failures observed in the lacquer coating may be due to action of moisture (humidity) or of cold, or both. Checking caused by moisture appears along the grain and is characterized by short cracks (usually not more than $\frac{1}{2}$ in. (13 mm) in length) occurring either singly or in clusters. These lines or clusters may progress along the grain in a discontinuous fashion. Should either veneer checking or

moisture checking be observed, the test should be discontinued, and rerun.

3.2 The checks may be perceptible only by visual observation of the panels at an angle, under a strong light, or the cracks may be wide enough to be readily discernible in direct daylight.

4. Summary of Test Method

4.1 Lacquer-coated wood panels are subjected first to a high temperature, followed by an exposure to low temperature, and then a return to room temperature for given periods of time, which constitutes one cycle. The method does not define the number of cycles which a lacquer must withstand, this condition being a requirement which must be agreed upon by the purchaser and the seller.

5. Significance and Use

5.1 A common type of failure of clear films applied to wood is cracking or checking that may occur over a period of time either with the grain or at an angle. This cold check test is designed to accelerate the appearance of checks or cracks by cycling the temperature.

NOTE 1—Except when elaborate precautions are taken to control the moisture content of the wood before and after finishing and during the test, failures may quite often occur as a result of dimensional changes in the wood due to moisture change rather than temperature change.

6. Apparatus

6.1 *Refrigerator (Dry Ice)*—The refrigerator should be a well-insulated box, cooled by air which has been circulated over solid carbon dioxide (dry ice). The air must strike the panels indirectly after passing over the solid carbon dioxide. The unit shall have a capacity and temperature control sufficient to cool the test panels in accordance with the requirements prescribed in Section 9.

6.2 *Mechanical Refrigerator*—As a less desirable alternative, a well-insulated mechanical refrigerator may be used having sufficient capacity to cool the test panels in accordance with the requirements prescribed in Section 9. The refrigerator should have some means for providing air circulation. It should be understood that results obtained with a mechanical refrigerator may not correspond to results obtained with a "dry ice" refrigerator. Consequently the type of unit employed shall be stated in the report.

6.3 *Oven*—The oven shall be electrically heated and of any standard type capable of being automatically controlled and of sufficient capacity to heat the panels under test to $120 \pm 5^\circ\text{F}$ ($49 \pm 3^\circ\text{C}$) within 1 h. A circulating fan shall be installed in the oven.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 1211 - 52. Last previous edition D 1211 - 74 (1980) ^{ϵ} .

² Discontinued; see 1976 *Annual Book of ASTM Standards*, Part 22.

³ *Annual Book of ASTM Standards*, Vol 06.01.

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7. Test Panels

7.1 Unless otherwise agreed, the test panel shall be cut from synthetic resin-bonded, five-ply plywood with faces and backs made with straight-grain walnut veneer having no sizing or other surface pretreatment. The dimensions of the test panel shall be 6 by 12 in. (152 by 305 mm), and approximately $\frac{3}{16}$ in. (8 mm) thick.

7.2 Solid wood, heavier or lighter veneers, or other species of wood may be agreed upon by the purchaser and the seller. Type of wood and panel size should be clearly defined in the report.

7.3 The wood selected shall have a moisture content of not less than 6.0 % and not more than 8.0 % at the time of testing. The moisture content of the wood may be determined by an electric moisture gage or in accordance with Methods D 805.

7.4 Panels may be conditioned to a moisture content of 7 to 8 % under any of the following conditions:⁴

Temperature, °F	Relative Humidity, %
65 to 75	35 to 40
90 to 100	40 to 45
120 to 125	45 to 50

8. Preparation of Panels

8.1 *Five-Ply Plywood* (With faces and backs made with straight-grain, closed face walnut veneer having no sizing or other surface pretreatment) shall be prepared as follows:

8.1.1 Sand panels until smooth with No. 240 sandpaper, or equivalent.

8.1.2 Fill with representative walnut wood paste filler and allow to dry for 24 h at not less than 70 nor more than 80°F.

8.1.3 Spray a sufficient number of coats of about equal thickness of the test lacquer on the panel within an 8-h period to give a total dry film thickness of 3 ± 0.5 mil (75 ± 13 μ m). Allow a minimum period of 2 h between coats. Do not edge or back the panel with any protective coating.

8.1.4 To minimize the effects of variations in the wood, apply a control lacquer of known performance to a portion of each panel in the same manner as the test lacquer. After aging, the panels may be cut (Note 2) into sections finished only with one lacquer, but each section should conform to the description given in Section 6.

NOTE 2—Cutting of panel is optional and may be contingent upon the size and capacity of the test equipment.

8.1.5 The thickness of films applied to panels may be determined with reasonable accuracy by placing a metal panel adjacent to the wood panel being prepared and measuring the film thickness on the metal panel with a magnetic thickness gage. The plywood panel must be sanded and rubbed before testing and the use of waxes or oils should be closely regulated since these can make a difference in the results obtained. For sanding and rubbing procedure, see Practice D 2571.

8.2 *Panels of Five-Ply Plywood with Walnut Veneer for Complete Lacquer System*—Proceed as agreed upon by the purchaser and the seller.

8.3 *Panels of Solid Wood, Lighter or Heavier Veneers, or Other Species of Wood*—Proceed as agreed upon by the purchaser and the seller.

8.4 *General Treatment*—Except where specific agreements exist between the purchaser and the seller, all panels shall be subjected to the following treatment:

8.4.1 Prepare at least three panels for each lacquer or lacquer system and each other variable, such as type of panel, that may be under test.

8.4.2 Air-dry overnight.

8.4.3 Age finished test panels for ten days under conditions that will maintain a moisture content of 6 to 8 %.

9. Procedure

9.1 Place a predetermined number of panels, depending on the capacity of the oven and refrigerator, vertically in racks so that air can pass between all panels. The lacquered sides of the panels shall not face the walls of the heating or cooling units. Place the racks for 1 h in an oven at $120 \pm 5^\circ\text{F}$ ($48.9 \pm 3^\circ\text{C}$) in such a manner as to avoid localized overheating. At the expiration of this period, transfer the panels within 1 min to a refrigerating unit maintained at $-5 \pm 2^\circ\text{F}$ ($-21 \pm 1^\circ\text{C}$) (unless special conditions indicate some other agreement between the purchaser and the seller). The number of panels shall be such that the time required to reach this temperature is not less than 30 min and not more than 45 min. The temperature can be measured by a thermocouple imbedded in the panel just underneath the film.

9.2 Remove the panels from the refrigerator after 1 h exposure. After allowing 15 min relaxation period, inspect the panels under a strong light. Best results will be obtained if the light source is directed from behind and over the shoulder of the observer. Circle checks with crayon, since they will often heal or at least appear to heal and become relatively invisible. Once the capacity of any given oven-refrigerator combination (number of panels that will permit specified rates of heating and cooling) has been determined, make all subsequent tests with this number of panels using dummy panels where necessary. If this precaution is not followed, rates of cooling and heating will differ each time the test is run, and reproducibility will suffer.

9.3 The period of 1 h at 120°F (49°C) followed by 1 h at -5°F (-21°C) and 30 min at room temperature (15 min relaxation period, 15 min to allow for inspection), constitutes one cycle. Repeat this cycle until failure occurs or until a prescribed minimum number of cycles has been obtained.

9.4 The failure end point is defined as the cycles on which innumerable fine lines appear as described in Section 3 or on which a total of four checks (1 to 2 in. (25 to 50 mm) in length) or two checks (more than 2 in. (50 mm) in length) appear in the film. Exclude the outer $\frac{1}{2}$ -in. (13 mm) perimeter of panel when examining for failure (cracks).

9.5 Unless a laboratory operates on three shifts there will be a relaxation period every night and over week ends. The report should show when these rest periods occurred.

10. Report

10.1 Report the following information:

10.1.1 Type of construction, variety, and size of wood panels used, for example, 5-ply, $\frac{3}{16}$ -in. (8-mm) walnut

⁴These conditions were taken from the curve on p. 191 of the *Wood Handbook*, Forest Products Laboratory, Madison, WI.

veneer, 6 by 12 in. (152 by 305 mm),

10.1.2 Type of refrigerator, that is, "dry ice" or mechanical,

10.1.3 Finishing system and schedule of application, and

10.1.4 Number of cycles passed without failure and note of all wood failures, specifying the cycle on which such failures occurred.

11. Precision

11.1 Results on all three panels should fall within ± 2 cycles of the mean of all three panels. If this does not occur, additional panels shall be run until there are at least three panels which fall within this range. All panels that have been run are to be taken into consideration. This requirement also applies to panels finished with the control lacquer. Precision as stated here applies only to the test panels described in Section 7.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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1. Scope

1.1 This standard covers the test method for determining the resistance of film to the action of wet film lacquer.

1.1.1 (English) wet film and up through

1.1.2 measurement (Section

1.2 This test method is applicable to safety of who, appropriate

2. References

2.1 ASTM D 823 Test

3. Summary

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4. Significance and Use

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² Annual.



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Standard Test Methods for Measurement of Wet Film Thickness of Organic Coatings¹

This standard is issued under the fixed designation D 1212; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of wet film thickness of organic coatings such as paint, varnish, and lacquer. Two methods are described as follows:

1.1.1 In *Test Method A*, the Wet Film Thickness Gage (English or Metric graduation (see 5.1)) is used to measure wet film thicknesses up to 60 mils on the English scale series, and up to 700 μm on the metric scale series (Sections 5 through 8).

1.1.2 In *Test Method B*, the Pfund Gage is used to measure wet film thicknesses up to 14.2 mils (360 μm) (Sections 9 through 13).

1.2 *This standard does not purport to address if any, the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

3. Summary of Test Methods

3.1 The material is applied in the laboratory to plane, rigid test panels or in the field to the surface being coated. The wet film thickness is determined as quickly as possible to reduce shrinkage due to solvent loss.

3.2 In *Test Method A*, a gage with one eccentric and two concentric wheels is rolled over the surface and the point where the eccentric wheel first touches the wet film is determined.

3.3 In *Test Method B*, a gage with a convex lower surface is pushed into the wet film until the center touches the substrate. The diameter of the spot of wet coating left on the convex surface is measured and the film thickness calculated from the diameter of the spot and the radius of curvature.

4. Significance and Use

4.1 Wet film thickness measurements aid in the prediction of dry film thickness. In instances where dry film thickness cannot be measured nondestructively, wet film thickness is frequently specified. Also, the ability to deter-

mine wet film thickness during application can provide the opportunity to correct the application procedures.

TEST METHOD A

5. Apparatus

5.1 *Wet Film Thickness Gage*³—Two versions of the gage are now in use⁴: The original design as shown in Fig. 1 and a later design as shown in Fig. 2. Both designs consists of an eccentric center wheel supported by two concentric wheels so as to provide two scales that are bilaterally symmetrical. As the gage is rolled on the film, there is a change in clearance between the wet film and the eccentric wheel. The point at which the film first touches the center wheel measures the thickness of the film.

5.2 The later design shown in Fig. 2, moves the eccentric wheel from between the concentric wheels, as shown in Fig. 1, to the outside of the gage and closer to one of the concentric wheels. Gage reading errors of parallax across the gage are eliminated as the two scales are placed directly on the eccentric wheel and errors when measuring on uneven support surfaces are reduced by placing the eccentric wheel closer to one of the supporting concentric wheels.

5.3 Best precision is obtained when using the near linear central portion of each scale, constituting about 80 % of its total range as shown in the diagram in Figs. 1 and 2. Therefore, the range of the particular gage selected for use should be such that the measured film thickness falls within, and preferably toward the center of the middle 80 % of each scale. Gages are available covering the following ranges in mils and microns:

Range, mils	Smallest Graduation, mils
0 to 2	0.1
0 to 4	0.2
2 to 12	0.5
10 to 30	1.0
20 to 60	2.0
Range, μm	Smallest Graduation, μm
0 to 40	2.0
0 to 100	5.00
50 to 250	10.00
200 to 700	25.00

³ Available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Ln., Silver Spring, MD 20910 and from the Paul N. Gardner Company, Inc., 316 N.E. First Street, Pompano, FL 33060.

⁴ Both versions of this gage (Interchemical (INMOT) and Model "C"), available from BYK-Gardner, Inc., are covered by U.S. Patents 2 507 592 and 3 128 558, and are held by Maynard R. Euverard, 113 Angell Town Ln., Williamsburg, VA 23185. Interested parties are invited to submit information regarding the identification of acceptable alternatives to this patented item to the Committee on Standards, ASTM Headquarters, 1916 Race St., Philadelphia, 19103. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved May 15, 1991. Published July 1991. Originally published as D 1212 - 52 T. Last previous edition D 1212 - 85.

² Annual Book of ASTM Standards, Vol 06.01.

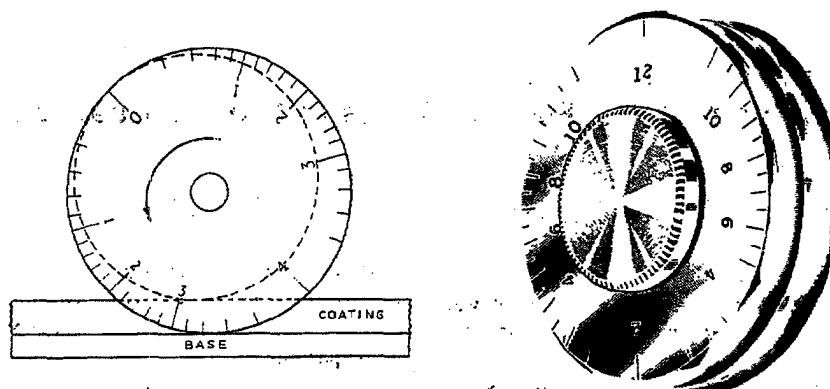


FIG. 1. Interchemical Wet Film Thickness Gages

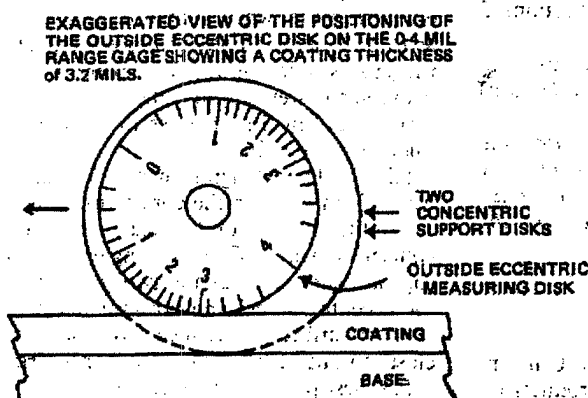


FIG. 2. Model "C" Wet Film Thickness Gages

6. Procedure

6.1 Apply the material in accordance with Test Methods D 823 to suitable plane rigid panels of area sufficiently large to permit film thickness measurements at least 1 in. (25 mm) from any edge. Determine the film thickness immediately after application.

6.2 Support the test panels on a suitable level base in such a way that there will be no movement or spring of the panels during the film thickness measurements. Place the gage on the wet film so that the minimum marking is at the top and the greatest clearance between the eccentric wheel and the film is directly over the wet film. Roll the gage over the film one-half revolution in one direction toward the minimum marking on the gage and repeat in the opposite direction. Read the points at which the coating first makes contact with the eccentric wheel and determine the mean which is considered as one reading.

6.3 If the coating contains a solvent that evaporates rapidly or if the solids content is low, make at least a second separate reading on a freshly applied film and calculate the mean of the separate readings.

7. Report

7.1 Report the mean of the separate readings and the range and smallest graduation of the gage used.

8. Precision⁵

8.1 In an interlaboratory study of this test method, in which two operators in at least six laboratories made measurements with the 0- to 2- and 0- to 4-mil gages on three materials, applied at two film thicknesses, the pooled within-laboratory standard deviation was found to be 0.12 mils with 34 df and the between-laboratories standard deviation 0.18 for two determinations and 0.17 for four determinations with 25 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Results, each the mean of opposite rolls of the 0- to 2- or 0- to 4-mil gage, obtained by the same operator should be considered suspect if they differ by more than 0.4 mil for two determinations and 0.5 mil for four determinations.

8.1.2 *Reproducibility*—Two results, each the mean of two separate determinations, obtained by operators in different laboratories, should be considered suspect if they differ by more than 0.55 mil.

⁵ Supporting data are available from ASTM Headquarters. Request D01-1023.

8.2 The proportion:

9. Apparatus

9.1 *Pfun* convex lens of 250 mm an outer tul surface out applied to t film. The d of diameter D correspo the menisc on the glass

9.2 Steel millimetres.

10. Procedure

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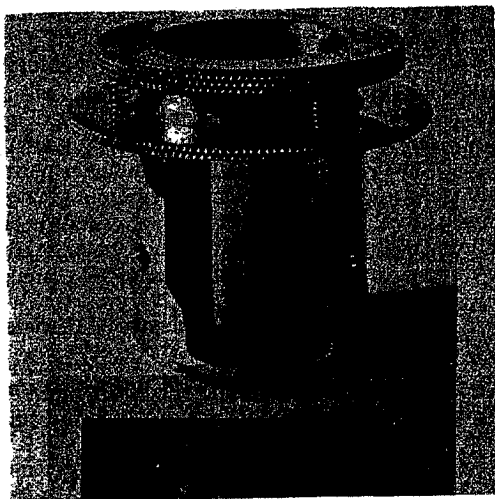
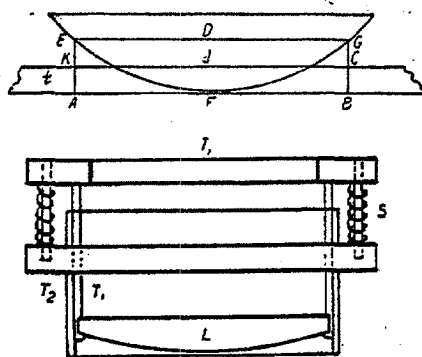


FIG. 3 Pfund Gage

8.2 The precision of higher range gages is expected to be proportional to film thickness.

TEST METHOD B

9. Apparatus

9.1 *Pfund Gage*⁶—The gage⁶ shown in Fig. 3 consists of a convex lens *L* with lower surface having a radius of curvature of 250 mm mounted in a short tube *T*₁, that slides freely in an outer tube *T*₂. The compression springs *S* keep the convex surface out of contact with the wet film until pressure is applied to the top of *T*₁, forcing the lens *L* down through the film. The displaced material forms an oversized circular spot of diameter *D*, on the convex lens (larger than the diameter *D* corresponding to the thickness *T* of the film) because of the meniscus effect due to wetting and surface tension effects on the glass lens.

9.2 *Steel Scale* (furnished with Pfund Gage), calibrated in millimetres.

10. Procedure

10.1 Apply the material to suitable plane, rigid panels of

area sufficiently large to permit film thickness measurements at least 1 in. (25 mm) from any edge. Determine the film thickness immediately after application.

10.2 Support the test panels on a suitable flat level base in such a way that there will be no movement or spring of the panels during the film thickness measurements. As shown in the second diagram in Fig. 3, place the gage on the coated surface and slowly force the inner tube down as far as it will go (point *F* on surface *AB* of the base) allowing displaced material to escape through the notches in the outer tube but preventing lateral motion of the gage. Upon releasing the pressure, an oversized circular spot is retained on the lens. Its diameter *D* is the length of the line *EG* and corresponds to the sum of two thicknesses: *AK* = *BC* = thickness of underlying undisturbed coating; and *KE* = *CG* = thickness of overlying displaced coating.

10.3 Remove the gage from the surface, measure to the nearest 0.5 mm in two directions at approximate right angles the diameter of the oversize spot on the lens using the steel scale, and determine the mean of the two measurements that is considered as one reading. Make at least two separate tests to obtain the grand mean film thickness.

10.4 Although this procedure refers to measurements on flat test panels, the same technique may be used in the field or shop on commercial articles, provided the substrate is not distorted at the point of contact to an extent that would affect the measurements.

10.5 If coarse particles in the wet film prevent the lens from making contact with the base at the point *F*, the results will not be valid.

11. Calculation

11.1 The relations between the diameter of the oversized spot on the lens in millimetres, the approximate thickness of the undisturbed wet film, and the coverage in square feet per gallon are given in a printed table supplied with the Pfund gage. It has been found that empirical equations give approximate values for the wet film thickness, *t*, as follows:

$$t \text{ (in mils)} = (D^2 \times 1000) / 16R \times 25.4 = 0.0943 D^2$$

$$t \text{ (in micrometres)} = (D^2 \times 1000) / 16R = 0.25 D^2$$

where:

D = diameter of spot, mm, and

R = radius of curvature of convex lens (250 mm).

based on the fundamental assumption that *t* is exactly one half of the total thickness of the film at the diameter *D*, as shown in the second diagram in Fig. 3.

11.2 The Pfund gage cannot be calibrated by the manufacturer prior to delivery. For the best results, a correction factor must be established for each type and thickness of material, based on freshly prepared films of known wet film thickness, as measured by Test Method A (Interchemical Wet Film Thickness Gage).

12. Report

12.1 Report the mean of at least two separate readings as the wet film thickness.

13. Precision

13.1 In an interlaboratory study of this method, in which two operators in five laboratories made measurements on three materials applied at two film thicknesses, the pooled

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⁶ Keirath, G., "Sphere Penetration Gage," National Institute of Standards and Technology Circular, NIST, Nat No. 585, Section 1.06, p. 4.

within-laboratory standard deviation was found to be 0.21 mil with 30 df and the between-laboratories standard deviation with 24 df 0.285 mil for two measurements and 0.265 mil for four measurements. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.1 *Repeatability*—Results, which are the mean of the two measurements at right angles, obtained by the same operator should be considered suspect if they differ by more

than 0.6 mil for two determinations and 0.8 mil for four determinations.

13.1.2 *Reproducibility*—Two results, each the mean of two separate determinations, obtained by operators in different laboratories, should be considered suspect if they differ by more than 0.85 mil.

14. Keywords

14.1 eccentric center wheel; glass panel reading surface; metals and metallic materials; paint coated panels; wet film thickness gage; wet film thickness measurement

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes¹

This standard is issued under the fixed designation D 1308; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 6011, 6081 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers determination of the effect of household chemicals on clear and pigmented organic finishes, resulting in any objectionable alteration in the surface, such as discoloration, change in gloss, blistering, softening, swelling, loss of adhesion, or special phenomena.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*
D 609 Methods for Preparation of Steel Panels for Testing Paint Varnish, Lacquer, and Related Products²

3. Summary of Test Method

3.1 Three test methods, each of which is particularly applicable to individual reagents under study, are described as follows:

3.1.1 *Spot Test, Covered*—The reagent is placed on the test surface and immediately covered with a watch glass.

3.1.2 *Spot Test, Open*—The test surface is subjected directly to the effect of substance, such as citrus fruit, oils, greases, beverages, etc.

3.1.3 *Immersion Test*—A suitably prepared panel is immersed in the test reagent.

4. Significance and Use

4.1 Resistance to various liquids used in the home is an important characteristic of organic finishes. These test methods provide the means by which the relative performance of coating systems may be evaluated. It should be recognized that continuous films are necessary for reliable results.

5. Test Panels

5.1 *Steel Panels*—See Method D 609.

5.2 *Other Metal Panels*, as agreed upon by the purchaser and the seller of the finish being tested.

6. Reagents

6.1 The choice of reagent shall be governed by ultimate coating use and by agreement between the purchaser and the seller of the finish being tested. The following reagents are suggested:

6.1.1 *Distilled Water*, cold.

6.1.2 *Distilled Water*, hot.

6.1.3 *Ethyl Alcohol* (50 % volume).

6.1.4 *Vinegar* (3 % acetic acid).

6.1.5 *Alkali Solution*.

6.1.6 *Acid Solution*.

6.1.7 *Soap Solution*.

6.1.8 *Detergent Solution*.

6.1.9 *Lighter Fluid and Other Volatile Reagents*.

6.1.10 *Fruit*—Piece of cut fruit, with cut portion placed face down on panel for time agreed upon between the purchaser and the seller.

6.1.11 *Oils and Fats*—Butter, margarine, lard, shortening, vegetable oils, etc.

6.1.12 *Condiments*—Mustard, catsup.

6.1.13 *Beverages*—Coffee, tea, cocoa.

6.1.14 *Lubricating Oils and Greases*.

6.1.15 *Other Reagents*, as agreed upon between the purchaser and the seller.

7. Procedure

7.1 *Panel Preparation*—Spot and direct application tests may be carried out on the fabricated article coated with the finishing system under evaluation, if sufficient plane surface is available. For immersion tests and tests where the finished article is not available, select panels in accordance with Methods D 609, or prepare special metal panels according to agreement between the purchaser and the seller of the finish. Apply the finish according to the method and the schedule prescribed by the user of the lacquer. This schedule includes number of coats, film thickness, and other features. Allow the finished panels to age 1 week at normal room conditions, about 77°F (25°C) and 50 % relative humidity, before testing.

7.2 *Spot Test, Covered*—Conduct the test at 73.5 $\pm$ 3.5°F (23 $\pm$ 2°C) and 50 $\pm$ 5 % relative humidity, or as agreed upon between the purchaser and the seller. Using a 5-mL pipet graduated in 0.1 mL, pipet onto the horizontal panel 1 mL of the reagents listed in Section 6 and immediately cover with a watch glass. After an interval agreed upon by the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 1308 - 54 T. Last previous edition D 1308 - 79 (1981) ^{ϵ} .

² *Annual Book of ASTM Standards*, Vol 06.01.

purchaser and the seller, wipe the spot clean and examine immediately for effects as listed in Section 1. Frequently used intervals are 15 min, 1 h, and 16 h, or by agreement between the producer and the user. If desired, allow the panel to recover for a specified period, and examine for return of original properties.

7.3 Spot Test, Open—Conduct the test at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, or as agreed upon between the purchaser and the seller. Place a small portion of the reagent on a horizontal panel or surface, with the exception of fruit juices, as mentioned in 5.1.10. After a time interval, as agreed upon between the purchaser and the seller, wipe the spot clean and examine immediately for effects as listed in Section 1. Frequently used intervals are 15 min, 1 h, and 16 h, or by agreement between the producer and the user. If desired, allow the panel to recover for a specified period and examine for return of original properties.

7.4 Immersion—Immerse panels to a depth of 50 % in the specified reagents (6.1.1, 6.1.2, 6.1.5, 6.1.6, 6.1.7, and 6.1.8) contained in beakers, at a temperature and length of time agreed upon between the purchaser and the seller. Withdraw the panels, wash with distilled water, and examine immedi-

ately for any of the effects listed in Section 1. If desired, allow the panels to recover for a specified period and examine for return of the original properties. In general, it will not be necessary to seal the edges of the applied film. If the reagent effect is noted only around the panel edges, the test should be repeated using a suitable edge sealer. When sealing of edges is required, the selection of the sealer should be a matter of agreement between the purchaser and the seller.

8. Report

8.1 Report the following information:

8.1.1 System and the testing method employed. The test conditions are an important factor in the results obtained and, therefore, should be defined in the report.

8.1.2 The type of the effect, if any (see Section 1).

9. Precision and Bias

9.1 This test is designed to provide a working procedure for examination of the effect of household chemicals on clear and pigmented organic finishes. The effects will be in terms of appearance, and numerical values are assigned if a rating scale is used. Precision numbered values are not usually obtained for this type of qualitative test.

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Standard Test Method for Settling Properties of Traffic Paints During Storage¹

This standard is issued under the fixed designation D 1309; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a laboratory procedure for simulating in 2 weeks the settling that might occur in traffic paint during approximately 12 months' normal storage.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Note in 5.2.

2. Referenced Document

2.1 ASTM Standard:

D 869 Test Method for Evaluating Degree of Settling of Paint²

3. Significance and Use

3.1 Paints, if not formulated or processed properly, or both, may settle excessively. This test method is an attempt to simulate the conditions that would accelerate settling of the pigment in order to evaluate settling properties within 2 weeks. The variables of this test method in conjunction with the very subjective method of evaluating the degree of settling (Method D 869) raise questions as to the usefulness of the results for specification compliance.

4. Apparatus

4.1 *Container*—Standard 1-pt (500-mL), friction top can paint container $3\frac{3}{8} \pm \frac{1}{16}$ in. (85.5 $\pm$ 1.5 mm) in diameter and $3\frac{1}{2} \pm \frac{1}{16}$ in. (98.5 $\pm$ 1.5 mm) in height.

4.2 *Spatula*, weighing 45 ± 1 g with square-end blade $4\frac{3}{4}$ in. (125 mm) in length and approximately $1\frac{3}{16}$ in. (20 mm) in width. A suitable spatula may be prepared by cutting the tip from an ordinary 5-in. (125-mm) flexible steel laboratory

spatula to the specified length.

4.3 *Freezer*, maintained at from -5 to -10°F (-21 to -23.3°C).

4.4 *Oven*, maintained at $160 \pm 2^{\circ}\text{F}$ ($71 \pm 1^{\circ}\text{C}$).

4.5 *Lid Clips*.

5. Procedure

5.1 Thoroughly mix the paint to be evaluated to a homogeneous consistency. Transfer enough of the paint to a 1-pt (500-mL) paint can to fill it to within $\frac{1}{2}$ in. (13 mm) of the top. Close the can tightly, securing the lid with four clips, and subject it to the following exposure cycle.

5.1.1 Monday through Friday:

5.1.1.1 Place in freezer at 8:00 a.m.

5.1.1.2 Transfer to oven at 10:00 a.m.

5.1.1.3 Transfer to freezer at 2:00 p.m.

5.1.1.4 Transfer to oven at 4:00 p.m.

5.1.2 *From 4:00 p.m. Friday until 8:00 a.m. Monday:*

5.1.2.1 Place in the oven at 4:00 p.m. Friday.

5.1.2.2 At 8:00 a.m. Monday morning, after the can has been in the oven over a weekend, place it in the freezer and continue the cycles described above for another 7 days. Each time the can of paint is removed from the oven give it one sharp "spank" on the table top. The spank consists of a short rapid jar of the bottom of the can of paint against the top of the table. The violence of the jar is such that if the bottom of the can of paint were spanked against the platform of a scale instead of a table top the scale would register approximately 5 lb (2.27 kg) weight.

5.2 At the completion of the exposure, allow the paint to cool at room temperature for 4 h and determine the degree of settling in accordance with Method D 869.

NOTE: Warning—Open cans slowly and carefully to allow release of residual vapor pressure.

6. Report

6.1 Report the "accelerated settling rating" in accordance with the degree of settling scale given in the Procedure section of Method D 869.

7. Precision and Bias

7.1 A round robin has been completed and precision and bias statements are being generated.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved March 25, 1988. Published May 1988. Originally published as D 1309 - 54 T. Last previous edition D 1309 - 83 ϵ 1.

² Annual Book of ASTM Standards, Vol 06.01.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Fineness of Grind of Printing Inks By the NPIRI Grindometer¹

This standard is issued under the fixed designation D 1316; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes the procedure for determining the fineness of grind of printing inks using a NPIRI grindometer. It evaluates the size of the largest particles in a finished dispersion but not average particle size or concentration of sizes.

1.2 This test method is applicable to any dispersion that is fine enough to fall within the 0–25 μm range of the specified grind gage. With a minor variation in procedure, it is applicable to both paste (nonvolatile) and liquid (volatile) inks.

NOTE 1—The 0–25 μm gage specified in this test method is similar in principle to the 0–100 μm Hegman gage described in Test Method D 1210. Sieve analysis for concentration of particles above 45 μm is covered in Test Method D 185.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 185 Test Method for Coarse Particles in Pigments, Pastes, and Paints²

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems³

E 691 Practice for Conducting an Interlaboratory Test Study to Determine the Precision of Test Methods⁴

3. Descriptions of Terms Specific to This Standard

3.1 *fineness of grind*—a measure of the size and prevalence of oversize particles in a printing ink dispersion.

3.2 *scratch*—a depression at least 10 mm in length in the surface of a grind gage drawdown. A scratch develops when a particle (or agglomerate) is trapped between the blade and the bottom of the path and is drawn along by the blade.

3.3 *speckle*—protuberance of particles above the surface of a grind gage drawdown. Speckles occur at gage depths greater than those at which scratches occur and are caused by

oversize particles that are not hard enough or of the proper size to produce scratches.

4. Summary of Test Method

4.1 This test method utilizes a grind gage having two precision machined grooves each 1 in. (25.4 mm) wide and with a 0–1 mil (0–25 μm) taper. The test specimen is drawn down the paths slowly if a nonvolatile (paste) ink, briskly if a volatile (liquid) ink. The drawdowns are examined for the scale readings at which four and ten scratches appear and at which a preponderance of speckles disappear. The mean of readings from four paths constitutes a single determination.

5. Significance and Use

5.1 Oversize particles in a printing ink may damage a printing plate and adversely affect the appearance of printed ink films. Fineness of grind measurements are useful for deciding when to stop the dispersion process and for determining if the test material meets specifications as agreed upon between the supplier and the customer.

5.2 Speckle endpoints identify the size of the largest particles in a finished printing ink. They provide a better measure of overall dispersion quality than do scratch endpoints but, while their single-operator precision is better, their between-laboratory precision is far poorer.

5.3 Scratch endpoints indicate the size and relative number of the coarsest hard particles in a finished printing ink. They provide a measure of grittiness; the higher the 4-scratch endpoint, the grittier the ink; the smaller the difference between the 4- and 10-scratch endpoints, the larger the number of gritty particles in this range. Scratch endpoints may, however, miss gritty particles that ride in the bank ahead of the scraper; for this reason, they are not recommended for premixes or other dispersions containing a preponderance of gritty particles.

6. Apparatus

6.1 *NPIRI Grindometer*,⁵ consisting of a block of hardened steel⁵ 1 in. (25 mm) in thickness,⁶ 3½ in. (89 mm) in width and 9½ in. (240 mm) in length. The top surface of the block is precision machined so as to contain two paths, each 1 in. in width, 6½ in. (165 mm) in length, and tapered uniformly in depth lengthwise from 1 mil (25 μm) to zero depth. Depth markings are inscribed on the shoulders at each 0.1 mil (2.5 μm) change in taper. Scale inscriptions and the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved Nov. 27, 1987. Published January 1988. Originally issued as D 1316 - 54. Last previous edition D 1316 - 68 (1979).

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.03, 08.03, and 14.02.

⁵ The NPIRI Grindometer, developed by the National Printing Ink Research Institute, may be obtained from the Precision Gage and Tool Co., 28 Volkenand Ave., Dayton, OH 45410.

⁶ Also available is a model 0.918 in. (23.3 mm) in thickness, which may serve as a type-high printing plate in a flat-bed press.

TABLE 1 Relation Among Grind Gage Scales

NOTE—gu—NPIRI grindometer unit.

NPIRI Scale, ^A gu	Depth ^B		Micrometer Scale, ^C μm	Hegman Scale ^D
	mils	μm		
0	0	0	0	8
1	0.1	2.5		
2	0.2	5	5	
3	0.3	7.5		
4	0.4	10	10	
5	0.5	12.5		7
6	0.6	15	15	
7	0.7	17.5		
8	0.8	20	20	
9	0.9	22.5		
10	1.0	25	25	6
15	1.5	38	50	5
20	2.0	50		4
25	2.5	63	75	3
30	3.0	75		2
35	3.5	88	100	1
40	4.0	100		0

^A Numbers above dashed lines are inscribed on the side or on the left shoulder of all standard NPIRI Grindometers. Numbers below dashed lines represent extended scales available on special gages.

^B Nominal depth, not necessarily thickness of material deposited in paths of grind gages. Path fillage is about 80 % for a high viscosity (sheet-fed, offset) ink; 60 % for a low viscosity (letterpress news) ink. Path fillage is also decreased by fast drawdowns.

^C Numbers above dashed lines are inscribed at every fifth calibration line on the right shoulder of newer NPIRI Grindometers.

^D Paint Scale in Test Method D 1210.

relationship among various scales are given in Table 1.

6.2 *Scraper*,⁷ a double edged blade conforming to Test Method D 1210.

6.3 *Ink Knife*, small.

7. Materials

7.1 *Rags or Tissues*, lint- and metal-free.

7.2 *Solvent*, naphtha or other as appropriate for the ink under test.

8. Care of Gage and Blade

8.1 *Cleanliness*—The scraper and the block must be perfectly clean prior to a drawdown. Any dirt or lint present may produce a scratch and give a false reading.

8.2 *Misuse*—The grindometer is a precision instrument and must be treated as such. Do not let any hard materials contact the gage surface or scraper in any manner that might result in scarring or nicking. Avoid tapping or scratching with other metal. Use only soft metal-free cloths for cleanup.

8.3 *Normal Wear*—Under steady usage over an extended period of time, both scraper and block will wear but localized scraper wear is much more rapid than block wear. Periodically check the blade as follows:

8.3.1 *Method 1*—Place a small quantity of an ink across one end of a flat glass plate and make a drawdown. A uniform drawdown indicates that the particular scraper edge is in good condition. A streak of ink or a heavier film of ink at the places corresponding to the shoulders of the

⁷ A second scraper should be procured for use while the first scraper is being reconditioned or is otherwise out of commission.

grindometer is evidence of blade damage or excessive wear. Repeat the drawdown with the other edge of the blade.

8.3.2 *Method 2* (from Test Method D 1210)—Face one edge of the scraper down across the bottom of the top surface of the grindometer or other smooth level surface. Place a strong light behind the scraper and examine the contact edge; any light coming through shows that the blade edge has been damaged and is not satisfactory for use. Wear and warpage can also be detected by rocking the blade back and forth. Repeat the procedure on the other edge of the blade.

8.4 *Rusting*—The grindometer is made of carbon steel and is subject to rusting. Apply a coating of grease or other rust-preventative and keep covered or encased when not in use.

NOTE 2: Caution—Do not use a gage or blade that exhibits damage or wear (see 8.2 to 8.4). Replace or return to the manufacturer for reconditioning.

9. Sampling

9.1 Approximately 2 to 3 mL of ink sample is sufficient to fill four paths of the grindometer. When taking samples either from a container or from a disperser, push aside the top layer and take the sample from below. This technique prevents skin or surface dust from being transferred to the gage.

9.2 If the sample is a liquid ink, hand stir the sample vigorously for 2 min, taking care that air bubbles are not whipped in.

10. Procedure

10.1 Place the gage on a flat, non-slippery surface so that the deep end of the grooves is farthest from the operator. Wipe clean immediately before the test. Be sure the gage surface is dry and free of lint and wipe marks.

10.2 Using the ink knife, transfer a small quantity of ink across the deep end of both grooves about 1/2 in. (12.5 mm) from the end of the block. Alternatively, different inks may be placed in the two respective paths.

10.3 Grasp the scraper in both hands and, holding in a vertical position, place it behind the ink of the gage. Apply adequate pressure to ensure positive contact of the blade with the surface of the gage and draw the ink down the length of the path. Ink remaining on the shoulders is evidence of insufficient pressure.

10.3.1 *Paste Inks*—Make the drawdown with a smooth steady stroke that takes 7 to 10 s to complete.

10.3.2 *Liquid Inks*—Make the drawdown with a brisk stroke that takes 1 to 2 s to complete. When testing fluid inks, it is essential to work quickly, as partial drying of the ink specimen or the ink film can influence test results.

10.4 Set the blade on a sheet of scrap paper, and immediately pick up the gage to read the endpoints. Rotate the gage in a light until the patterns, when viewed from the side, are clearly visible. Record endpoints (see Section 11) within 10 s of completing the drawdown.

10.5 Immediately clean the gage and repeat the procedure in 10.2 through 10.4 until readings have been made on 10 paths per sample. If a reference standard was used in one path, alternate its position on the repeat tests.

TABLE 2 Precision of Grindometer Endpoints for Roll-Milled Dispersions

Endpoints	Standard Deviation, gu	Degrees of Freedom	Maximum Allowable Difference, gu(μm)
A. Repeatability (single operator)			
4-scratch	0.31	36	0.87 (2.2)
10-scratch	0.27	36	0.76 (1.9)
Speckles	0.15	30	0.43 (1.1) ^a
B. Reproducibility (between laboratories)			
4-scratch	0.72	30	2.1 (5.0)
10-scratch	0.70	30	2.0 (5.0)
Speckles	1.97	24	5.6 (14.2) ^a

^a Comparable figures from the precision statement of Test Method D 1210 are 10 μm for repeatability and 25 μm for reproducibility.

11. Reading of Endpoints and Report

11.1 Read the following endpoints to $\frac{1}{2}$ gu (see NPIRI scale in Table 1) or to 1 μm or both.

11.1.1 The first point from the deep end at which four scratches, each at least 10 mm long, occur simultaneously. The scratches need not reach the bottom of the plate groove.

11.1.2 As in 11.1 for ten scratches.

11.1.3 The place at which speckles disappear.

11.2 Record the three types of endpoints on a total of four paths and compute the respective means.

11.3 When reporting endpoints, mean figures may be rounded to $\frac{1}{2}$ gu, or to 1 μm , or both.

12. Precision and Bias^a

12.1 Precision:

12.1.1 An interlaboratory study of this test method was conducted in which one operator in each of seven laboratories made two determinations, each the mean of readings on four drawdowns, on two different days on six carbon black dispersions. The samples represented two different levels of premixing, each followed by one, two, or four passes on a roll mill. The results were analyzed statistically in accordance with Practice E 691. On the basis of the standard deviations shown in Table 2, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1.1 *Repeatability*—Two results, each the mean of readings on four drawdowns, obtained by the same operator should be considered suspect if they differ by more than the scale readings given in the last two columns of Table 2, Section A.

12.1.1.2 *Reproducibility*—Two results, each the mean of readings on four drawdowns, obtained by operators in different laboratories should be considered suspect if they differ by more than the scale readings given in the last two columns of Table 2, Section B.

12.2 *Bias*—Bias cannot be determined as there is no standard material. In the interlaboratory study described in 12.1.1, speckle endpoints were more sensitive to expected differences in degree of dispersion than were scratch endpoints.

^a Supporting data are available from ASTM Headquarters. Request RR: D01-1054.

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Standard Test Method for Fire Retardancy of Paints (Cabinet Method)¹

This standard is issued under the fixed designation D 1360; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method determines quantitatively the fire retardant properties of a coating or coating system on a wood surface and the leaching effect of water on the fire retardancy of the coating or coating system. Specifically, this test method determines the weight loss and char index of coated panels subjected to a flame and the effect of leaching of the coating on these parameters.

1.2 This test method should be used solely to measure and describe the properties of materials, products, or systems in response to heat and flame under controlled laboratory conditions and should not be considered or used for the description, appraisal, or regulation of the fire hazard of materials, products, or systems under actual fire conditions.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 Federal Standard:²

Fed. Spec. TT-V-119—Varnish, Spar, Phenolic Resin

2.2 Military Standard:²

Mil. Spec. MIL-A-22397—Adhesive, Phenol, and Resorcinol Resin Base for Marine Use

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *fire retardancy, n*—in paint, the ability of a paint to retard the spread of a flame over coated substrate usually at the sacrifice of the paint film.

4. Summary of Test Method

4.1 *Fire Retardancy*—Panels are conditioned before and after coating with the material under test. They are subjected to a flame from a small amount of burning alcohol or other solvent and the weight loss and char index are determined.

4.2 *Leaching*—Similarly prepared panels are immersed in water for a specified time and then subjected to the fire-retardancy test. The weight loss, char index, and difference in weight loss and char index between leached and unleached panels are determined.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.22 on Health and Safety.

Current edition approved Oct 26, 1990. Published December 1990. Originally published as D 1360 - 55 T. Last previous edition D 1360 - 90.

² Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, ATTN: NPODS.

5. Significance and Use

5.1 This test method determines the relative fire-retardant properties of coatings and the water leaching of the fire-retardant material from the applied coating. Leaching simulates the effect of high humidity, weathering, and washing on the fire retardancy of a coating, and is specified in various regulations and specifications for fire-retardant paints.

6. Apparatus

6.1 *Cabinet*³—A glass and metal box as shown in Fig. 1.

6.2 *Assembly*,³ consisting of supports for the test panels and solvent cup. The assembly is shown within the cabinet in Fig. 1.

6.3 *Cylindrical Cup*, to hold solvent, made of brass to the following dimensions:

Outside diameter, in. (mm)	1 5/16 (24)
Outside height, in. (mm)	1 1/16 (17)
Wall thickness, in. (mm)	1/32 (1)
Volume, mL	6.0
Operating capacity, mL	5.0

6.4 *Balance*, weighing to 0.1 g.

6.5 *Buret or Pipet*, calibrated in millilitres.

6.6 *Container or Water Bath*, 12.5 by 6.5 by 6.5 in. (320 by 165 by 165 mm), galvanized steel with cover to accommodate 15 panels, or other sizes to accommodate 5 to 10 panels.

6.7 *Oven* capable of maintaining a temperature of $120 \pm 3^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$).

6.8 *Constant Temperature and Humidity Cabinet or Room*, maintained at $50 \pm 5\%$ relative humidity and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$).

7. Test Materials

7.1 *Ignition Fuel*, absolute ethyl alcohol (ethanol) or comparable solvent blend.

NOTE 1—A mixture of 71.4 % reagent grade isopropanol and 28.6 % reagent grade methanol having the same heat of combustion as pure absolute ethanol may be used as the source of ignition.

7.2 Test Panels:

7.2.1 Unless otherwise specified or agreed, yellow poplar heartwood panels 1/4 by 6 by 12 in. (6 by 150 by 305 mm), close-grain, and as nearly edge grain as possible, free from knots and other imperfections, with surfaces planed and sanded. Panels shall be of solid wood or edge glued sections to obtain the width, provided that no strip is less than 1 1/2 in.

³ A suitable cabinet and assembly may be obtained from Custom Scientific Instruments, Inc., 13 Wing Drive, Cedar Knolls, NJ, 07927. Drawings of cabinet, assembly, etc., may be obtained from ASTM Headquarters. Order PCN 12-413-600-14.

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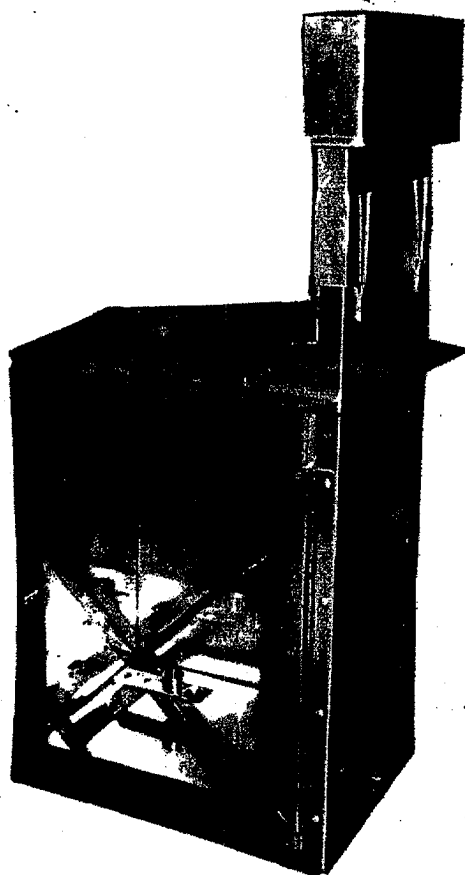
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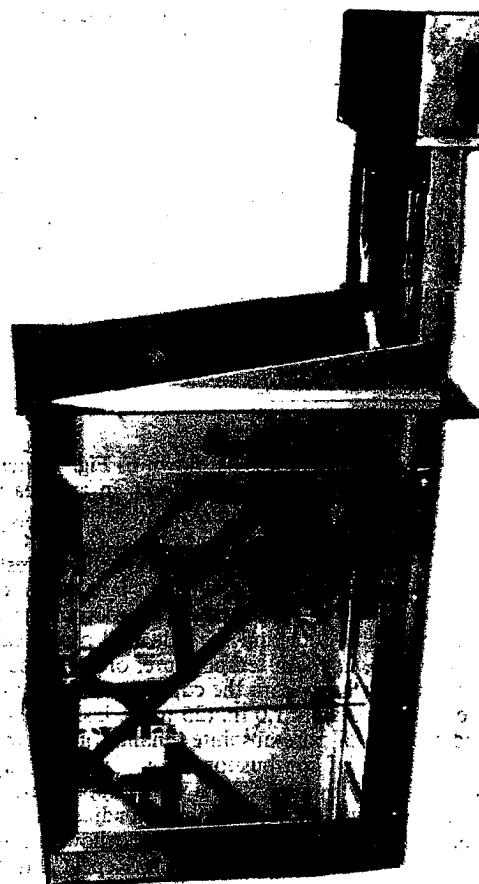
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View A



View B

FIG. 1 Apparatus

(38 mm) wide and that no more than two glue lines occur in any one panel. The adhesive used in glueing these panels shall conform to Mil. Spec. MIL-A-22397. Test panels shall be kiln dried so as to contain no more than 10.0 weight % moisture and after conditioning as specified in 8.1 shall weigh from 27 to 30 lb/ft³ (125 to 140 g/panel).

7.2.2 Douglas fir or other woods shown in Table 1 may be used where required. Table 1 shows the densities and weight per panel for each wood after conditioning.

NOTE 2—Asbestos board (or inorganic reinforced cement board), steel, or concrete may be used upon agreement between the purchaser and the seller.

8. Preparation of Test Panels

8.1 Condition panels for 14 days in a controlled atmosphere of 50 ± 5 % relative humidity and 73.5 ± 3.5°F (23 ± 2°C) prior to coating. Seal the ends of the panels with two coats of varnish conforming to Fed. Spec. TT-V-119 after the conditioning. Allow each coat of varnish to air dry 18 to 24 h.

8.2 Brush apply the material under test in one coat at 250 ft²/gal (6 m²/L) (wet film thickness of 6.4 mils) or in two coats at 500 ft²/gal (12 m²/L) (3.2 mils per coat) to each face

of each panel, obtaining as uniform coverage as possible. Application can be by other conventional means and in as many coats to obtain the necessary weight of coating or desired wet or dry film thickness. Also coat the edges and sides of the panels with the same material. When applied in more than one coat, dry 24 h between coats under standard conditions (see 6.8) and, after the final coat, dry 14 days under the controlled conditions. Prior to subjecting the specimens to the burning test, place them in the oven at 120 ± 3°F (50 ± 2°C) for a period of 40 h.

8.3 Calculate the weight of the coating to be applied using the following equation:

$$W = \frac{(6 \times 12)}{144S} D \times 454$$

where:

W = weight of applied wet coating, g,
 D = coating density, lb/gal, and
 S = spreading rate in ft²/gal.

8.3.1 Use the following equation when metric units are employed:

$$W = (46.4D)/S$$

where:

TABLE 1 Densities and Panel Weights of Various Woods

NOTE—Conditioned at relative humidity $50 \pm 5\%$ and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) for 14 days.

Type of Wood	Density (9 to 10 weight % Moisture), lb/ft ³ (kg/m ³)	Weight of 6 by 12 by 1/4 in. (152 by 305 by 6 mm) Panel, g
Red cedar	21.4–22.4 (345–360)	101–106
Douglas fir	29–35 (465–560)	137–165
White pine–ponderosa pine	22.4–26.4 (360–420)	106–125
Southern yellow pine	31–37 (495–590)	147–175
Redwood	27.2–28.2 (435–450)	129–133

W = weight of applied wet coating, g.
 D = coating density, g/mL, and
 S = spreading rate, m²/L.

9. Procedure—Fire Retardancy

9.1 Assemble the apparatus as shown in Fig. 1, including the stack extension and downdraft hood, in an area where facilities are available for the removal or escape of combustion products (standard laboratory hood without forced ventilation). Weigh the panel to be tested to the nearest 0.1 g and record the weight. Center the panel, face down, on the angular supports with the lower edge 2 in. (50 mm) from the angle formed by the floor and the side wall of the cabinet. Place the fuel cup, at room temperature, on the pedestal so that the vertical distance from the cup lip nearest to the face of the specimen is exactly 1.0 in. (25 mm). Using a buret or pipet, add 5.0 mL of pure absolute ethanol or any mixture with the same heat of combustion (Note 1) to the cup and ignite without delay by means of a flame approximately 1/2 in. (13 mm) in length. Close the door and adjust the draft to assure complete burning of the alcohol. Allow the test to continue until all flames self-extinguish. Repeat this procedure with at least five replicate panels.

9.2 **Weight Loss**—Cool each panel to room temperature and weigh to the nearest 0.1 g. Determine the weight loss in grams of each of the five specimens by subtracting from its original weight (see 9.1). Calculate the mean and standard deviation for the weight loss. If the standard deviation of the five replicates is greater than 10% of the mean, test five additional panels and calculate the mean weight loss of the ten panels.

9.3 **Char Index**—Cut the panels by means of a fine-toothed saw along the lines of maximum length and width of attack. Measure in centimetres the maximum width of charring of the wood panel below the paint film on the lateral cut. Likewise, measure the maximum length of charring found on the longitudinal cut. Measure the maximum depth to which charring has penetrated the wood as evidenced on the longitudinal or lateral cuts. Determine the

char index by multiplying the maximum char length, width, and depth figures. Calculate the mean and standard deviation for the char index. If the standard deviation of the five replicates is greater than 10% of the mean, test five additional panels and calculate the mean char index on the basis of the ten panels.

10. Procedure—Leaching Test

10.1 Prepare, coat, and condition 15 panels of each material under test as described in Section 8.

10.2 At the end of 14 days at 50 % humidity and 73.5°F (23°C), immerse the 15 panels in 2250 ± 30 mL of water (150 mL/panel) (Note 3) for 40 h at $120 \pm 3^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$).

NOTE 3—The water bath is made to contain 15 panels and 2250 mL of water to cover the specimens. If less than 15 coated panels are immersed, add blank panels to make up the 15. Place panels so that their faces do not touch. Cover the bath to prevent excessive evaporation.

10.3 Subject each of the 15 panels to the fire retardant test in accordance with 9.1 and calculate the differences in mean weight loss and mean char index between the leached and the unleached set of specimens as follows:

Δ wt. loss = mean wt. loss of leached specimens – mean wt. loss of unleached specimens.
 Δ char index = mean char index of leached specimens – mean char index of unleached specimens.

11. Report

11.1 Report the following information:

11.1.1 The number of coats and the coverage in ft²/gal (m²/L) of the total numbers of coats for each set of each material under test,

11.1.2 The mean weight loss and the mean char index of the set exposed only to the fire retardancy test, the number of specimens used in calculating the means, and the final standard deviation, and

11.1.3 The difference in mean weight loss and mean char index between the sets of unleached and leached specimens as determined in 10.3.

12. Precision and Bias

12.1 The precision of this test is to be determined.

12.2 **Bias**—The bias of this test method cannot be determined as the value of fire retardancy can be defined only in terms of a test method.

13. Keywords

13.1 cabinet method; char index; fire retardancy; government specification; paint

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Standard Test Method for Abrasion Resistance of Clear Floor Coatings

This test method covers the determination of the abrasion resistance of clear floor finishes such as lacquer, varnish, and related finishes when applied to small test panels.

Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials, this test method was discontinued in 1989.

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Standard Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base¹

This standard is issued under the fixed designation D 1400; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the nondestructive measurement of the dry film thickness of electrically nonconductive coatings applied over a nonferrous metal base using commercially available eddy current instruments.

1.2 This test method is not applicable to coatings that will be readily deformable under the load of the measuring instrument.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting³

3. Summary of Test Method

3.1 Instruments covered by this test method measure coating thickness by the use of eddy currents. The instrument probe must be placed directly on the coating surface to take a reading.

4. Significance and Use

4.1 The instrument probe coil is energized by alternating current which induces eddy currents in the metal substrate. The eddy currents in turn create opposing alternating magnetic fields in the substrate that modify the electrical characteristics of the probe coil. The extent of such changes is dependent upon the distance between the probe and the metal, the distance being shown on the instrument meter as the thickness (mils or micrometers) of the intervening coating.

4.2 The accuracy of the measurements may be influenced when made closer than 1 in. (25 mm) to an edge or 3 in. (75 mm) to another mass of metal.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Paint Films.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 1400 - 56. Last previous edition D 1400 - 81.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

5. Apparatus

5.1 *Eddy Current Thickness Gages*, commercially available, suitable to measure coating thickness accurately.⁴

6. Test Specimens

6.1 When this test method is used in the field, the specimen is the coated structure or article on which the thickness is to be evaluated.

6.2 For laboratory use apply the materials to be tested to panels of the composition and surface conditions on which it is desired to determine the thickness.

NOTE 1—Applicable test panel description and surface preparation methods are given in Practices D 1730.

NOTE 2—Coatings should be applied in accordance with Test Methods D 823, or as agreed upon between the purchaser and the seller.

7. Calibration of Apparatus

7.1¹ Calibrate the instrument in an area free of stray magnetic fields, such as power lines, generators, or welding equipment. There shall be no vibration apparent on the test piece when the instrument is being calibrated.

7.2 Use a bare section of the substrate after the specified surface preparation method has been accomplished. If an uncoated section of the substrate is not available, uncoated test panels of a similar metal type over which the specified preparation has been performed may be used.

7.3 Use nonmagnetic thickness shims for calibration. Most shims are non-precision and therefore, thickness must be verified with a micrometer.

7.4 Select calibration shims in the expected thickness range to be measured. If a coating is expected to be 3 mils (75 μ m) in thickness, calibrate the instrument at 3 mils. Then check the calibration using shims of both a lesser and greater thickness to determine the thickness range over which the instrument will register accurately.

7.5 Follow the manufacturer's instructions for the specific adjustment of the instrument.

8. Procedure

8.1 Use the instrument only after it has been calibrated in accordance with Section 7.

8.2 Assure that the coating is dry prior to use of the instrument.

⁴ Apparatus found to be suitable include the Permascope, Dermatron, Minitest, and Minitecor. Sources for the instruments are available from ASTM, 1916 Race St., Philadelphia, PA 19103.

8.3 Inspect the probe tip and surface to be measured to assure that they are clean, otherwise erroneous readings can result.

8.4 Take readings in areas free of vibration, electrical, or magnetic fields.

8.5 If thickness readings are found outside the range of accuracy determined in 7.4, repeat the calibration procedure in that range. Check the calibration frequently during use to assure that the instrument continues to read properly.

8.6 Take a sufficient number of readings to characterize the sample.

8.6.1 For laboratory measurements a recommended minimum is three for a 3 by 6 in. (75 by 150 mm) panel and more in proportion to size.

8.6.2 For field measurements a recommended minimum is five determinations at random for every 100 ft² (10 m²) of the surface area. Each of the five determinations should be the mean of three separate gage readings within the area of a 1/2 in. (12 mm) diameter circle.

8.7 Take measurements no closer than 1 in. (25 mm) to an edge or 3 in. (75 mm) to another mass of metal. If such measurements are necessary, recheck the calibration in the specific area to determine the effect the edge or mass of metal has on the instrument reading.

8.8 Report the instrument used, serial number, range, and mean of thickness readings found.

9. Precision

9.1 *Precision*—On the basis of an interlaboratory study of this test method in which eddy-current instruments from four manufacturers were used in several laboratories to measure a moderate range of dry-film thickness on the same set of panels, the within-laboratory and between-laboratories coefficients of variation were found to be as shown in Table 1. Based upon these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results obtained by the same operator using instruments from the same category should be considered suspect if they differ by more than the maximum allowable difference values given in Table 1 for the appropriate film thickness.

9.1.2 *Reproducibility*—Two results, each the mean of four measurements, obtained by operators in different laboratories using instruments from the same category should be considered suspect if they differ by more than the maximum allowable difference values given in Table 1 for the appropriate film thickness.

TABLE 1 Precision of Film Thickness Measurements

	Within Laboratory:					
	Coating Thickness					
	0.5 to 1.5 mils			3 to 3.5 mils		
	Degrees of Freedom (DF)	Coefficient of Variation (v_w), %	Maximum Allowable Difference (MAD), %	Degrees of Freedom (DF)	Coefficient of Variation (v_w), %	Maximum Allowable Difference (MAD), %
Instrument:						
Permascope EC	28	3	8.7	14	2	6.0
Minitest FN 250	...	...	...	2	2	
				16	2	
Minitest FN 250	4	6		...	...	
Elcometer 150 FN	38	8		A	A	
Demitron	16	8		A	A	
Pooled	58	7.9	22.3			
	Between Laboratories					
	Coating Thickness					
	0.5 to 1.5 mils			3 to 3.5 mils		
	Degrees of Freedom (DF)	Coefficient of Variation (v_b), %	Maximum Allowable Difference (MAD), %	Degrees of Freedom (DF)	Coefficient of Variation (v_b), %	Maximum Allowable Difference (MAD), %
Permascope EC	8	3		8	5 ^c	13.7 ^c
Elcometer 150 FN	6	5 ^b		4	3	
Pooled	14	3.98	12.1	12	4.44	
Minitest FN 250	A	A		A	A	
Demitron	A	A		A	A	

^a Results are not provided.

^b Between-laboratories precision is usually larger than within-laboratory precision.

^c Percent relative precision is usually lower for thicker films.

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Standard Test Methods for Indentation Hardness of Organic Coatings¹

This standard is issued under the fixed designation D 1474; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace Method 6212 of Federal Test Method Standard No. 141A. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

^{ε1} NOTE—Keywords were added editorially in January 1991.

1. Scope

1.1 These test methods cover the determination of the indentation hardness of organic materials such as dried paint, varnish, and lacquer coatings, when applied to an acceptable plane rigid surface, for example, metal or glass.

1.2 Two methods are covered as follows:

	Sections
Method A—Knoop Indentation Hardness	6 to 12
Method B—Pfund Indentation Hardness	13 to 19

1.3 Method A, which has the greater precision, provides hardness values in terms of Knoop Hardness Number (KHN). Method B provides hardness in terms of Pfund Hardness Number (PHN). Although the hardness value scales of these methods differ, the methods agree in the ranking of coating hardness.

1.4 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry-Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

3. Terminology

3.1 Definitions:

3.1.1 *indentation hardness*—the resistance to penetration by an indenter.

3.1.2 *Knoop indenter*—a pyramidal diamond of prescribed dimensions.

3.1.3 *Pfund indenter*—hemispherical quartz or sapphire indenter of prescribed dimensions.

3.1.4 *Knoop hardness number, KHN*—the indentation hardness determined with a Knoop indenter, and calculated as follows:

$$KHN = L/A_p = L/l^2 C_p$$

where:

L = load applied to the indenter, kg,

l = measured length of long diagonal of the indentation, mm,

C_p = indenter constant relating l^2 to A_p , and

A_p = projected area of indentation, mm².

3.1.5 *Pfund hardness number, PHN*—the indentation hardness determined with a Pfund indenter, and calculated as follows:

$$PHN = L/A = 4L/\pi d^2 = 1.27 (L/d^2)$$

where:

L = load/kg applied to the indenter, kg,

A = area of projected indentation, mm², and

d = diameter of projected indentation, mm.

4. Significance and Use

4.1 Indentation hardness measurements have proven to be useful in rating coatings on rigid substrates for their resistance to mechanical abuse, such as that produced by blows, gouging, and scratching. These measurements do not necessarily characterize the resistance to mechanical abuse of coatings that are required to remain intact when deformed.

5. Test Specimens

5.1 The substrate for the coating shall be an acceptable plane rigid surface such as glass or metal.

5.2 The coating thickness on any one panel shall be uniform within 0.1 mil (3 μm). Coatings to be compared shall be of equal thickness within 0.2 mil (5 μm). For maximum accuracy, the minimum permissible coating thickness shall be such that the depth of indentation does not exceed three fourths of the coating thickness, to minimize the effect of the substrate.

5.3 At least three replicate specimens shall be tested for each coating to be evaluated.

5.4 Coatings should be applied in accordance with Test Methods D 823 and their dry film thickness should be

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Sept. 27, 1985. Published November 1985. Originally published as D 1474 - 57 T. Last previous edition D 1474 - 68 (1979)^{ε1}.

² Annual Book of ASTM Standards, Vol 06.01.

measured in accordance with Test Methods D 1005, D 1186, or D 1400.

METHOD A—KNOOP INDENTATION HARDNESS

6. Summary of Method

6.1 This method consists of applying a load to the surface of a coating by means of a pyramidal shaped diamond having specified face angles, and converting the measurements of the resultant permanent impression to a hardness number.

7. Apparatus

7.1 *Hardness Tester*,³ consisting of a load applicator, a Knoop indenter, and a microscope fitted with a movable micrometer stage. The apparatus shall mechanically bring the indenter into contact with the test surface with negligible impact, apply the selected full load, maintain it for 18 ± 0.5 s, and withdraw the indenter.

7.2 *Knoop Indenter*—The Knoop indenter is a pyramidal diamond with included longitudinal angles of $172^\circ 30'$ and included transverse angle of $130^\circ 0'$.

NOTE 1—The ratio of the long to the short diagonal of the impression is approximately 7:1; the ratio of the long diagonal to the depth of penetration is approximately 30:1.

7.3 *Microscope*—The microscope shall have a filar micrometer eyepiece and sufficient objectives to permit the measurement of the length of impression to within $\pm 1\%$. The specimen shall be firmly supported on a movable micrometer stage attached to the microscope.

8. Calibration

8.1 Adjust the illumination in the microscope to give maximum contrast when viewing an indentation.

8.2 By means of a calibrated scale, determine the factor for each microscope objective that will convert the filar scale units of the eyepiece to millimetres.

8.3 With a 25-g load on the indenter, determine the KHN of a calibrated standard (Note 2) whose assigned value is not greater than 50 KHN. If the obtained value is within $\pm 5\%$ of the assigned value, the instrument is considered to be in calibration.

NOTE 2—A suitable source of calibrated standards in this hardness range is not available. Therefore, by agreement of the parties concerned, a stable specimen (such as an aged coating of a baked enamel applied to a flat substrate) should be used to calibrate the participating hardness testers.

9. Procedure

9.1 Unless otherwise specified, make the hardness determinations at $73.5 \pm 3.50^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity after equilibrating the specimens under these conditions for at least 24 h.

9.2 Rigidly attach the specimen to the movable stage so that the surface to be measured is normal to the direction of indentation.

³ Among the hardness testers meeting the apparatus requirements for this method are the Tukon Micro-hardness Tester and the Riehle Kentron Micro-hardness Tester, available from the Page-Wilson Measurement System Div., 929 Connecticut Ave., Box 9021, Bridgeport, CT 06602.

NOTE 3—The panel should be so mounted that it cannot move with respect to the stage in any direction during the course of the test.

9.3 Use the microscope to select an area of the test specimen that is free of surface irregularities and imperfections. Place this area under the indenter by means of the movable micrometer stage.

NOTE 4—If good impressions cannot be obtained because of the roughness of the surface of the specimen, gently polish the surface with No. 400 carborundum and finish off with jewelers rouge before making the impression.

9.4 Preset the apparatus to apply a 25-g load and perform the test according to the manufacturer's instructions. Maintain the time the indenter is in contact with the specimen for 18 ± 0.5 s.

NOTE 5—For maximum accuracy, care must be taken that the indenter does not penetrate the coating to a depth beyond three fourths of the coating thickness. This is necessary to eliminate any major substrate effect on the hardness measurement.

9.5 Immediately after the completion of the cycle, adjust the movable stage so that the indentation is in the field of the microscope. Focus the microscope on the indentation so that both extremities of the long diagonal (that is, where the upper edges of the indentation just converge) are as sharp as possible. Measure the length of the long diagonal of the impression with the filar micrometer eyepiece.

NOTE 6—Select a microscope objective that will cause the length of impression to be between 200 and 800 filar units to assure maximum accuracy in measurement.

9.6 From the measurements obtained in 9.5, the information given in Note 1, and the measured film thickness at the place of indentation, calculate the depth of indenter penetration. If the depth of penetration exceeds three-fourths of the coating thickness, the results may be influenced by substrate proximity. Therefore, for maximum accuracy it is desirable to prepare thicker specimens and repeat the test. Instead of this procedure, at the option of purchaser and seller, an indenter load of less than 25 g may be used.

9.7 Repeat the procedure described in 9.3, 9.4, and 9.5 until at least five impressions have been made at widely spaced locations on the specimen.

10. Calculation

10.1 Calculate the mean indentation length in filar units.

10.2 Convert this mean indentation length to KHN by means of the appropriate tables supplied with the instrument.

NOTE 7—If a conversion table is not available, the KHN may be calculated as follows:

$$\text{KHN} = 0.025/l^2 C_p$$

where:

0.025 = load applied, kg, to the indenter,

l = length of long diagonal of indentation, mm, and

C_p = indenter constant = 7.028×10^{-2} .

11. Report

11.1 Report the following information:

11.1.1 Mean and range of KHN values obtained for each specimen, stating the number of indentations made and the indenter load used;

- 11.1.2 Mean film thickness of each specimen, based on the measurements made at the points of indentation,
- 11.1.3 Specimen preparation and conditioning techniques used, and
- 11.1.4 Mean and range of KHN values of the replicate panels.

12. Precision

12.1 On the basis of an interlaboratory test of this method in which operators in six laboratories tested seven coated panels having a broad range of hardness, the within-laboratory coefficient of variation was found to be 3 % with 21 degrees of freedom and the between-laboratories coefficient of variation 8 % with 30 degrees of freedom. Based upon these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of three determinations on a specimen, obtained by the same operator should be considered suspect if they differ by more than 9 % of their mean value.

12.1.2 *Reproducibility*—Two results, each the mean of three determinations on a specimen, obtained by operators in different laboratories should be considered suspect if they differ by more than 24 % of their mean value.

METHOD B—PFUND INDENTATION HARDNESS

13. Summary of Method

13.1 This method consists of applying a load to the surface of a coating, by means of a transparent colorless quartz or synthetic sapphire hemisphere having a specified diameter, and converting the measurement of the resultant observed impression under load to a hardness number.

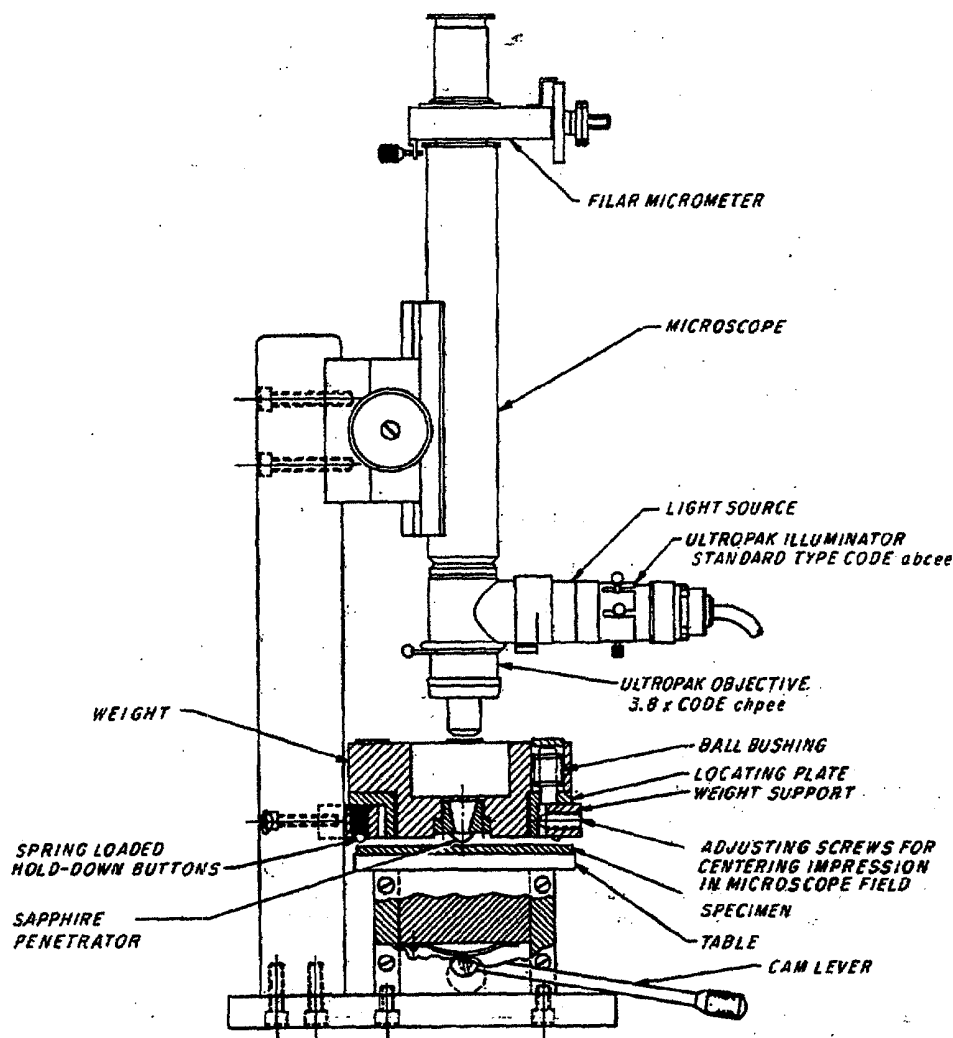


FIG. 1 A Pfund Hardness Tester (Shown Before Application of Weight)

14. Apparatus

- 14.1 Hardness method shall be sapphire in apparatus shall be brought with negligible
- 14.2 Pfund transparent sphere with maximum
- 14.3 Microscope measurement The specimen stage.
- 14.4 The 0.5 s.

15. Standards

- 15.1 Add maximum
- 15.2 By for each method units of thickness
- 15.3 With PHN of a coating not greater than $\pm 5\%$ of the observed to be

16. Procedure

- 16.1 Urmination : humidity, for not less than 16.2 Ki such that direction
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- 16.4 A measure t eyepiece c the diam the inden controlled

⁴ A hardness Pfund Indent: 1415 Park Av

14. Apparatus

14.1 *Hardness Testers*—The hardness testers used in this method shall consist of a load applicator, a Pfund quartz or sapphire indenter, and a microscope fitted with a stage. The apparatus shall be constructed so as to permit the indenter to be brought manually into contact with the specimen surface with negligible impact.

14.2 *Pfund Indenter*^{*}—The Pfund indenter (Fig. 1) is a transparent colorless quartz or synthetic sapphire hemisphere with a spherical radius of 0.125 in. (3.18 mm) and a maximum spherical eccentricity of 0.002 in. (0.05 mm).

14.3 *Microscope*—The microscope shall have a filar micrometer eyepiece and sufficient objectives to permit the measurement of the diameter of impression to within $\pm 1\%$. The specimen shall be rigidly supported on the microscope stage.

14.4 *Timer*, capable of measuring a time interval of 60 ± 0.5 s.

15. Standardization

15.1 Adjust the illumination in the microscope to give maximum contrast when viewing an indentation.

15.2 By means of a calibrated scale, determine the factor for each microscope objective that will convert the filar scale units of the eyepiece to millimetres.

15.3 With a 1.0-kg load on the indenter, determine the PHN of a calibrated standard (Note 2) with an assigned value not greater than 40 PHN. If the value obtained is within $\pm 5\%$ of the assigned value, the instrument shall be considered to be in calibration.

16. Procedure

16.1 Unless otherwise specified, make the hardness determination at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, after holding the specimens under these conditions for not less than 24 h.

16.2 Rigidly attach the specimens to the instrument stage such that the surface to be measured is normal to the direction of indentation (Note 3).

16.3 With a 1.0-kg load on the indenter, carefully bring the indenter into contact with the specimen surface, apply the full load, and start the timer.

16.4 At the end of 60 s, while still under full load, rapidly measure the diameter of the circular impression with the filar eyepiece of the microscope (Note 6). It is very important that the diameter measurements be made rapidly so that the time the indenter is in contact with the specimen is closely controlled.

^{*}A hardness tester meeting the apparatus requirements of this method is the Pfund Indentation Hardness Tester, available from the United States Testing Co., 1415 Park Ave., Hoboken, NJ 07030.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

16.5 From the measurements obtained in 16.4, the information given in 14.2, and the measured film thickness at the place of indentation, calculate the depth of indenter penetration. If the depth of penetration exceeds three fourths of the coating thickness, the results may be influenced by substrate proximity. Therefore, for maximum accuracy it may be desirable to prepare thicker specimens and repeat the test.

16.6 Repeat the procedure in 16.3 and 16.4 until a total of at least five impressions have been made at widely spaced locations on the specimen.

17. Calculation

17.1 Calculate the mean indentation diameter in filar units.

17.2 Convert this mean indentation diameter to millimetres using the appropriate factor determined in 15.2.

17.3 Calculate the PHN as follows:

$$\text{PHN} = 1.27/d^2$$

where:

d = mean indentation diameter, mm.

18. Report

18.1 Report the following information:

18.1.1 Mean and range of PHN values obtained for each specimen, stating the number of indentations made,

18.1.2 Mean film thickness of the specimen, based on measurements made near the points of indentation,

18.1.3 Specimen preparation and conditioning techniques used, and

18.1.4 Mean and range of PHN values of the replicate panels.

19. Precision

19.1 On the basis of an interlaboratory test of this method in which operators in four laboratories tested seven coated panels having a broad range of hardness, the within-laboratory coefficient of variation was found to be 6 % with 28 degrees of freedom and the between-laboratories coefficient 12 % with 18 degrees of freedom. Based upon these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

19.1.1 *Repeatability*—Two results, each the mean of three determinations on a specimen, obtained by the same operator should be considered suspect if they differ by more than 18 % of their mean value.

19.1.2 *Reproducibility*—Two results, each the mean of three determinations on a specimen, obtained by operators in different laboratories should be considered suspect if they differ by more than 36 % of their mean value.

20. Keywords

20.1 hardness (indentation); hardness (Pfund); Knoop hardness tester; Pfund hardness tester



Standard Test Method For Density of Paint, Varnish, Lacquer, and Related Products¹

This standard is issued under the fixed designation D 1475; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense to replace Method 4184.1 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers the measurement of density of paints, varnishes, lacquers, and components thereof, other than pigments, when in fluid form.

1.2 For higher precision when working with nonpigmented materials (drying oils, varnishes, resins and related materials), Test Method D 1963 can be used to determine specific gravity and, thence, density.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

- D 1963 Test Method for Specific Gravity of Drying Oils, Varnishes, Resins and Related Materials at 25/25°C²
- D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter³
- E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)⁴

3. Terminology

3.1 Definitions:

3.1.1 *density*—the mass (weight in vacuum) of a unit volume of the liquid at any given temperature. In this method, it is expressed as the weight in grams per millilitre, or as the weight in pounds avoirdupois of one U. S. gallon, of the liquid at the specified temperature; in the absence of other temperature specification, 25°C is assumed.

3.1.2 *specific gravity*—the ratio of the mass of a unit volume of a material at a stated temperature to the mass of the same volume of distilled water at the same temperature.

4. Summary of Test Method

4.1 The accurately known absolute density of distilled water at various temperatures (Table 1) is used to calibrate the volume of a container. The weight of the paint liquid contents of the same container at the standard temperature

(25°C) or at an agreed-upon temperature is then determined and density of the contents calculated in terms of grams per millilitre, or pounds per gallon at the specified temperature.

5. Significance and Use

5.1 Density is weight per unit volume. It is a key property in the identification, characterization, and quality control of a wide range of materials. Density measurements in terms of weight per-gallon are commonly used to check paint quality. If the density is not within specification, there is a good chance that there was a mischarge or other serious problem.

5.2 This test method is suitable for the determination of density of paint and related products and components when in liquid form. It is particularly applicable when the fluid has too high a viscosity or when a component is too volatile for a density balance determination.

5.3 This test method provides for the maximum accuracy required for hiding power determinations. It is equally suitable for work in which less accuracy is required, by ignoring the directions for recalibration and consideration of temperature differentials, and using the container as a "weight-per-gallon" cup.

5.4 Automatic equipment for measuring density is available (see Test Method D 4052) from several manufacturers. Such apparatus has been used for resins and latices as well as for oils and solvents. Before such equipment is used for a given product, results must be checked very carefully. Particularly with paints and resins, there are possibilities of gumming, fouling, and other interferences with operation.

6. Interferences

6.1 Highly viscous materials may entrap air and give erroneous low density values.

6.2 Paint liquids may be trapped in the ground glass of metal joints of the pieces of apparatus and give erroneous high density values.

7. Apparatus

7.1 *Pycnometer*—Any type, or weight-per-gallon cup having a capacity of from 20 to 100 mL may be used, provided that it may be filled readily with a viscous liquid, adjusted to exact volume, and covered to exclude loss of volatile matter.

7.2 *Thermometers*, graduated in 0.1°C, such as are supplied with glass pycnometers.

7.3 *Constant-Temperature Bath*, held at $25 \pm 0.1^\circ\text{C}$ is desirable.

7.4 *Laboratory Analytical Balance*.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as D 1475 - 57 T. Last previous edition D 1475 - 85.

² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 05.03.

⁴ Annual Book of ASTM Standards, Vol 14.02.

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TABLE 1 Absolute Density of Water, g/mL

°C	Density
15	0.999127
16	0.998971
17	0.998772
18	0.998623
19	0.998433
20	0.998231
21	0.998020
22	0.997798
23	0.997566
24	0.997324
25	0.997072
26	0.996811
27	0.996540
28	0.996260
29	0.995972
30	0.995684

NOTE 1—The usual weight-per-gallon cup and similar specialized pycnometers may have filled weights that exceed the capacity of the usual laboratory analytical balance. In such cases, use of a hanging pan, triple-beam balance, with scales graduated to 0.01 g has been found to provide results the mean of which was consistent with the overall precision and accuracy of the method.

7.5 Desiccator and Desiccated Balance, or a room of reasonably constant temperature and humidity are desirable.

8. Calibration of Pycnometer or Cup

8.1 Determine the volume of the container at the specified temperature by employing the following steps:

8.1.1 Clean and dry the container and bring it to constant weight. Chromic acid (see 8.1.1.1) or other effective glass cleaner and nonresidual solvents may be used with glass containers and solvents with metal containers. For maximum accuracy, continue rinsing, drying, and weighing until the difference between two successive weighings does not exceed 0.001 % of the weight of the container. Fingerprints on the container will change the weight and must be avoided. Record the weight, M , in grams.

8.1.1.1 Precaution—Chromic acid cleaning solution is corrosive to skin, eyes and mucous membranes and can cause severe burns. Avoid contact with eyes, skin or clothing. In making dilute solution, always add acid to water with care. In case of contact, flush skin with water, using a shower if exposure is severe. Flush eyes for 15 minutes with copious amounts of water. Immediately call a physician. Remove clothing immediately and wash before reuse. Chromic acid cleaning solution is a strong oxidizer. Avoid contact with organic or reducing substances as a fire could result. See supplier's Material Safety Data Sheet for further information. Other cleaners are much safer and may be equally effective.

8.1.2 Fill the container with freshly boiled distilled water at a temperature somewhat below that specified. Cap the container, leaving the overflow orifice open. Immediately remove excess overflowed water or water held in depressions by wiping dry with absorbent material. Avoid occluding air bubbles in the container.

8.1.3 Bring the container and contents to the specified temperature using the constant-temperature bath or room if

necessary. This will cause further slight flow of water from the overflow orifice due to the expansion of the water with the rise of the temperature.

8.1.4 Remove the excess overflow by wiping carefully with absorbent material, avoiding wicking of water out of orifice, and immediately cap the overflow tube where such has been provided. Dry the outside of the container, if necessary, by wiping with absorbent material. Do not remove overflow that occurs subsequent to the first wiping after attainment of the desired temperature (Note 2). Immediately weigh the filled container to the nearest 0.001 % of its weight (Note 3). Record this weight, N , in grams.

NOTE 2—Handling the container with bare hands will increase the temperature and cause more overflow from the overflow orifice, and will also leave fingerprints; hence, handling only with tongs and with hands protected by clean, dry, absorbent material is recommended.

NOTE 3—Immediate and rapid weighing of the filled container is recommended here to minimize loss of weight due to evaporation of the water through orifices, and from overflow subsequent to the first wiping after attainment of temperature where this overflow is not retained by a cap.

8.1.5 Calculate the container volume as follows:

$$V = (N - M) / \rho$$

where:

V = volume of container, mL,

N = weight of container and water, g (7.1.4),

M = weight of dry container, g (7.1.1), and

ρ = absolute density of water at specified temperature, g/mL (see Table 1).

8.1.6 Obtain the mean of at least three determinations.

9. Procedure

9.1 Repeat the steps in Section 8, substituting the sample for the distilled water and a suitable nonresidual solvent for the acetone or alcohol (see 8.1.2 and Note 4). Record the weight of the filled container, W , and the weight of the empty container, w , in grams.

NOTE 4—Trapping of paint liquids in ground glass or metal joints is likely to result in high values of density that appear to increase with the viscosity and density of the material; such errors should be minimized by firm seating of the joints.

NOTE 5—Trapping of air bubbles will result in low values for density. The tendency to trap air increases with increasing viscosity. Specimens should not be tested if they contain bubbles or foam. Slow stirring, standing, or the application of a vacuum may remove bubbles. If these do not work, a dilution may be necessary (see Appendix X1).

9.2 Calculate the density in grams per millilitre as follows:

$$D_m = (W - w) / V$$

where:

D_m = density, g/mL.

9.3 Calculate the density in pounds per gallon as follows:

$$D = (W - w) K / V$$

where:

D = density, lb/gal,

K = 8.3454 (Note 6), and

V = volume of container, mL (see 8.1.6).

NOTE 6—The factor K , 8.3454, is calculated from volume-weight relationship as follows:

$$8.345404 = [(2.54)^3 \times (231.00)] / (453.59237)^c$$

^a (2.54)³ is the conversion factor for millilitres to cubic inches.

^b 231.00 is the conversion factor for cubic inches to gallons.

^c 453.59237 is the conversion factor for grams to pounds.

10. Report

10.1 In reporting the density, state the test temperature to the nearest 0.1°C, the units, and the value calculated to the third place to the right of the decimal point (for example, $D = x.xxx$ lb/gal at 25°C); state the mean, the range, and the number of replicate determinations.

11. Precision and Bias

11.1 The precision estimates are based on an interlaboratory study in which one operator in each of six different laboratories analyzed in duplicate on two different days five samples of paint ranging in density from 8.5 to 12.5 lbs/gal.

The results were analyzed statistically in accordance with Practice E 180. The within-laboratory coefficient of variation was found to be 0.20 % relative with 25 degrees of freedom and the between-laboratory coefficient of variation was 0.61 % relative with 20 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.6 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 1.8 % relative.

11.2 No data on bias has been generated for this test method.

12. Keywords

12.1 density; pycnometer; weight per gallon; weight per gallon cup

APPENDIX

(Nonmandatory Information)

X1. Diluting of a Material to Improve Air Release

X1.1 To reduce viscosity and improve air release, a known weight of a material that traps air may be diluted with a known weight of a solvent or another diluent of known density. After careful blending to achieve homogeneity and release air, the density of the diluted material is measured by the technique described in this test method. The following equation may be used to calculate the density of the original material:

$$D_o = \frac{W_o}{\frac{W_o + W_d}{D_{d1}} - \frac{W_d}{D_{d2}}}$$

where:

D_o = density of original material,

D_{d1} = density diluted measured in test,

D_{d2} = density diluent,

W_o = original weight, and

W_d = diluent weight.

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Standard Practice for Effect of Chemical Agents on Organic Finishes Used in the Transportation Industry¹

This standard is issued under the fixed designation D 1540; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1}NOTE—Paragraph 1.2 was changed editorially and footnote 4 added editorially in May 1987.

1. Scope

1.1 This practice covers determination of the effect of commonly encountered chemical agents on applied organic finishes used in the transportation industry.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Note 3.*

NOTE 1—This practice is a companion standard to Test Method D 1308.

2. Referenced Documents

2.1 ASTM Standards:

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 925 Test Methods for Rubber Property—Staining of Surfaces (Contact, Migration, and Diffusion)³
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes²

3. Summary of Test Method

3.1 The test surface is subjected to test by contact, as described in 7.2, 7.3, or 7.4, with an agent suspected of having a deleterious effect, under conditions appropriate to that agent.

4. Significance and Use

4.1 This practice is designed to provide a working procedure for the examination of the effect of chemical agents on applied transportation finishes. Any change in surface appearance or condition reflects an objectionable tendency of the finish to be affected by the agent used. The change is manifested by softening, swelling, discoloration, loss of gloss, blistering, loss of adhesion, or other special phenomena.

5. Test Panels

5.1 *Steel Panels*—Steel panels as described in Methods D 609.

5.2 Other test surfaces may be agreed upon between the purchaser and the seller of the finish being tested.

6. Chemical Agents

6.1 The choice of the agent shall be governed by ultimate coating use and by agreement between the purchaser and the seller of the finish being tested. The following staining agents are listed as being of general interest.

- 6.1.1 Glycol based antifreeze up to 100 volume, %.
- 6.1.2 Acid, alkali, and salt solutions,
- 6.1.3 Soap and synthetic detergent solutions,
- 6.1.4 Lubricating oils and greases,
- 6.1.5 Polish abrasives, creams, waxes,
- 6.1.6 Road oils and tars; seam sealers,
- 6.1.7 Rubber or other elastomers; plastics, tapes,
- 6.1.8 Gasolines,
- 6.1.9 Water,
- 6.1.10 Hydraulic fluids,
- 6.1.11 Alcohol windshield washing compounds, and
- 6.1.12 Others.

7. Procedure

7.1 *Panel Preparation*—Carry out spot and direct application tests on the fabricated article coated with the finishing system under evaluation, if sufficient plane surface is available. Otherwise, select panels in accordance with 5.1. Apply the finishing system in accordance with the method and schedule prescribed by the purchaser. Age the finished film to suit the exposure conditions, as agreed between the purchaser and the seller of the film-forming material.

7.2 *Spot Test for Liquid Agents*—Conduct the test at $73 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity. Place a small portion of the staining agent on a horizontal test surface, making several spots approximately 1 in. (25 mm) in diameter. After the time agreed upon has elapsed, such as 4, 8, 24, and 72 h, wipe off a spot, and examine it immediately for visual and physical evidence of the effects listed in Section 4. Polish the test surface lightly with any suitable liquid car cleaner, and evaluate the surface in accordance with agreement on the basis of removability. Expose the test surface to sunlight for at least 8 h, or to strong ultraviolet light (Note 2) at 150°F (65°C) or less as agreed upon, for 1 h. Evaluate the surface on the basis of either disappearance of defects or development of defects not previously visible.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Tests for Protective Coatings.

Current edition approved Sept. 1, 1982. Published November 1982. Originally published as D 1540 - 58 T. Last previous edition D 1540 - 61 (1973).

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 09.01.

Prepare surfaces for water spotting tests by exposing them to an RS ultraviolet lamp for 6 h, and then polishing. Then proceed as follows: apply water drops, heat in an oven at 150°F for 15 min, and then lightly polish. Any variations in the procedure shall be as agreed upon between the purchaser and the seller of the film-forming material.

NOTE 2—The RS sunlamp manufactured by the General Electric Co. is recommended for this test. Other ultraviolet light sources are acceptable if agreed upon by the purchaser and seller, and if the appropriate exposure equivalent is known and used.

7.3 *Contact Test for Solids*—The materials listed in 6.1.7 may produce a deterioration of the appearance in several of the ways listed in Section 4, but must be held in close contact with the surface. Frequently the agent may cause an effect only when heated, therefore, they should be heated for this test. Test finishes with rubber as a staining agent, in accordance with Test Methods D 925. Plastics may be held in contact with the finish, and tested as described in 7.2.

7.4 *Gasoline* (Caution: Note 3)—To determine the staining effect of gasoline, place some in a buret. Allow the gasoline to fall 4 in. (100 mm) onto the test surface placed below and at an angle of 20° from the horizontal. The dropping rate should be about 10 drops per minute. Place an

RS sunlamp (Note 2) 15 in. (380 mm) from the test surface so that its rays shine at 90° to the surface. Continue the test until 200 mL of gasoline have been dropped during 3½ h.

NOTE 3: Caution—This test should be run where there is adequate ventilation, and provision should be made for draining the drippings into a safety can. Other precautions against fire should be observed.

7.5 In evaluating the results, the 0 to 10 scale prescribed in ASTM STP 500⁴ should be used.

8. Report

8.1 Report the following information:

8.1.1 The system and the test method employed. Test conditions are an important factor in the results obtained, and therefore, should be defined in the report.

8.1.2 The type and appearance of the effect obtained, if any, (see Section 4). It is customary to record stain-free contact time as a basis of comparison. Where agreed, the effect of the agent may also be evaluated by the use of a color difference meter spectrophotometer, glossmeter, hardness instrument, or other applicable apparatus.

⁴ Paint Testing Manual, ASTM STP 500, ASTM, 1972.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Qualitative Detection of Rosin in Varnishes¹

This standard is issued under the fixed designation D 1542; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 5031, 5032 of Federal Test Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

¹ NOTE—Editorial changes were made throughout, including the title, in May 1988.

1. Scope

1.1 This test method covers procedures for the qualitative detection of rosin in varnishes by the Lieberman-Storch Test, and the Halphen-Hicks Test. The rosin may be present as either free rosin (abietic acid), esterified rosin, or as metal salts.

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Apparatus

- 2.1 Test Tubes, 2.5 by 15 cm.
- 2.2 Filter Funnel, 75 mm in diameter.
- 2.3 Porcelain Spot Plate.

3. Reagents

- 3.1 Acetic Anhydride.
- 3.2 Bromine Reagent—Dissolve 1 part by volume of bromine in 4 parts by volume of carbon tetrachloride (CCl_4).
- 3.3 Phenol Reagent—Dissolve 1 part by volume of phenol in 2 parts by volume of carbon tetrachloride (CCl_4).

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.33 on Varnish and Resins, Including Shellac.

Current edition approved Sept. 19, 1960. Published November 1960. Originally published as D 1542 - 58 T. Last previous edition D 1542 - 58.

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3.4 Sulfuric Acid Reagent—Add 35.7 mL of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) slowly to 34.7 mL of water, and cool to room temperature. Store in a glass-stoppered bottle.

4. Procedure

4.1 Lieberman-Storch Test—Place 0.1 to 0.2 g of the sample in a test tube, and add 15 mL of acetic anhydride. Heat gently until the specimen is dissolved or dispersed. Cool and filter with an ashless rapid filter paper, into a clean test tube. Place a few drops of the clear solution in a depression of the spot plate, and add one drop of sulfuric acid reagent, so that the acid will mix slowly with the filtrate. If rosin is present, a fugitive violet color develops immediately. A pink or brown coloration should be ignored. A control specimen containing rosin should be run simultaneously.

4.2 Halphen-Hicks Test—Dissolve a small quantity of the sample in 1 to 2 mL of the phenol reagent. Pour the solution into a cavity of the spot plate until it just fills the depression. A portion of the solution will spread out on the flat part of the plate a short distance beyond the rim of the cavity, unless too much of the carbon tetrachloride has been lost by evaporation, when a drop or two more should be added to produce the spreading effect referred to. Immediately in an adjacent cavity, place 1 mL or more of the bromine reagent, so that the bromine vapors evolved will contact the surface of the solution in the other cavity. Sometimes it is necessary to blow a gentle current of air in the proper direction to accomplish this satisfactorily, or both cavities may be covered by a watch glass of suitable size. The development of a fugitive violet coloration, best observed upon a flat portion of the test plate, indicates the presence of rosin.

Standard Test Method for Color Performance of White Architectural Enamels

This test method describes a laboratory procedure for evaluating the color permanence of white architectural enamels. Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials. This test method was discontinued in 1992.



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Standard Test Method for Color of Transparent Liquids (Gardner Color Scale)¹

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This standard is issued under the fixed designation D 1544; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 4248 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

¹ NOTE—Editorial changes were made throughout in April 1989.

1. Scope

1.1 This test method covers the measurement of the color of transparent liquids by means of comparison with arbitrarily numbered glass standards. It applies to drying oils, varnishes, fatty acids, polymerized fatty acids, and resin solutions. Its application to other materials has not been tested.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method²
- E 308 Method for Computing the Colors of Objects by Using the CIE System³

3. Apparatus

3.1 *Glass Standards*, 18, numbered separately, and having the color characteristics given in Table 1. A suitable procedure for their calibration is contained in Appendix A1. The color shall be produced by the glass components only.

3.2 *Glass Tubes*, clear, 10.65 mm in inside diameter and about 114 mm in outside length. (Viscosity tubes, as described in Test Method D 1545, are satisfactory.)

3.3 Suitable apparatus for comparing sample and standard. The apparatus may be of any design, but should have the following characteristics:

3.3.1 *Illumination*—CIE Illuminant C.

3.3.2 *Surrounding Field*—The field should not differ significantly in brightness from the samples and standards and should be essentially achromatic.

3.3.3 *Field of View*—The specimen and one or more

TABLE 1 Color Specifications of Reference Standards

Gardner Color Standard Number	Chromaticity Coordinates ^A		Luminous Transmittance Y, %	Transmittance Tolerance, ±
	x	y		
1	0.3177	0.3303	80	7
2	0.3233	0.3352	79	7
3	0.3329	0.3452	76	6
4	0.3437	0.3644	75	5
5	0.3558	0.3840	74	4
6	0.3767	0.4061	71	4
7	0.4044	0.4352	67	4
8	0.4207	0.4498	64	4
9	0.4343	0.4640	61	4
10	0.4503	0.4760	57	4
11	0.4842	0.4818	45	4
12	0.5077	0.4638	36	5
13	0.5392	0.4458	30	6
14	0.5646	0.4270	22	6
15	0.5857	0.4089	16	2
16	0.6047	0.3921	11	1
17	0.6290	0.3701	6	1
18	0.6477	0.3521	4	1

^A A duplicate standard shall have chromaticity coordinates that differ from the reference standard by no more than one third of the difference in x or y between adjacent reference standards. In any one set, no two standards shall be closer together than two thirds of the difference in x or y between corresponding reference standards.

standards should subtend a visual angle of about 2 deg and be in the field of view simultaneously.

3.3.4 *Separation of Standard and Specimen*—There should be a perceptible separation between specimen and standard, but this should be as small as is mechanically possible.

4. Procedure

4.1 Fill a glass tube with the material under test. If the material is perceptibly cloudy, first filter it.

4.2 Compare with glass standards, determining which standard most closely matches the specimen in brightness and saturation. Ignore hue differences.

5. Report

5.1 Report the color as the number of the standard most closely matching the specimen. If more precise measurements are needed, report as either matching a standard or lighter or darker. Thus, between colors 5 and 6, the steps will be 5, 5+, 6-, and 6.

¹ This method is under the jurisdiction of Committee D-1 on Paint and Related Coatings and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved March 10, 1980. Published May 1980. Originally published as D 1544 - 58 T. Last previous edition D 1544 - 68 (1974).

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vol 14.02.

6. Precision and Bias

6.1 On the basis of a study in which one observer at each of 80 laboratories made duplicate determinations on four samples, the "between" and "within" standard deviations were found to be 0.5 and 0.1 color number, respectively. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

6.2 *Repeatability*—Two results obtained by a single operator should be considered suspect if they differ by more than two thirds of a color number.

6.3 *Reproducibility*—Two results, each of the mean of duplicate measurements, made by operators in different laboratories should be considered suspect if they differ by

more than four thirds of a color number.

NOTE 1—If desired, liquid standards matching the colors given in Table 1, in glass tubes similar to the sample tubes may be used. These may be filled with potassium chloroplatinate for the light colors and solutions of ferric chloride and cobalt chloride in hydrochloric acid for the darker colors. The specifications and approximate composition of these solutions are given in Test Method D 1544 - 58 T.⁴ Many Glass Standards in current use do not conform to the values of Table 1.

NOTE 2—The precision data were obtained using an instrument in which two standards are viewed simultaneously. There are other instruments available for color matching which would be expected to give similar results, but the statement above applies only to the instrument checked.

⁴ See 1961 Book of ASTM Standards, Part 8.

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION OF GLASS REFERENCE STANDARDS

X1.1 Select a dual beam spectrophotometer with a sufficiently small light beam at the sample position so that all rays will pass through the standards to be calibrated. Alternatively equip the spectrophotometer with a condensing lens to accomplish this purpose.

X1.2 Place the standards in turn in the sample position of the spectrophotometer. If the comparator is provided with a separate green filter in front of the light source, place this

filter in the reference beam of the dual beam spectrophotometer during calibration of each standard.

X1.3 Obtain spectral transmittance data for each glass reference standard by following Method E 308.

X1.4 From the spectral transmittance data for each reference standard calculate the CIE tristimulus values, X , Y , Z , and the chromaticity coordinates, x , y , for CIE Illuminant C (see Method E 308).

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Standard Test Method for Viscosity of Transparent Liquids by Bubble Time Method¹

This standard is issued under the fixed designation D 1545; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 4271 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of the viscosity in bubble seconds by timing. The bubble seconds are approximately equal to stokes for most liquids.

1.2 The test method is applicable to transparent liquids that are free from crystalline or gel particles.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 Definition:

2.1.1 *viscosity*—the resistance experienced by one portion of a liquid moving over another portion of the liquid. The absolute unit of viscosity in the cgs system is the poise which is expressed as dyne-seconds per square centimetre. Stokes are equal to poises divided by density. The absolute SI viscosity unit is the pascal-second.

3. Apparatus

3.1 *Constant-Temperature Bath*—Any suitable bath capable of maintaining temperature at $25 \pm 0.1^\circ\text{C}$ with water as the bath medium.

3.2 *Standard Viscosity Tubes*,² of clear glass and with flat bottoms, 10.65 ± 0.025 mm in inside diameter; 114 ± 1 mm in outside length. Plainly legible lines shall be located as follows (Note 1):

27 ± 0.5 mm
 100 ± 0.5 mm
 108 ± 0.5 mm

The distance between the first and second lines shall be 73 ± 0.5 mm.

NOTE 1—All distances shall be measured from the outside bottom of the tube.

3.3 *Reference Standards*—A series of standard viscosity

tubes (3.2) filled with transparent liquids having predetermined viscosities in stokes and bubble seconds and spaced in logarithmically even increments of about 26 % ($\log 1.260 = 0.100$), as listed in Table 1. The standards shall be marked alphabetically or numerically as shown in Table 1 without reference to stokes or bubble time (Note 2). Also listed in Table 1, for general and historical reference only, is the long established series of Gardner-Holdt letter standards, in tubes that do not have the three lines and are shorter than the standard viscosity tubes described in 3.2.

NOTE 2—For convenience, the reference standards can be divided in three series: light series, 15 tubes marked 0.22 to 8.0; heavy series, 14 tubes marked 10 to 200; and very heavy series, 7 tubes, marked 250 to 1000.

3.4 *Timing Device*—Stopwatch or electric stop clock capable of being read to a precision of 0.1 s.

3.5 *Tube Racks*, capable of inverting one or more viscosity tubes 180° to within 1° of a vertical position while rack and tubes are immersed in the constant temperature bath.

3.6 *Viscosity Tube Corks*, No. 2 short.

4. Procedure

4.1 Fill a standard viscosity tube with the material to be tested to approximately level with the 108-mm line.

4.2 Transfer the tube to a constant 25°C temperature bath with the cork loosely inserted. Hold at this temperature for 10 min.

NOTE 3—Adequate control of the temperature bath is essential. A variation of 0.1°C in the temperature of the bath will cause a 1 % variation in the timed bubble travel.

4.3 At the end of 10 min adjust the level of the liquid so that the bottom meniscus will be level with the 100-mm line. Insert the cork so that the bottom of the cork is on the level with the 108-mm line. This will ensure a bubble of suitable and uniform size.

4.4 Insert the tube in the rack and immerse in the 25°C water bath. Allow the tube(s) to stand with cork down in the bath a minimum of 20 min before determining the viscosity.

NOTE 4—For viscosities of liquids that have a timed bubble travel of 4 s or less, more precise results can be obtained by comparison against reference standards having a predetermined viscosity or timed bubble travel.

4.5 To read, invert the tube quickly and determine the time required for the bubble to rise in seconds. When determining the time in seconds start the timing device when the top of the bubble becomes tangent to the 27-mm line on

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 1545 - 58 T. Last previous edition D 1545 - 76(1989)¹.

² Empty standard viscosity tubes are available from the R. P. Cargille Laboratories, Inc., 33 Village Park Rd., Cedar Grove, NJ, or Byk-Gardner Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

TABLE 1 Recommended Numerical Standards for Comparator Viscosity Tubes^A

Bubble Seconds ^B	Tube ^C Number	Stokes	Gardner-Holdt Letter	Tube ^C Number	Stokes	Gardner-Holdt Letter
		0.10		10	10	W
		0.13	A3	13	13	X
		0.16		16	16	Y
0.75	0.22	0.20	A2	20	20	Z
		0.25		25	25	Z1
0.81	0.34	0.32	A1	32	32	Z2
		0.40		40	40	Z3
0.90	0.50	0.50	A	50	50	
		0.63	B	63	63	Z4
1.00	0.68	0.80	C	80	80	
1.15	0.92					Z5
1.30	1.15	1.00	D	100	100	
		1.25	E	125	125	
1.55	1.45	1.60	F	160	160	Z6
		2.00	G	200	200	
1.85	1.80	2.50	H	250	250	
2.20	2.15		I			
2.65 ^D	2.65 ^D	2.50	J	250	250	
			K			
3.20	3.20	3.20	L	320	320	
			M			
			N			
			O			Z7
4.00	4.00	4.00	P	400	400	
			Q			
5.00	5.00	5.00	R	500	500	
			S			
			T			Z8
6.30	6.30	6.30	U	630	630	
8.00	8.00	8.00	V	800	800	Z9
10.0	10.0	10.0		1000	1000	Z10

^A Arranged to show relationship between stokes, bubble seconds, and Gardner-Holdt letters. Stokes are shown in logarithmic progression.

^B The bubble time, in seconds, of the numerical tubes under 4 s was determined by a technique employing a movie camera.

^C For reference purposes only. Numbered tubes are no longer commercially available.

^D Above 2.65 the bubble seconds as measured by the kinematic method are approximately equivalent for most products. Below 2.65 this relationship does not hold.

the tube. Stop the timing when the top of the bubble becomes tangent with the 100-mm line. This gives a 73-mm timed bubble travel. All timings shall be made with the tube in an exact vertical position.

NOTE 5—Positioning of the tube in a vertical position is mandatory. A tube one radius off the vertical will give an error of approximately 10 % in the time of bubble travel.

5. Report

5.1 Report the following information:

5.1.1 The viscosity obtained by the timing method expressed as "bubble seconds" or "approximate stokes."

NOTE 6—The time in seconds or "bubble seconds" is an approximate measurement of stokes when the bubble time method is applied to most raw materials and finished products encountered by the paint, varnish, and lacquer industries. This relationship does not hold for silicones or water dispersions of gums or similar materials. Viscosity results by this test method are influenced by non-Newtonian behavior and by surface tension. Other variations on bubble-tube viscometers are described in ASTM STP 500, Section 3.2.9.³

6. Precision and Bias

6.1 On the basis of an interlaboratory study⁴ of this test method in which ten laboratories tested liquids ranging in viscosity from 4.5 to 440 St, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

6.1.1 Two results obtained by the same operator should be considered suspect if they differ by more than 4.9 % relative.

6.1.2 Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 9.0 % relative.

³ Paint Testing Manual, ASTM STP 500, ASTM, 1972.

⁴ See Holt, K. E., *Proceedings, ASTM*, Vol 57, 1957, pp. 297-300, and *Official Digest*, Federation of Societies for Paint Technology, Vol 30, May 1958, pp. 540-543.

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Standard Method for Performance Tests of Clear Floor Sealers¹

This standard is issued under the fixed designation D 1546; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers the testing of clear floor sealers for appearance of finish, treatment of worn areas, the application of finishing materials such as varnish, liquid or paste waxes either solvent or emulsion type, and resistance to ink staining.

NOTE 1—The resistance of the sealer to other reagents may be determined by this procedure when agreed upon by the purchaser and the seller.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 *U.S. Federal Specifications:*
 - P-W-155 Solvent Wax²
 - P-W-158 Emulsion Wax²
 - TT-I-563 Blue-Black Ink²
 - TT-V-71-f Varnish²

3. Apparatus

- 3.1 *Maple Panels*—First grade maple flooring 2½ by 12 in. (64 by 305 mm).
- 3.2 *Sandpaper*—No 0 and 00 garnet paper.
- 3.3 *Cheesecloth*.
- 3.4 *Blotting Paper*.

4. Materials

- 4.1 *Blue-Black Ink*, (Federal Specification TT-I-563).
- 4.2 *Varnish*, (Federal Specification TT-V-71-f).
- 4.3 *Solvent Wax*, (Federal Specification P-W-158).

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.33 on Varnish and Resins, Including Shellac.

Current edition approved Sept. 28, 1962. Published November 1962. Originally published as D 1546 - 58 T. Last previous edition D 1546 - 58.

² Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

4.4 *Emulsion Wax*, (Federal Specification P-W-155).

5. Panel Preparation

5.1 Sand the five maple panels with No. 00 garnet paper until the surface is flat and smooth. Remove the excess sanding dust with a clean dry cloth.

5.2 Apply a liberal coat of sealer to the panels using a pad of clean cheesecloth. Note fluidity and ease of application. Allow the sealer to set 15 min for absorption into the wood. Wipe off the excess sealer using a pad of cheesecloth (note whether the excess sealer removed in this manner can be wiped readily and cleanly from the surface). The surface shall be free of all excess sealer. Allow the panels to dry 24 h at a temperature between 70 and 90°F (21.1 and 32.2°C).

5.3 Apply a second coat of sealer following the procedure described in 5.2.

NOTE 2—Other methods of application and panel preparation may be used when agreed upon by the purchaser and the seller.

6. Procedure

6.1 *Appearance of Finish*—Examine the panel, treated as described in Section 5, for the presence of surface film. Note whether the treated surface of the panel has a soft, even sheen and whether the grain of the wood is clouded or obscured.

6.2 *Resistance to Ink Stain*—Place several drops of blue-black ink on the surface of a treated panel at various places and allow to remain for 3 min. Take up the ink with blotting paper and wipe the spots lightly with a damp cloth. Examine the surface for any indication of the presence of ink.

6.3 *Treatment of Worn Spots*—Simulate on a treated panel worn areas by rubbing the surface with No. 0 steel wool. Over these panels apply a thin coat of sealer. Burnish the worn areas with No. 00 steel wool immediately after application of the sealer. When dry, examine the treated areas to determine whether they blend with the surface of the panel without showing any signs of lap marks.

6.4 *Application of Finishing Materials*—To a treated panel apply a finish coat of the varnish. To a second treated panel apply a coat of the solvent wax. To a third treated panel apply the emulsion wax. Note whether the properties of the finishing materials are adversely affected by the sealed surface or whether the finishing material adversely affects the sealed surface.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Acid Value of Organic Coating Materials¹

This standard is issued under the fixed designation D 1639; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Methods 5071, 5072, 5073 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the measurement of the free acidity present in the nonvolatile portion of varnishes, oils, certain resins, and paint vehicles, by the reaction with standard alkali solution.

1.2 If carboxylic anhydrides are present, only one half of the reactive groups will be titrated and indicated by this test method.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 362 Specification for Industrial Grade Toluene²

D 770 Specification for Isopropyl Alcohol²

D 1259 Test Methods for Nonvolatile Content of Resin Solutions³

D 1644 Test Methods for Nonvolatile Content of Varnishes⁴

D 1960 Test Method for Loss on Heating of Drying Oils²

3. Terminology

3.1 Definition:

3.1.1 *acid value*—the number of milligrams of potassium hydroxide (KOH) required to neutralize the alkali-reactive groups in 1 g of material under the conditions of test (see 6.4).

4. Significance and Use

4.1 This test method is used to determine the free acidity present in the nonvolatile portion of varnishes, oils, certain resins, and paint vehicles by the reaction with a standard alkali solution. Use of this test method provides a means whereby the relative applicability of the varnish, oil, resin, or paint vehicle to the particular end use may be estimated by the buyer and the seller.

4.2 This test method also provides a convenient method of process control for the manufacture of certain resins and paint vehicles designed to meet particular requirements of the buyer and the seller.

5. Reagents and Solvents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of methanol, ethanol, or isopropanol.

5.3 *Potassium Hydroxide, Methyl Alcohol (Methanol) Solution* (1 mL = 5.6 mg KOH)—Dissolve 6.6 g of potassium hydroxide (KOH) in 1 L of methyl alcohol. Standardize against National Institute of Standards and Technology standard potassium hydrogen phthalate Standard Reference No. 84, using phenolphthalein as the indicator (5.2). Do not adjust the concentration of the solution, but calculate the milligrams of KOH per litre of solution, K. (See Note.)

NOTE 1—At the discretion of the purchaser and the seller, an aqueous solution of potassium hydroxide of 0.1 N or 0.5 N may be used.

NOTE 2—Potassium hydroxide, methyl alcohol (methanol) solution may be purchased from most major laboratory chemical supplier houses. This material should be standardized as just indicated.

5.4 *Neutral Solvent Mixture*—Mix equal parts by volume of isopropyl alcohol (isopropanol) (see Specification D 770) and industrial toluene (see Specification D 362). Neutralize the mixture using 0.1 N KOH solution (4.3) and phenolphthalein indicator solution (5.2) until the pink color persists for 1 min.

6. Procedure

6.1 Weigh or transfer into a 250-mL Erlenmeyer flask the specimen mass, S, prescribed in Table 1.

6.2 Add 100 mL of neutral solvent. Mix until all material is dissolved, warming if necessary. Cool to room tempera-

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as D 1639 - 51. Last previous edition D 1639 - 89.

² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

TABLE 1 Specimen Size

Expected Acid Value	Approximate Specimen Mass, g	Accuracy of weighing, plus or minus, mg
0 to 5	20	50
Over 5 to 15	10	50
Over 15 to 30	5	50
Over 30 to 100	2.5	1
Over 100	1	1

ture, about 77°F (25°C), before titrating. Add 1 mL of phenolphthalein indicator solution and titrate with the 0.1 N KOH solution (5.3) to the end point, which is a pink color persisting for 30 s (V).

6.3 Repeat with a second specimen.

6.4 In the case of materials containing a solvent, determine the nonvolatile content in accordance with Test Methods D 1259, D 1644, D 1960 or other applicable method agreed upon between the purchaser and the seller.

7. Calculation

7.1 Calculate the acid value *A* as follows:

$$A = \frac{VK}{S \times N}$$

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where:

V = volume of KOH solution required for titration of the specimen, mL,

K = weight of KOH per millilitre of KOH solution, g,

S = specimen weight, g, and

N = nonvolatile content of the material expressed as a decimal fraction.

7.2 Calculate the mean of the two runs.

8. Report

8.1 Report the acid value of the nonvolatile matter to the nearest decimal (0.1).

9. Precision

9.1 Because of the many types of material covered by this test method, the precision is not as good as might be expected of a quantitative analytical method. The following criteria should be used for judging the acceptability of results:

9.1.1 *Repeatability*—Duplicate results obtained by the same operator should be considered suspect if they differ by more than 10 % of the mean acid value.

9.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 20 % of the mean acid value.

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Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature¹

This standard is issued under the fixed designation D 1640; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Editorial changes were made throughout in April 1989.

1. Scope

1.1 These test methods cover the determination of the various stages and rates of film formation in the drying or curing of organic coatings normally used under conditions of ambient room temperature.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D202 Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation²

D823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

D1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers³

D2091 Test Method for Print Resistance of Lacquers³

2.2 U.S. Government Standards:

Fed. Spec. No. CCC-C-440, Cheesecloth⁴

Fed. Spec. No. CCC-C-419b, Type III, Army Duck⁴

2.3 TAPPI Standards:⁵

T 402 Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products

3. Significance and Use

3.1 These test methods are used to determine the various stages and rates of drying, curing, and film formation of organic coatings for the purpose of comparing types of

coatings or ingredient changes, or both. This is significant in the development of organic coatings for various end uses and also for production quality control.

4. Coatings and Recommended Film Thicknesses

4.1 Whenever tests are to be performed on coatings not listed in Table 1, there should be a prior agreement between the purchaser and seller as to the substrate, film thickness, and application method for testing the specific coating involved.

5. Test Conditions

5.1 Conduct all drying tests in a well-ventilated room or chamber, free from direct drafts (Note 1), dust, products of combustion, laboratory fumes and under diffused light (see 5.4). Make all measurements at a temperature of $23 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ relative humidity with the coated panels in a horizontal position while drying.

NOTE 1—A device to equalize air change conditions has been developed by F. Scofield.⁶ Relative humidity should be controlled for moisture-cured and two-package urethane coatings, since their cure is greatly affected by the existing moisture conditions.

5.2 Tests should be carried out at practical viscosities at which films can be applied to the proper film thickness with resultant good flow and leveling properties. In the absence of any specific material specification, instructions for preparation of the film should be determined and agreed upon between the purchaser and the seller.

5.3 Films to be tested should have practical thicknesses commensurate with performance characteristics expected under actual usage for the type under test. All testing should be done within an area, any point of which is not less than $\frac{1}{2}$ in. (15 mm) from the film edge.

5.4 *Light Conditions*—Illumination of the films during the entire drying test period should be about 25 ft-candles (270 lx) from normal laboratory or sky sources, never from direct sunlight or other sources high in nonvisible radiant energy.

6. Preparation of Test Specimens

6.1 Carry out all tests as described in 6.1.1, 6.1.2 and 6.1.3, unless otherwise noted.

⁶ Gardner and Sward, *Paint Testing Manual*, ASTM STP 500, ASTM, 13th edition, 1972, p. 269.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved March 25, 1983. Published July 1983. Originally published as D 1640 - 59 T. Last previous edition D 1640 - 69 (1974).

² *Annual Book of ASTM Standards*, Vols 10.01 and 15.09.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁵ Available from Technical Association of the Pulp and Paper Industry, Technology Park, P.O. Box 105113, Atlanta, GA 30348.

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6.1.2 Ap panels or agreed upo glass plates that tend to plates can l glass by gr water betw

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TABLE

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^b See 6.1

6.1.1 All test specimens shall be prepared and tested by one operator properly skilled in the methods to be used. Apply the specimens in duplicate at a time arranged so that examination intervals will fall within the normal working hours of the operator.

6.1.2 Apply the materials to be tested on clean glass panels or other specific substrate of suitable dimensions agreed upon between the purchaser and the seller. Ground-glass plates are more suitable for certain types of coatings that tend to crawl, such as low-viscosity drying oils. Suitable plates can be prepared by roughening the surface of polished glass by grinding a paste of silicon carbide (grit 1-F) and water between two glass plates.

6.1.3 The test films preferably shall be cast with a doctor blade having a clearance sufficient to give the recommended dry film thickness indicated in Table 1. When a suitable doctor blade is not available, or it has been agreed upon to apply the film in some other manner, the various conventional and automatic methods of spray, dip, flow, and brush application may be used, provided dry film thicknesses conform to the requirements given in Table 1. See Test Methods D 823 for a description of the spray and dip methods of application.

6.1.4 Measure the dry film thickness of test films with the proper film thickness gage. This shall be a micrometer, dial comparator, or dial indicator as described in Test Methods D 1005. When plates of small area are used, measurement of dry film thickness can be made by weighing plates before and after coating and calculating from plate area and coating solids.

7. Procedure

7.1 When test methods or end points other than those listed in 7.2 to 7.9 are used, there shall be a prior agreement between the purchaser and the seller.

7.2 **Set-To-Touch Time**—To determine set-to-touch time, lightly touch the test film with the tip of a clean finger and immediately place the fingertip against a piece of clean, clear glass. Observe if any of the coating is transferred to the glass. For the purpose of this test, the pressure of the fingertip against the coating shall not be greater than that required to transfer a spot of the coating from $\frac{1}{8}$ to $\frac{3}{16}$ in. (3 to 5 mm) in cross section. The film is set-to-touch when it still shows a tacky condition, but none of it adheres to the finger.

7.3 Dust-Free Times:

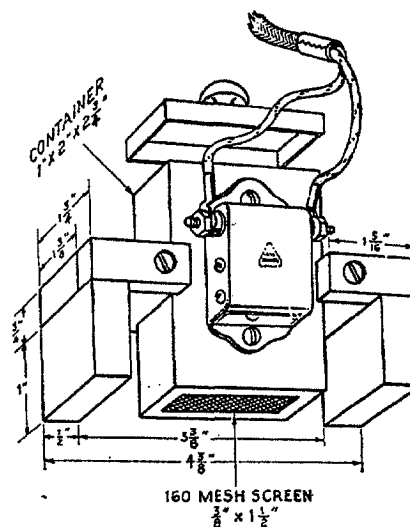
7.3.1 **Cotton Fiber Test Method**—Separate a number of individual fibers from a mass of absorbent cotton with the

TABLE 1 Recommended Film Thickness of Materials to be Tested^a

Material	Dry Film Thickness
Drying oils	1.25 ± 0.25 mil (32 ± 6 μ m) ^b
Varnishes	1 ± 0.1 mil (25 ± 2 μ m) (See 7.3.2)
Laquers	0.5 ± 0.1 mil (12.5 ± 2 μ m) (See 7.4.2)
Resin solutions	0.5 ± 0.1 mil (12.5 ± 2 μ m)
Enamels	1.5 ± 0.25 mil (36.5 ± 6 μ m)
Oil paints	1.8 ± 0.2 mil (46 ± 2.5 μ m) (see 6.2)
Water paints	1 ± 0.1 mil (25 ± 2 μ m)

^a This table is a general guide to be used when nothing more specific is agreed upon between the purchaser and the seller.

^b See 6.2 and 7.4.1. Add driers a minimum of 24 h before test.



NOTE—1 in. = 25.4 mm.

FIG. 1 Typical Dust-Free Tester

aid of tweezers. At regular drying intervals, drop several of the cotton fibers from a height of 1 in. (25 mm) onto a marked section of the film. The film is considered to have dried dust free when the cotton fibers can be removed by blowing lightly over the surface of the film.

7.3.2 **Powder Test Method**—Deposit finely divided calcium carbonate (pigment grade) on the film at definite intervals during the drying period and, after the film has dried tack-free, remove by blowing with a gentle stream of air and wiping with a soft rag or camel-hair brush. The coating is considered dust-free at the time interval when the pigment can be removed completely.

7.3.2.1 Deposit the calcium carbonate from a box through a 160-mesh screen in a pattern $1\frac{1}{2}$ in. (40 mm) long and $\frac{3}{8}$ in. (10 mm) wide. Fasten a vibrator to the side of the container to facilitate the passage of the pigment through the screen in a uniform pattern. Support the apparatus so that there is a clearance of about $\frac{1}{8}$ in. (3 mm) between the screen and the test film. Figure 1 shows a typical dust-free tester⁷ and Fig. 2 shows the pattern obtained in testing a black enamel. The enamel shown is considered dust free at the 4½-h rating.

7.4 Tack-Free Times:

7.4.1 Paper Test Method:

7.4.1.1 **Test Paper**—The test paper shall be K-4 Power Cable Paper⁸ that when conditioned in accordance with the TAPPI Standard Method T 402, conforms to the following requirements:

⁷ The dust-free tester was designed and built by Technical Subcommittee 37 of the New York Paint and Varnish Production Club and is described in "Investigation of Methods of Measuring Drying Time," *Official Digest*, Federation of Paint and Varnish Production Clubs, No. 286, November 1948, pp. 836-843. This paper also includes a study of the Zapon tack tester.

⁸ Paper meeting these requirements may be obtained from Crocker Technical Papers, Inc., 431 Westminster St., Fitchburg, MA 01420, their Grade R 20-34.

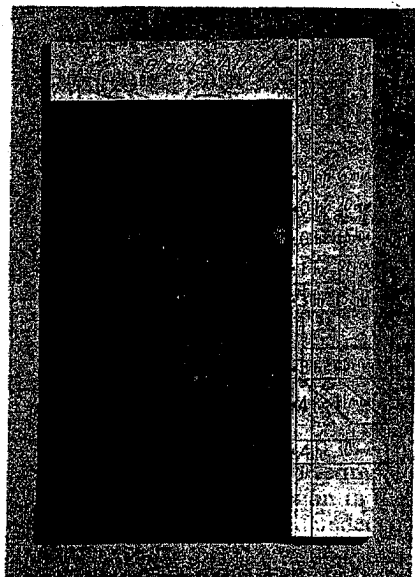


FIG. 2 Example Pattern from Dust-Free Drying Time Test

Basis weight (24 by 36/500), lb	90 ± 5
Thickness, mils (μm)	6.65 (17)
Air resistance (s/100 cm ² /in. ²)	350
Coefficient of static friction ⁴	0.5
Friction angle, °	22
Tensile strength, machine direction/cross direction	119/32
Tear, machine direction/cross direction	180/250
Elongation, machine direction/cross direction, %	3.0/7.0
pH of water extract	7.4
Ash content, max, %	0.6

⁴ All tests except this one shall be run in accordance with Test Method D 202. All values for properties are typical values and not specification limits.

7.4.1.2 Lay a 2 by 3-in. (50 by 75-mm) piece of the special test paper on the film and place upon it a steel cylinder 2 in. in diameter, and of such weight 6.28 lb, (2.85 kg), as to produce a pressure of 2 psi (13.8 kPa). At the end of 5 s, remove the cylinder and invert the test panel. The film is considered free from after-tack when the paper drops off of the test film within 10 s.

7.4.2 A variation of the test method described in 7.4.1 using the same test paper can be used to test the tack-free time of insulating varnishes. In this method the piece of paper shall be 1½ in. (40 mm) in width and 6 in. (150 mm) in length. The varnish is considered tack-free when this strip of paper does not adhere to it when it is pressed on the surface of the varnish for 1 min by a cylindrical 1-lb (450-g) weight, 1 in. (25 mm) in diameter. In this test, apply the paper in the vicinity of the center of the specimen at right angles to the length of the coated specimen.

7.4.3 *Mechanical Test Method (Tack Tester)*⁹—The tack tester to be used in this method comprises essentially a base or surface-contacting portion 1-in. (25-mm) square and a counter-balancing portion 1 by 2 in. (25 by 50 mm) in area. Both portions are made up from a continuous metal strip

⁹ The standard tack tester is fully described in the U. S. Patent 2,406,989, Sept. 3, 1946.

0.016 to 0.018 in. (0.41 to 0.46 mm) in thickness. To prepare the apparatus for use (see 7.4.3.1), fit the base with several thicknesses of masking tape and paper strips to provide a means of attaching the aluminum foil and so adjust the angle of the 1 by 2-in. counter-balancing strip so that a weight of 5 g placed in the geometric center of the base portion is just sufficient to overcome the unbalanced force.

7.4.3.1 The tester is prepared for use by carrying out the following steps in sequence:

(1) Wrap the metal base with three thicknesses of masking tape, sticky side out,

(2) Cover the outer layer with a good grade of paper, except for two exposed strips, equally spaced, about ¼ by 1 in. (6.4 by 25 mm) in area on the top of the tester, and

(3) Cover the paper on the contact side of the base with one thickness of pressure-sensitive cellulose tape previously fixed to the metal base of the tester. The cellulose tape serves two purposes:

First, to pull the layers of masking tape firmly against the front of the metal base, and

Second, to provide a smooth surface for the foil. Attach the aluminum foil to the base of the tester by pressing gently but firmly a 1 by 2-in. (25 by 50-mm) piece of foil, 0.0005 in. (13 μm) in thickness against one of the ¼ by 1-in. (6.4 by 25-mm) exposed strips of masking tape on the top surface of the base. Wrap the foil tightly and smoothly around the base, exposing the shiny side, and finally press the outer end gently against the remaining exposed strip of masking tape. When it finally becomes necessary to replace wrinkled or soiled aluminum foil, the ends are easily removed from the masking tape by exerting a slow, even, upward pull sufficient to overcome the tack of the tape without tearing the foil.

7.4.3.2 A film is considered to have dried tack-free when the tack tester tips over immediately on removing a 300-g weight allowed to act for 5 s on the counter-weighted metal square base fitted with masking tape and aluminum foil.

7.5 Dry-To-Touch Time:

7.5.1 *Drying Oils*—Continue testing after the set-to-touch time has been observed. The film is considered dry when it no longer adheres to the finger and does not rub up appreciably when the finger is lightly rubbed across the surface.

7.5.2 *Lacquers (and Sealers)*—Touch the film lightly at varying intervals of time. The film is considered dry when no pronounced marks are left by the finger touching the film in the same area on each observation. Test sealers on wood or other porous substrates as agreed upon between the purchaser and the seller.

7.6 Dry-Hard Time:

7.6.1 With the end of the thumb resting on the test film and the forefinger supporting the test panel, exert a maximum downward pressure (without twisting) of the thumb on the film. Lightly polish the contacted area with a soft cloth. The film is considered dry-hard when any mark left by the thumb is completely removed by the polishing operation.

7.7 Dry-Through (or Dry-To-Handle) Time:

7.7.1 Place the test panel in a horizontal position at a height such that when the thumb is placed on the film, the arm of the operator is in a vertical line from the wrist to the shoulder. Bear down on the film with the thumb, exerting the maximum pressure of the arm, at the same time turning

the thumb. The film there is evidence

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the thumb through an angle of 90° in the plane of the film. The film is considered dry-through or dry-to-handle when there is no loosening, detachment, wrinkling, or other evidence of distortion of the film.

7.8 Dry-To-Recoat:

7.8.1 A film is considered dry for recoating when a second coat or specified topcoat can be applied without the development of any film irregularities such as lifting or loss of adhesion of the first coat, and the dry time of the second coat does not exceed the maximum specified (if any) for the first coat.

7.9 Print-Free Time:

NOTE 2—This procedure is similar to Test Method D 2091, except that the time to reach the print-free condition is determined, while Test Method D 2091 is used to evaluate whether a film is print free at a specified time.

7.9.1 *Test Panels*—Apply the material under test to clean plane panels, at least 3 by 6 in. (75 by 150 mm) in size, made of wood, metal, glass, plastic or other material as agreed upon between the purchaser and the seller.

7.9.2 *Imprinting Fabric*—Eight-ounce Army duck conforming to Type III of U.S. Fed. Spec. No. CCC-C-419b or cheesecloth conforming to Fed. Spec. No. CCC-C-440.

7.9.2.1 A pad should be used with the cheesecloth only, made of nonwoven felt cloth at least 0.05 in. (1.3 mm) thick, weighing 7 oz/yd² (0.24 kg/m²) and larger than the plane end of the weight.

7.9.3 *Weights*—Consisting of metal cylinders not less than 2 in. (50 mm) in diameter with plane ends perpendicular to the axis and of a length to give a pressure of ½ or 1 lb/in.² (3.5 or 6.9 kPa).

7.9.4 Procedure:

7.9.4.1 Apply the test material to several of the specified or agreed-upon panels by a film applicator, or other specified method, as described in Test Methods D 823 in either single or multiple coats, as agreed upon between the purchaser and

the seller. In the absence of a specified dry film thickness, the values listed in Table 1 should be used.

7.9.4.2 Allow the coated panels to dry under the conditions specified in Section 5, unless otherwise agreed. At appropriate intervals, starting shortly before the coating is expected to be print-free, carry out the print-free test as described in Test Method D 2091, comparing the appearance with the photographic standards appearing therein, until the test shows the coating to be print-free.

8. Frequency of Testing

8.1 It is suggested that test intervals be set at periods of approximately 10 % of the total test time. If frequency varies considerably from the 10 % interval or such time interval is impractical, the intervals used shall be reported.

9. Report

9.1 Reports of tests shall include all applicable conditions that deviated from the standards as outlined or special conditions or tests used and the results of the test.

10. Precision

10.1 Because of the subjective nature of the drying time tests, the agreement to be expected between laboratories depends upon their understanding of the terms used, and is difficult to establish with certainty. Within any laboratory, the agreement depends upon the material being tested, some coatings being much sharper in their end point than others, but duplicate determinations should agree within 10 % of the time of drying.¹⁰

11. Keyword

11.1 drying time

¹⁰ See Prane, J. W., "A Latin Square Drying Time Study," *Paint Industry Magazine* (August 1961), for a study of precision of drying time measurements.

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Standard Test Method for Exterior Durability of Varnishes¹

This standard is issued under the fixed designation D 1641; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the testing of the exterior durability of varnishes applied to a wooden base.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standard:

D962 Specification for Aluminum Powder and Paste Pigments for Paints²

2.2 Military Specification:

MIL-V-16399 Varnish, Moisture-Proof³

3. Apparatus

3.1 *Maple Panels*, of close-grained clear maple, 3 by 12 by 1/2 in. (76 by 305 by 13 mm), with all edges rounded to a 1/4-in. (6.4-mm) radius.

3.2 *Varnish Brushes*, 1-in. (25-mm) pure bristle.

3.3 *Sandpaper*, 180-grit.

3.4 *Balance*, capable of weighing to an accuracy of 0.1 g.

3.5 *Tung Oil Phenolic Aluminum Sealer*, 12 1/2 gal (47.3 L) "oil length" (12 1/2 gal of oil/100 lb (45.4 kg) of resin.

NOTE 1—This sealer is composed of a vehicle made in accordance with Military Specification MIL-V-16399 Type B, 27 June, 1951, plus aluminum paste meeting the requirements of Specification D 962, Type II, Class B, using: 1 gal MIL-V-16399 Type B varnish,³ and 2 lb aluminum paste.

4. Preparation of Test Panels

4.1 *Weight of Panel*—Weigh the test panel to the nearest 0.1 g, prior to varnishing.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.33 on Varnish and Resins, Including Shellac.

Current edition approved Sept. 10, 1959. Published November 1959. Replaces Sections 56-60 of D 154.

² *Annual Book of ASTM Standards*, Vol 06.02.

³ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

4.2 *Application of First Coat*—For the first coat, thin a portion of the varnish in the proportion of 1 pt of mineral spirits thinner to 1 gal (500 ml in 4 L) of varnish. Coat both sides, ends, and all edges of the test panel, using a small brush and taking the precaution to brush the coat out well. Allow the varnish coat to dry 24 h at room temperature before applying the second coat.

4.3 *Application of Second Coat*—Before applying the second coat, lightly sand the panel with 180-grit sandpaper, and reweigh the panel. Record the final weight of the first coat. Do not touch the sides of the panel after sanding. Using the varnish as received, apply a second coat, covering both sides and all edges of the panel. Apply the second coat as evenly as possible. Allow it to dry for 24 h before applying the third coat.

4.4 *Application of Third Coat*—Before applying the third coat, lightly sand the panel with 180-grit sandpaper, taking care not to touch the sides of the panel after sanding. Weigh the panel, and record the weight of the second coat. Using the varnish as received, apply the third coat, covering both sides and all edges of the panel. Apply this coat with a full brush, laying the varnish on as evenly as possible. Allow it to dry for 24 h. Weigh the panel, and record the weight of the third and final coat.

4.5 *Application of Aluminum Sealer*—Using the aluminum sealer supplied with the varnish samples, coat one side and all the edges of the panel with a smooth, even coat. Great care should be taken to see that the ends and edges of the panel are completely covered. Allow the panel to dry a minimum of 24 h before exposure.

5. Exposure

5.1 Expose all panels outdoors in the early Spring, at 45° facing south. The weather conditions at the site of exposure should duplicate, if possible, the conditions under which varnish will be exposed in regular use.

6. Panel Examination

6.1 Inspect the panels at monthly intervals after the initiation of the exposure test. Sponge off the panels with a pad of cheesecloth under a gentle stream of cold water, and immediately dry thoroughly with a soft cloth or low-pressure stream of air. Examine each panel for the following failure characteristics, grading each type of failure from "1" to "10,"

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- "1" signifying complete failure, and "10" signifying no evidence-of-failure characteristics.
- 6.1.1 General appearance,
 - 6.1.2 Cracking,
 - 6.1.3 Checking,
 - 6.1.4 Dulling,
 - 6.1.5 Discoloration, and
 - 6.1.6 Recoatability.

7. Report⁴

7.1 Report the results of the examination of the test panels in accordance with Section 6.

⁴ The cooperative, experimental results on which this method is based were published in the *Official Digest*, Federation of Paint and Varnish Production Clubs, April 1952, pp. 219 to 220.

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Standard Test Methods For Elasticity or Toughness of Varnishes¹

This standard is issued under the fixed designation D 1642; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These methods have been approved for use by agencies of the Department of Defense to replace Methods 4151, 4152 of Federal Test Method Standard No. 141 and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These methods cover the determination of elasticity or toughness of any varnish which is miscible with the diluents specified, under the conditions of the test.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Methods

2.1 Elasticity or toughness of the varnish may be determined by proportionately reducing its elasticity or toughness by the addition of a standard solution of a resin. The long-accepted use of run-kauri gum may now be less convenient due to a limited supply of this resin. Accordingly, an equally acceptable resin is prescribed in Method A (Sections 3 to 6). Method A is based on the preparation of a rosin-pentaerythritol ester, the properties of which in terms of acid value and melting point can be controlled, and on a solvent that is free of any inherent polymerization tendency. Where desired, run-kauri gum may be used as prescribed in Method B (Sections 7 to 9).

2.2 For varnishes that are less elastic or tougher than zero standard resin reduction, Method C (Sections 10 to 13), based on proportionately increasing the elasticity or toughness of the varnish by means of the addition of linseed oil, is provided.

METHOD A—STANDARD ROSIN PENTAERYTHRITOL ESTER RESIN REDUCTION

3. Preparation of the Rosin-Pentaerythritol Ester Solution

3.1 Apparatus:

3.1.1 *Flask*, 3-litre, 3-neck flask.

3.1.2 *Gas Burner* having a capacity of 12 000 Btu/h (3.5 kW).

3.1.3 *Agitator*—Motor-driven agitator with blade peripheral speed of 600 ft (182.9 m)/min. The agitator shall be of the three-blade type with a blade width of $\frac{3}{4}$ in. (19 mm) and a blade diameter of $1\frac{1}{4}$ in. (44.5 mm).

¹ These methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.33 on Varnish and Resins, Including Shellac.

Current edition effective Dec. 24, 1970. Originally issued 1959. Replaces D 1642 - 59. Replaces Sections 20-32 of D 54.

3.1.4 *Thermometer*—An ASTM Partial Immersion Thermometer having a range from 20 to 760 F, 16 in. (406 mm) in length, and conforming to the requirements for Thermometer 3F in ASTM Specification E 1, for ASTM Thermometers.²

3.1.5 Carbon Dioxide Supply.

3.2 Materials:

3.2.1 W. W. Gum Rosin:

Melting point	173 to 177 F (78 to 81 C)
Acid number	165 to 167

3.2.2 Pentaerythritol:

Hydroxyl content	49.5%
Ash content	less than 0.2%
Melting point	at least 250 C

3.2.3 Hi-Flash Naphtha:

Type	aromatic
Color	water-white
Distillation range	first drop not below 150 C
Dry point	not above 193 C
Acid wash color	not darker than No. 10
	color standard ³
Specific gravity 15.56/15.56 C	0.855 to 0.890
Flash point (Tag open cup)	105 F (40.66 C)

3.3 *Procedure*—Heat 3 lb (1361 g) of rosin in the flask fitted with the thermometer, a carbon dioxide inlet extending below the surface of the reacting liquid, and a loose cork stuffing box fitting the center neck of the flask through which is extended the agitator shaft. When the rosin reaches a temperature of 450 F (232 C), start the motor stirrer, adjust the CO₂ flow so that about two bubbles break to the surface per second, and add 0.33 lb (150 g) of pentaerythritol slowly through the center neck while keeping the agitator running. The size of the hole in the cork accommodating the agitator shaft should be large enough to allow the escape of CO₂ and water vapor from the flask and prevent any pressure build-up. After all of the pentaerythritol has been added, heat to 600 F (315 C) and hold for an acid number of 8. The softening point should be about 263 F (128 C) as determined by the Test Method for Softening Point by Ring and Ball Apparatus (ASTM Designation: E 28).⁴ The time required is about 6½ h at this temperature. When the resin reaches the correct acid number and melting point, turn off the heat. Allow the carbon dioxide to flow until the flask has cooled to 400 F (204 C), then pour the liquid resin into a tared receiver and carefully reduce to 33½ % nonvolatile matter content.

² *Annual Book of ASTM Standards*, Vols 05.03 and 14.01.

³ ASTM Method D 848, Test Method for Acid Wash Color of Industrial Aromatic Hydrocarbons, see *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.03.

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⁵ *Annual Book of*

with hi-flash naphtha. Store the solution in tightly-closed amber glass bottles.

4. Test Panels

4.1 Test panels shall be No. 31 gage (0.227 mm) bright tin plate weighing not more than 0.51 nor less than 0.39 lb/ft² (25 or less than 19 g/dm²). It is important that the tin plate shall be within the limits prescribed. The panel shall be about 3 by 5 in. (76 by 120 mm) and shall be thoroughly cleaned with benzene immediately before using (cleaned but not buffed) and wiped dry with a clean cloth.

5. Procedure

5.1 *Reduction of the Varnish*—Carefully determine the nonvolatile matter content of the varnish under test, as described in ASTM Methods D 1644, Test for Nonvolatile Content of Varnishes.⁵ Then take at least 10 g of the varnish and add to it an amount of the standard resin solution equivalent to the specified percentage reduction by weight of the nonvolatile matter in the varnish. Mix the varnish and the solution thoroughly.

5.2 *Application of Reduced Varnish*—Flow a coat of the reduced varnish on one of the tin panels described in Section 4. Let stand in a nearly vertical position at room temperature 70 to 90 F (21 to 32 C) for 1 h. Place the panel in a horizontal position in a properly ventilated oven and bake for 5 h at 203 to 212 F (95 to 100 C). Remove the panel from the oven and allow to cool at 77 F (25 C) for 30 min.

5.3 *Bending of Test Panel*—Quickly bend the coated panel over a 1/8-in. (3-mm) rod held firmly by suitable supports. With the coated side of the panel uppermost, place it in position on the rod so that the rod is equidistant from the top and bottom edges of the panel. Bend the panel double in 1 s at a temperature of 77 ± 0.2 F (25 ± 0.1 C). Examine the film at the bend under a magnification of 5 diameters, using diffused light having a color temperature of 6500 K and intensity of about 165 ft-candles (1770 lx). (For those not equipped with a standard illuminant, viewing the panel at a north window illuminated by a fairly light overcast sky will approximate these conditions.) No cracks shall occur over the bent area between the points 1/4 in. (6.4 mm) from each side of the panel.

6. Report

6.1 Report varnishes that do not show cracks under this test as passing the percentage of standard resin reduction specified.

6.2 If a limiting percentage rather than a specified percentage is desired, successive changes of 10 % standard resin reduction, based on the nonvolatile matter content of the varnish, shall be made until a limiting percentage is found. For example, varnishes may be reported as passing 40 % standard resin reduction and breaking at 50 %.

METHOD B—RUN-KAURI REDUCTION

7. Preparation of Standard Run-Kauri Solution

7.1 Weigh into a side-tube type distillation flask of suitable size an amount of clear, hard, bright pieces of "No. 1

Grade" kauri gum, equivalent to approximately one third of the volumetric capacity of the flask. Attach the flask to a water-cooled condenser. Carefully melt and distill the gum, collecting the distillate in a tared receiver. Stop the distillation when 25 % by weight of the gum has been collected in the tared receiver. A thermometer so placed in the distilling flask that its bulb is level with the discharging end of the flask should read about 600 F (316 C) at this point. Pour the residue in the distillation flask into a clean pan, and when cool break it up into small pieces.

7.2 Place in a tared beaker a given weight of the small pieces of the run-kauri gum, which shall be free from all dust or powder, together with twice its weight of freshly distilled gum spirits of turpentine conforming to ASTM Specification D 13, for Spirits of Turpentine,⁴ using only that portion distilling between 307 and 338 F (153 and 170 C). Dissolve the gum in the turpentine by heating the contents of the beaker slowly to a temperature of 302 F (150 C). The contents of the beaker should be stirred occasionally to prevent the sootened gum from adhering to the bottom of the beaker, thus avoiding possible over-heating of the gum and facilitating its solution. Allow the solution to cool to between 167 and 212 F (75 and 100 C) and filter through a fluted filter paper into a previously weighed, clean, brown glass bottle equipped with a screw cap.

7.3 *Examination of Run-Kauri Solution for Solvent Compatibility*—To 5 ml of the run-kauri solution in a clean, clear glass tube add 5 ml of mineral spirits conforming to ASTM Specification D 235, for Petroleum Spirits (Mineral Spirits),⁴ and in addition having an aniline point of between 55 and 60 C determined in accordance with ASTM Method D 611, Test for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents.⁶ Mix thoroughly and allow to stand for 1 h at room temperature, between 70 and 90 F (21 and 32 C). At the end of 1 h the mixture shall be completely miscible and show no clouding, precipitation, or separation. If clouding, precipitation, or separation occurs, the run-kauri solution shall be considered unsatisfactory for use.

7.4 *Adjustment of Run-Kauri Solution to Proper Nonvolatile Matter Content*—Transfer 10 to 15 ml of the filtered run-kauri solution to a stoppered flask or weighing pipet and determine the percentage of nonvolatile matter as described in Methods D 1644, with the following modifications: Add approximately 2 g of raw castor oil, accurately weighed, to each 80-mm dish or lid before adding the run-kauri solution and heat for 3 h at 115 ± 2 C. At least three determinations shall be made, using samples of run-kauri solution weighing 1.2 ± 0.1 g, and at least two blanks shall be run on the castor oil alone under the same conditions to determine its loss in weight. Calculate the nonvolatile matter content of the run-kauri solution, making allowance for the loss in weight of the raw castor oil. From the average nonvolatile matter content thus determined and the weight of the run-kauri solution, calculate the amount of freshly distilled gum spirits of turpentine required to adjust the nonvolatile matter content of the run-kauri solution to 33.3 weight %. After the

⁶ Annual Book of ASTM Standards, Vol 05.01 and 06.03.

⁵ Annual Book of ASTM Standards, Vol 06.01.

addition of the required amount of the turpentine, repeat the nonvolatile matter determination and record the nonvolatile matter content, which shall be $33.3 \pm 0.1\%$.

8. Procedure

8.1 Using test panels prepared as in Section 4, and run-kauri solution as in Section 7, follow the procedure described in Section 5.

9. Report

9.1 Report as directed in Section 6, expressing the results in terms of run-kauri reduction instead of standard resin reduction.

METHOD C—ADDITION OF LINSEED OIL

10. Scope

10.1 This method is applicable to varnishes that are less elastic or tough than zero standard resin reduction. It is identical with that described in Method A, except that the $33\frac{1}{2}\%$ solution of standard resin in hi-flash naphtha is replaced by a $66\frac{2}{3}\%$ solution of heat-bodied linseed oil for proportionately increasing the elasticity of the varnish under test.

11. Preparation of Standard Bodied Oil Solution

11.1 Heat a high grade of alkali-refined linseed oil having an acid number less than 1.0 in an open kettle at a temperature of 300 ± 5 C until the viscosity of the oil after cooling is between 6 and 10 St (0.0006 and 0.0010 m²/s) at 77 F (25 C).

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11.2 Standardize a quantity of the heat-bodied linseed oil by reducing it with one half its weight of pure redistilled turpentine conforming to Specification D 13,⁴ using only that portion of the turpentine distilling between 307 and 338 F (153 and 170 C).

12. Procedure

12.1 Prepare the test panels and perform the addition of the standard bodied oil solution to the varnish and the flowing on, baking, and bending of the test panel exactly as described in Section 5.

13. Report

13.1 Report the minimum percentage of the oil solution that must be added to the varnish, based on its nonvolatile matter content, so that the final mixture when flowed on a test panel and baked does not crack on the subsequent bending over a 3-mm rod.

14. Precision

14.1 The precision of this method is limited by the character of the testing ingredients and procedures and the interpretation and visual observation of the results. These results are not specifically numerically oriented and, therefore, comparison with standards of control materials known to have acceptable properties, determine the precision.



Standard Test Methods for Gas Checking and Draft Test of Varnish Films¹

This standard is issued under the fixed designation D 1643; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of the gas checking and draft test of varnish films.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature²

GAS CHECKING

3. Scope

3.1 This test method³ covers two alternative procedures for detecting nonuniform appearance that may develop while a film of varnish dries in an atmosphere that has been fouled by products of combustion. The nonuniform appearance is generally related to wrinkling, which is sometimes microscopic and which is variously described as checking, crow's-footing, crystallization, or dulling. Appearance non-uniformities that develop on dried films and are described by such terms as orange-peel, haze, silking, flooding, etc., are not within the scope of this test method.

4. Summary of Test Method

4.1 The varnish film under test is exposed to foul gas for various periods before the film has been converted from liquid to substantially solid, since the foul gas is effective, within the scope of these test methods, only before the varnish film has become set-to-touch or dust-free.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and the direct Responsibility of Subcommittee D 01.33 on Varnish and Resins, Including Shellac.

Current edition approved Sept. 19, 1960. Published November 1960. Originally published as D 1643 - 59 T. Last previous editions D 1643 - 59 T. Replaces Sections 2 to 14 of Method D 154.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ See *Paint Testing Manual*, ASTM STP 500, ASTM, 1972.

PROCEDURE A—BELL JAR TEST

5. Apparatus

5.1 The form and arrangement of the apparatus shall be designed to provide an even distribution of the products of combustion over the test panels.

5.1.1 *Glass Bell Jar*, approximately 8 in. (203 mm) in diameter and 12 in. (300 mm) in height, inside dimensions, having a ground-glass rim.

5.1.2 *Ground-Glass Base Plate*, of suitable size.

5.1.3 *Metal Frame*, fitted inside the bell jar and provided with a suitable support for holding a tin-plate disk 6 in. (152 mm) in diameter in a horizontal position 2 in. (51 mm) above the wick of the lamp.

5.1.4 *Additional Horizontal Supports*, for holding the various varnished panels under test.

5.1.5 *Small Kerosine Glow Lamp*, without a chimney, or a small alcohol lamp filled with kerosine, using a round wick not over 1/4 in. (6.4 mm) in diameter and adjusted to give a flame of 1/2 in. (20 mm) in height.

5.1.6 *Tin-Plate Panels*, 3 by 5 in. (76 by 127 mm) or smaller, cut from commercial No. 31 gage (0.227 mm) bright tin plate, weighing 0.39 to 0.51 lb/ft² (1.9 to 2.5 kg/m²) and carefully cleaned with toluene and dried before use.

6. Procedure

6.1 Determine the normal set-to-touch or dust-free drying time of the varnish under test in accordance with Test Methods D 1640. Divide this time by five to arrive at the different drying periods at which the material is to be tested. Thus, if a material sets to touch in 1 h, test the specimens for resistance to gas at drying periods of 12, 24, 36, and 48 min, respectively.

6.2 Flow the varnish to be tested on the tin-plate panels at the determined intervals of time. Allow the panels to drain in a nearly vertical position at room temperature 65 to 75°F (18 to 24°C).

6.3 After the required interval of drying time has passed, place the panels in a horizontal position on the upper supports of the frame. Light the lamp and place it on or near the center of the ground-glass base plate. After the bell jar has been placed tightly over the frame and lamp, the flame should extinguish in about 4 min.

6.4 Remove the panels and examine for gas effects after they have remained in the jar for 30 min.

7. Report

7.1 Describe the presence or absence of gas effects on the test panel for each drying interval.

Diameter of Dish	Weight of Nonvolatile Residue	
	45 mm	100 mm
For a 2.5-mil varnish film	0.12 g	0.60 g
For a 5-mil varnish film	0.25 g	1.20 g

8.2 Place the dish on the hot plate at $150 \pm 3.5^\circ\text{C}$ for 10 min. Cool to room temperature in a desiccator, then reweigh.

9. Calculation

9.1 Calculate the nonvolatile matter as described in Section 5.

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10. Report

10.1 Report the nonvolatile matter of the sample to the nearest 0.1 %.

INDEX TERMS

11. Index Terms

11.1 These test methods are indexed under the following terms: nonvolatile matter content; varnishes.

1. Scope

1.1 Test methods for determining the nonvolatile matter content of all types of varnishes, including those used in the paint and coating industry.

1.2 These test methods are applicable to the determination of nonvolatile matter content of varnishes, including those used in the paint and coating industry.

2. Referenced Standards

2.1 ASTM D 609 - Standard Test Method for Determining the Nonvolatile Matter Content of Paints by Gravimetric Analysis
2.2 ASTM D 1193 - Standard Test Method for Determining the Nonvolatile Matter Content of Paints by Gravimetric Analysis
2.3 ASTM D 3924 - Standard Test Method for Determining the Nonvolatile Matter Content of Paints by Gravimetric Analysis

3. Summary of Test Method

3.1 The test method involves the weighing of a known amount of varnish onto a tin can, followed by immersion in a water bath at $150 \pm 3.5^\circ\text{C}$ for 10 minutes. The varnish is then removed from the water bath and allowed to cool to room temperature. The weight of the nonvolatile matter is determined by weighing the residue left in the tin can.

4. Significance and Use

4.1 This test method is used to determine the nonvolatile matter content of varnishes, which is a measure of the amount of solid material in the varnish. This information is used to compare the nonvolatile matter content of different varnishes and to determine the nonvolatile matter content of a varnish for use in a specific application.

5. Reagents and Materials

5.1 The test method requires the use of a tin can, a water bath, and a balance.

¹ These test methods are published in the Annual Book of ASTM Standards, Vol. 11.01, Paint and Related Coatings, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.
² Annual Book of ASTM Standards, Vol. 11.01, Paint and Related Coatings, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.
³ Annual Book of ASTM Standards, Vol. 11.01, Paint and Related Coatings, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.



Standard Test Methods for Resistance of Dried Films of Varnishes to Water and Alkali¹

This standard is issued under the fixed designation D 1647; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of the resistance of dried varnish films to immersion in water and dilute alkali at room temperature.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

D 1193 Specification for Reagent Water³

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials²

3. Summary of Test Methods

3.1 *Test Method A*—The material under test is flowed onto tinplate panels and dried for 48 h. The panels are then immersed to half their length in reagent water for 18 h, or other agreed upon time, removed, and examined visually.

3.2 *Test Method B*—The material under test is applied to glass test tubes by dipping and dried for 72 h. The tubes are then suspended in dilute sodium hydroxide for periods ranging from 1 to 24 h, removed, rinsed, and after drying for 30 min, examined visually.

4. Significance and Use

4.1 Dried Varnish Films are a source of primary protection for surfaces. Exposure to water and dilute alkali solutions are two factors which tend to break down this protective coating. This test method can be used as a comparison basis between manufacturer and consumer to determine the ability of the varnish to resist water and dilute alkali.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended

that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

TEST METHOD A—WATER RESISTANCE OF DRIED FILMS

6. Apparatus and Materials

6.1 *Beaker*, glass, 600 mL or larger.

6.2 *Tinplate Panels*, 3 by 5 in. (75 by 125 mm) cut from commercial No. 31 gage (0.225-mm) bright tin plate, weighing 0.4 to 0.5 lb/ft² (1.90 to 2.50 kg/m²) and carefully cleaned and dried before use in accordance with Methods B or C of Methods D 609.

7. Procedure

7.1 Flow the varnish onto the tin panels, allow to drain in a nearly vertical position, and dry for 48 h in the standard atmosphere described in Specification D 3924.

7.2 Place the panels in a beaker containing about 2.5 in. (65 mm) of water at room temperature, immersing the ends that were uppermost during drying, and allow to remain in the water for 18 h, or other suitable period as agreed upon between the purchaser and the seller.

7.3 Remove the panels from the water, wipe carefully, and allow to dry at room temperature. Note the time required for whitening, if any, to disappear. Blooming, which sometimes occurs on immersion, is considered a type of whitening.

8. Report

8.1 Report the results of the water test as follows:

8.1.1 Not visibly affected,

8.1.2 Whitening disappears within 20 min,

8.1.3 Whitening does not disappear within 20 min, but disappears within 2 h,

8.1.4 Whitening does not disappear within 2 h, but disappears within 24 h, or

8.1.5 Whitening does not disappear within 24 h.

TEST METHOD B—DILUTE ALKALI RESISTANCE OF DRIED FILMS

9. Reagent

9.1 *Sodium Hydroxide Solution* (30 g/L)—Dissolve 30 g

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved April 28, 1989. Published June 1989. Originally published as D 1647 - 59. Last previous edition D 1647 - 83.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

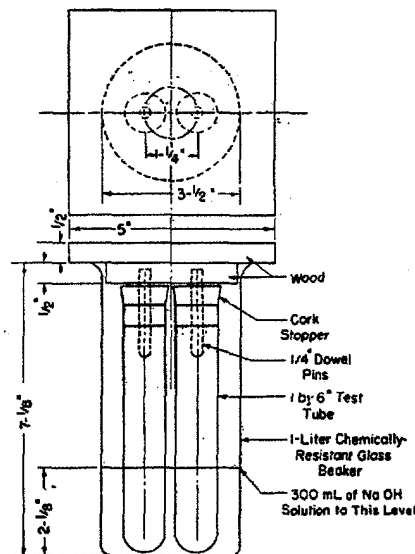
of sodium hydroxide (NaOH) in water and dilute to 1 L.

10. Procedure

10.1 Thoroughly clean and dry twenty 1 by 6-in. (25 by 150-mm) test tubes in toluene. Dip the tubes into the varnish under test, remove immediately, invert the tubes, and allow the varnish to dry for 72 ± 1 h in the standard atmosphere described in Specification D 3924. A suggested test schedule to fit into normal working hours is shown in Table 1.

10.2 Into each of ten 1000-mL, tall-form lipless, chemically resistant plastic or glass beakers, place 300 mL of the NaOH solution. Suspend a set of two varnish-coated tubes in each beaker so that the tubes do not touch the bottom or sides of the beaker and are immersed for a distance of approximately 2 in. (50 mm). As a suspending device (see Fig. 1), use a wooden cover plate and two dowels, and two one-hole cork stoppers, the dowels and the corks fitting into the tubes and the cover plate fitting the beaker as tightly as possible. Maintain the NaOH solution at a temperature of $23 \pm 2^\circ\text{C}$.

10.3 Remove a set of two varnish-coated tubes after immersion for each of the following time periods: 1, 2, 3, 4, 5, 6, 7, 8, 16, and 24 h. Rinse the tubes under a gentle stream of water, allow to air-dry for 30 min, and examine for film



NOTE—1 in. = 25.4 mm.

FIG. 1 Apparatus for Alkali Resistance Test

TABLE 1 Suggested Test Schedule

Day	Hour	Operation
Monday	8:30 a.m.	coat 16 tubes
	4:00 p.m.	coat 4 tubes
Thursday		dry tubes for 72 h
	8:30 a.m.	start alkali immersion on first 16 tubes
	9:30 a.m. and each hour thereafter to 4:30 p.m.	remove one set of tubes from alkali, rinse, and air-dry for 30 min
	10:00 a.m. and each hour thereafter to 5:00 p.m.	examine varnish film on set of tubes removed 1/2 h earlier
Friday	4:00 p.m.	start alkali immersion on last 4 tubes
	8:00 a.m.	remove one set of tubes from alkali, rinse, and air dry for 30 min
	8:30 a.m.	examine varnish film on set of tubes removed and rinsed 1/2 h earlier
	4:00 p.m.	remove last set of tubes, rinse, and air-dry for 30 min
	4:30 p.m.	examine varnish film on last set of tubes

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

whitening, blistering, or removal. The end point of the test is the number of hours immersion at which the first signs of film whitening, blistering, or removal are noted on the vertical sides of the tubes or on the spherical bottom portion of the tube. The results may be compared with those from materials known to give acceptable performance. For referee work, the test on materials known to be acceptable should be made simultaneously.

11. Report

11.1 Report the results of the alkali test as follows:

11.1.1 Type of varnish, and

11.1.2 End point of the test, h.

12. Precision

12.1 Precision has not been determined due to the multiplicity of ambient test conditions.



Standard Test Methods for Water Vapor Transmission of Organic Coating Films¹

This standard is issued under the fixed designation D 1653; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace Method 6171 of Federal Test Method Standard No. 141A. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the determination of the rate at which water vapor passes through films of paint, varnish, lacquer, and other organic coatings. The films may be free films or they may be applied to porous substrates.

1.2 Two test methods are covered as follows:

1.2.1 *Test Method A—Dry Cup Method*, and

1.2.2 *Test Method B—Wet Cup Method*.

1.2.3 Agreement should not be expected between results obtained by different methods. The method that most closely approaches the conditions of use should be selected.

1.3 The values stated in inch-pound units are to be designated as the standard. Metric-inch pound conversion factors are stated in 13.2.1.2 and 13.2.2.2.

1.4 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 823 Test Method for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²

D 1193 Specification for Reagent Water³

D 4708 Practice for Preparation of Free Films of Organic Coatings²

E 104 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions⁴

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *water vapor transmission rate, WVT*—the steady water vapor flow in unit time through unit area of a body, between two specific parallel surfaces, under specific conditions of temperature and humidity at each surface. Accepted

inch-pound unit is grains per square foot per hour. Accepted SI unit is grams per square metre per 24 h.

3.1.2 *water vapor permeance, WVP*—the steady water vapor flow in unit time through unit area of a body (WVT) induced by unit vapor pressure difference (Δp) between the two surfaces of a coating. Therefore, $WVP = WVT/\Delta p$. Accepted inch-pound unit is grains per square foot per hour per inch of mercury (called a perm). Accepted SI unit is grams per square metre per 24 h per millimetre of mercury (called a metric perm).

4. Summary of Test Methods

4.1 In Test Method A (Dry Cup Method), the test specimen is sealed to the open mouth of a cup or dish containing desiccant, and the assembly placed in a test chamber with a controlled atmosphere. Two sets of exposure conditions are acceptable for this test method.

4.1.1 *Condition A*, consisting of 50 % relative humidity at 73°F (23°C), and

4.1.2 *Condition B*, consisting of 90 % relative humidity at 100°F (38°C).

4.2 In Test Method B (Wet Cup Method), the test specimen is sealed to the open mouth of a cup or dish containing water, and the assembly placed in a test chamber with a controlled atmosphere. Two sets of exposure conditions are acceptable for this test method:

4.2.1 *Condition A*, consisting of 50 % relative humidity at 73°F (23°C), and

4.2.2 *Condition C*, consisting of very low (near zero) relative humidity at 73°F (23°C).

4.3 In both methods, periodic weighings of the cup or dish are made to determine the rate of water vapor movement through the specimen.

5. Significance and Use

5.1 One of the factors affecting the performance provided by an organic coating is the degree of resistance to the passage of water vapor. Hence, the water vapor transmission characteristics of coatings are important in assessing their performance in practical use.

5.2 The purpose of these test methods is to obtain values of water vapor transfer through coatings that range in permeability from high to low. These values are for use in design, manufacture, and marketing.

5.3 The water vapor transmission is not a linear function of film thickness, temperature or relative humidity.

5.4 Values of water vapor transmission rate (WVT) and water vapor permeance (WVP) can be used in the relative

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 1653 - 59 T. Last previous edition D 1653 - 91.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁴ Annual Book of ASTM Standards, Vol 11.03.

rating of coatings only if the coatings are tested under the same closely controlled conditions of temperature and relative humidity, and if their thicknesses are equal.

6. Apparatus

6.1 *Perm Cup⁵ or Dish*, consisting of a container made of a noncorroding material, impermeable to water or water vapor. If the cup or dish is made of aluminum, it must be anodized or given a protective clear coating to prevent corrosion.

6.1.1 One type of cup that is suitable has a flanged edge and is equipped with a separate corresponding flange, so that the test specimen can be held between them. The contacting faces of the flanges shall be ground to such flatness that when the film is in position, moisture transfer can occur only through the exposed film area. For hard films, or films having a very rough surface, a soft rubber gasket may be inserted between the film and the flange. The flanges shall then be held together with suitable clamps.

6.1.2 Another suitable cup is any open circular dish to which the test film can be sealed with wax or sealant.

6.2 *Test Chamber*, with a controlled temperature and relative humidity as specified in Section 4. Air shall be circulated throughout the chamber to maintain uniform conditions at all test locations. For low or high humidity conditions, a standard desiccator or other suitable cabinet may be used. For maintaining constant relative humidity by means of aqueous solutions, refer to procedures outlined in Practice E 104.

6.3 *Analytical Balance*, having an adequate capacity for the weight of the test cups and a sensitivity of 1 mg.

7. Reagents and Materials

7.1 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type IV of Specification D-1193.

7.2 *Desiccant*, consisting of either anhydrous calcium chloride (CaCl_2) or anhydrous magnesium perchlorate ($\text{Mg}(\text{ClO}_4)_2$). The calcium chloride should be dried at 400°F (200°C) before use. If calcium chloride will react with the test specimen, an adsorbing desiccant such as a silica gel may be used but the moisture gain by this desiccant during the test must be limited to 4 %. Use caution in handling magnesium perchlorate because of possible chemical reaction that may be produced if it comes in contact with some organic materials.

7.3 *Sealant*, such as wax for attaching the test specimen to the top of the perm cup or dish. It must be highly resistant to the passage of water vapor. It must not lose weight to, or gain weight from, the atmosphere in an amount, over the required period of time, that would affect the test results by more than 2 %. It must not affect the vapor pressure in a water-filled dish.

NOTE 1—Among acceptable sealants are (1) a 60:40 mixture of microcrystalline wax and refined crystalline paraffin wax, (2) tissue embedding wax, and (3) a 50:50 mixture of beeswax and rosin.

⁵ Perm Cups, 10 cm² and 25 cm² in area, available from Paul N. Gardner Co., Inc., 316 N.E. First Street, Pompano Beach, FL 33061, and BYK/Gardner, Inc., Gardner Laboratory, 2435 Linden Ln., Silver Spring, MD 20910 have been found satisfactory for this purpose.

7.4 *Release Paper*, prepare free films, whenever possible to eliminate the potential interference of substrates, on release paper⁶ or glass substrates following procedures outlined in Practice D 4708.

7.5 *Film Support*, for preparing films that are too brittle or otherwise unsatisfactory for handling as free films, support materials such as paper charts,⁷ filter paper, and glass cloth have been found satisfactory in some instances. Such support can have an effect on the test results.

8. Test Specimens

8.1 It is very important that the test specimens be smooth, and completely continuous films of uniform thickness throughout the test area. Apply air dry coatings to substrates using one of the methods described in Test Methods D 823 or Practice D 4708.

NOTE 2—Special test conditions may require that the coating be applied by brushing, roller coating or other special methods. The thickness of the coating applied shall be within normal range for the type of material under test, and shall not vary by more than 5 % of the total thickness in any test series.

NOTE 3—When applying bake finishes, bake for specified time and temperature in accordance with manufacturers' instructions. Permeance may vary with the baking schedule or the time of air drying.

8.2 Air dry the coated material in a horizontal position for 7 days in a room preferably maintained at 73.5 ± 3.5°F (23 ± 2°C) and 50 ± 5 % relative humidity. If the material to be used is a free film, remove it from the substrate and allow the previously unexposed surface to dry for an additional 7 days. The drying schedule may be modified as recommended by manufacturer.

NOTE 4—Examine test film for the presence of pinholes or other defects before commencing the test. It may be advisable to apply the test material in two coats, applying the second coat perpendicular to the first coat and allowing for an adequate dry period between coats to achieve the desired film thickness.

8.3 Measure the thickness of the test specimen in several places with a micrometer using procedures in Test Methods D 1005 to ensure a uniform thickness.

8.4 When coatings are applied to support materials, the coated side of the test specimen should be placed away from the desiccant or towards the water in the cup, depending upon use. Coatings should not be applied to both sides of a support. Glass cloth is preferred for coatings that are cured by baking.

TEST METHOD A—DRY CUP METHOD

9. Test Conditions

9.1 Unless other conditions are agreed upon between the purchaser and the seller, the tests shall be performed under one or more of the following conditions:

9.1.1 *Condition A*—Test chamber or cabinet maintained at 73° ± 1°F (23 ± 0.6°C) and 50 ± 2 % relative humidity.

9.1.2 *Condition B*—Test chamber or desiccator maintained at 100 ± 1°F (38 ± 0.6°C) and 90 ± 2 % relative humidity.

⁶ Release Paper, Form RP-1K, available from the Leneta Co., P.O. Box 110, Ho-Ho-Kus, NJ 07423, has been found satisfactory for this purpose.

⁷ Paper charts, Form NWK, available from the Leneta Co., have been found suitable for this purpose.

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10. Procedure

10.1 Prepare at least three perm cups or dishes for each test material as follows:

10.1.1 Fill the cups with desiccant to within 1/4 in. (6 mm) of the top edge.

10.1.2 If the cups are equipped with flanges, place the test specimen between the flanges and adjust the clamps to hold it firmly in position.

10.1.3 If the cups are not equipped with flanges, seal the test specimen to the top edge of the cups with wax as follows:

10.1.3.1 First carefully place the test specimen, cut to the size of the cup, on a thin cardboard ring soaked in molten wax.

10.1.3.2 Then place the specimen-covered frame over the mouth of the cup. Thoroughly seal the frame to the edge of the cup at the temperature to be used in the test. If the coating is on a substrate or support, place the coated side away from the desiccant in the cup.

NOTE 5—Imperfections in the film that are not readily visible will produce inconsistent results; consequently make sure that the test is always run in triplicate.

10.4 Weigh the loaded cups to 1 mg and place them in the test cabinets or desiccators. Record time, temperature and relative humidity.

10.5 Remove the cups for periodic weighing to determine weight gain. Coatings expected to have high WVT ratings, over 10 perms, may require weighings more frequently than once a day.

10.6 In general, weigh the cups every 24 h for a period of 3 weeks, or until the weight change has become constant. Record the time that weighings are taken to the precision of approximately 1 % of the time span between weighings. Thus, if weighings are made every day, a time to the nearest 15 minutes would be allowed.

10.7 Shake cups containing desiccant everyday to prevent surface saturation of the material. If moisture absorbed by the desiccant exceeds 20 % of the desiccant weight, discontinue the test.

10.8 Return the cups to the test chamber immediately after weighing.

TEST METHOD B—WET CUP METHOD

11. Test Conditions

11.1 Unless other conditions are agreed upon between the purchaser and the seller, perform the test(s) under one or more of the following conditions:

11.1.1 *Condition A*—Test chamber or cabinet maintained at 73 ± 1°F (23 ± 0.6°C) and 50 ± 2 % relative humidity.

11.1.2 *Condition C*—Test chamber or desiccator maintained at 73 ± 1°F and very low (near zero) relative humidity.

12. Procedure

12.1 Prepare at least three cups for each test material as follows:

12.1.1 Fill the cups with water to within 1/4 in. (6 mm) of the top edge.

12.1.2 If the cups are equipped with flanges, place the test specimen over the opening of the cups between the flanges and adjust the clamps to hold them firmly in position. If the

coating is on a substrate or support, place the coated side towards the water in the cups.

12.1.3 If the cups are not equipped with flanges, seal the test specimens to the top edge of the cups with wax in accordance with procedures in 10.1.3.1 and 10.1.3.2.

12.2 Weigh the loaded cups to 1 mg and place them in the test chamber. Record time, temperature and relative humidity. Follow procedures outlined in 10.5 and 10.6.

12.3 Return the cups to the test chamber immediately after weighings.

13. Calculations

13.1 For each material tested, plot the weight change against elapsed time. When a straight line adequately fits the plot of at least four properly spaced points, a nominally steady state exists and the slope of the straight line is the rate of water vapor transmission.

13.2 Calculate one or more of the following, depending on the water vapor transmission characteristics to be determined:

13.2.1 Calculate the water vapor transmission rate, WVT:

13.2.1.1 In inch-pound units as follows:

$$WVT = (G/t)/A = \text{grains per ft}^2 \text{ per 1 h} \quad (1)$$

where:

G = weight change, grains (from the straight line),

t = time during which G occurred, h, and

A = test area, ft²,

1 g = 15.43 grains.

13.2.1.2 In metric units as follows:

$$WVT = (G/t)/A = \text{grams per m}^2 \text{ per 24 h} \quad (2)$$

where:

G = weight change, g (from the straight line),

t = time during which G occurred, h, and

A = test area, m².

13.2.2 Calculate the permeance, WVP:

13.2.2.1 In inch-pound units as follows:

$$WVP = WVT/\Delta p = \text{grains per ft}^2 \text{ per 1 h per in. of mercury (perms)} \quad (3)$$

where:

$\Delta p = S(R_1 - R_2)$,

S = in. Hg (saturation vapor pressure at test temperature),

(see Table 1),

R_1 = relative humidity at vapor source, and

R_2 = relative humidity at vapor sink.

13.2.2.2 In metric units as follows:

$$WVP = WVT/\Delta p = \text{grams per m}^2 \text{ per 24 h per millimetre of mercury, (metric perms)} \quad (4)$$

where:

$\Delta p = S(R_1 - R_2)$,

S = mm Hg (saturation vapor pressure at test temperature), (see Table 1),

R_1 = relative humidity at vapor source, and

R_2 = relative humidity at vapor sink.

14. Report

14.1 Report the following information:

14.1.1 Method of coating application and curing procedure used.

TABLE 1 Saturation Vapor Pressure

Temperature		Pressure	
°F	°C	in. Hg	mm Hg
72.3	22.4	0.800	20.316
72.5	22.5	0.805	20.440
72.7	22.6	0.810	20.565
72.9	22.7	0.816	20.690
73.1	22.8	0.819	20.815
73.3	22.9	0.824	20.941
73.4	23.0	0.829	21.068
73.6	23.1	0.834	21.196
73.8	23.2	0.840	21.324
74.0	23.3	0.845	21.453
74.2	23.4	0.850	21.583
74.4	23.5	0.855	21.714
74.5	23.6	0.860	21.845
99.3	37.4	1.894	48.102
99.5	37.5	1.904	48.364
99.7	37.6	1.914	48.627
99.9	37.7	1.925	48.891
100.1	37.8	1.935	49.157
100.2	37.9	1.946	49.424
100.4	38.0	1.956	49.692
100.6	38.1	1.967	49.961
100.8	38.2	1.978	50.231
101.0	38.3	1.988	50.502
101.1	38.4	1.999	50.774
101.3	38.5	2.010	51.048
101.5	38.6	2.021	51.323

14.1.2 Mean film thickness of the test specimens for each material.

14.1.3 Type of film support used, if any.

14.1.4 Method used (Test Method A (Dry Cup) or Test Method B (Wet Cup)).

14.1.5 Test temperature and relative humidity in the test chamber.

14.1.6 Computed rate of water vapor transmission (WVT), either in inch-pound or in metric units.

14.1.7 The computed permeance in terms of both perms and metric units.

15. Precision and Bias⁸

15.1 In an interlaboratory study of these test methods in which one operator in each of three laboratories determined in triplicate the permeance of three coatings covering the range of interest for vapor barrier coatings, the interlaboratory standard deviation for the dry-cup procedure was found to be 0.073 perms with 6 df and the interlaboratory wet-cup coefficient of variation 21.54 % relative with 6 df, with no results having been discarded. Based upon these values, the following criteria should be used for judging, at the 95 % confidence level, the acceptability of results:

15.1.1 Repeatability:

15.1.1.1 *Test Method A*—Two results, each the mean of triplicate runs, obtained by the same operator should be considered suspect if they differ by more than 0.25 perms at dry-cup permeance of less than 1.0 perms.

15.1.1.2 *Test Method B*—Two results, each the mean of triplicate runs, obtained by the same operator should be considered suspect if they differ by more than 74.2 % relative at wet-cup permeances of 5 to 30 perms.

15.1.2 *Reproducibility*—Intralaboratory precision is normally better than interlaboratory precision. However, one participant in this exchange has noted relatively large differences when the same coating was tested in quadruplicate with fairly long intervals between tests.

15.2 Bias is not applicable to these test methods.

16. Keywords

16.1 permeability; permeance; perms; water vapor permeance; water vapor transmission

⁸ Supporting data are available from ASTM Headquarters. Request RR:D1 - 1064.

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Standard Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments¹

This standard is issued under the fixed designation D 1654; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Editorial changes were made throughout in July 1984.

1. Scope

1.1 This method covers the treatment of previously painted or coated specimens for accelerated and atmospheric exposure tests and their subsequent evaluation in respect to corrosion, blistering associated with corrosion, loss of adhesion at a scribe mark, or other film failure.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

B 117 Method of Salt Spray (Fog) Testing²

B 287 Method of Acetic Acid-Salt Spray (Fog) Testing³

D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces⁴

D 714 Test Method for Evaluating Degree of Blistering of Paints⁴

D 1014 Test Method for Conducting Exterior Exposure Tests of Paints on Steel⁴

D 2803 Test Method for Filiform Corrosion Resistance of Organic Coatings on Metal⁴

3. Significance and Use

3.1 This method provides a means of evaluating and comparing basic corrosion performance of the substrate, pretreatment, or coating system, or combination thereof, after exposure to corrosive environments.

4. Apparatus

4.1 **Scribing Tool**—A straight-shank tungsten carbide tip, lathe cutting tool (Brazed tool, Style E, with 1/64-in. nose radius) is recommended; however, other styles may be used

provided they are ground to a single point having an included angle of $60 \pm 15^\circ$. Any other type of scribing instrument such as a scalpel, razor blade, knife, or sharp pointed tool is unacceptable unless agreed upon between the producer and the user.

4.2 **Straightedge**—Any straightedge of sufficient length and rigidity to guide the scribing tool in a straight line.

4.3 **Air Compressor**—A source of compressed air capable of producing 80 psi (550 kPa) open line pressure.

4.4 **Air Gun**—An air dusting gun and nozzle combination⁵ to meet the following specification:

Air Consumption, ft ³ /min (m ³ /min)	Pressure, psi (kPa)	Nozzle Diameter, in. (mm)
8.4 (0.24)	80 (550)	0.12 (3.0)

4.4.1 A guard consisting of barriers, baffles, or screens is required to protect the operator and other individuals near the area where the air is being used. The guard must be placed between the air nozzle and the operator. A device such as a sand-blasting cabinet has been found to be acceptable.

NOTE 1—The use of an air gun without a guard is in violation of the U. S. Occupational Safety and Health Administration regulation.

4.5 **Scraping Tool**—A rigid metal spatula, dull knife, or similar instrument.

4.6 **Scale**—Any rule with 1-mm divisions.

4.7 **Grid**, plastic or wire with 1/2-in. (13-mm) squares necessary to permit measurements of the required accuracy.

5. Preliminary Treatment of Test Specimens

5.1 Scribed Specimens:

5.1.1 Where specified or agreed upon, prepare each specimen for testing by scribing it in such a manner that the scribe can be exposed lengthwise when positioned in the test cabinet. This position will allow solution droplets to run lengthwise along the scribe.

5.1.2 Scribe the specimen by holding the tool at approximately a 45° angle to the surface. Position the tool so that only the carbide tip is in contact with the surface. Pull the scribing tool to obtain a uniform V-cut through the coating that is being tested. The scribe should be of sufficient length to cover the significant test area but should not contact the

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Tests For Protective Coatings.

Current edition approved Nov. 26 and Dec. 3, 1979. Published January 1980. Originally published as D 1654 - 59. Last previous edition D 1654 - 74.

² Annual Book of ASTM Standards, Vols 03.02 and 06.01.

³ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Spray gun and nozzles, Model No. 22-L and 0010 have been found satisfactory and may be obtained from Spray Systems Co. Equivalents may be used.

edge of the specimen. The scribe must penetrate all coatings on the metal, leaving a uniformly bright line free of burrs. Quality of the scribe may be observed with the aid of low-power magnification. Note, mark, and describe defects, coding, and flaws that may affect results.

5.1.3 Scribe lines other than those of a single, straight nature may be used if agreed upon between the producer and the user.

5.1.4 Expose scribed specimens in accordance with 6.1 and rate in accordance with Section 7.

5.2 *Unscribed Specimens*—Specimens coated with paint undercoats, oils, or waxes may be evaluated without a scribe. Expose such specimens in accordance with Section 6 and rate for corrosion of the general surface in accordance with Section 8.

6. Exposure of Test Specimens

6.1 Expose test specimens in accordance with Methods B 117, B 287, D 1014, D 2803, or any other applicable test method, as agreed upon between the producer and the user. The length of test and evaluation intervals should be agreed upon prior to exposure of specimens.

7. Procedure A—Evaluation of Scribed Specimens

7.1 *Method 1 (Air Blow-Off)*—Rinse each specimen after completion of the exposure period, using a gentle stream of water at a temperature up to 110°F (45°C). Holding the nozzle at approximately a 45° angle, blow along the entire scribe line, disturbing the surface adjacent to the scribe mechanically by the air nozzle to ensure an opening for the air blast. Complete the air blasting within 15 min of specimen removal from the exposure cabinet. If the air blasting cannot be completed within the prescribed time, immerse the specimens in water at room temperature or store in a plastic bag to avoid any drying effect.

7.2 *Method 2 (Scraping)*—Rinse the specimen after completion of the exposure period, using a gentle stream of water at a temperature up to 110°F (45°C). Scrape the specimen vigorously with an instrument described in 4.5 while under the gentle stream of the rinse water. Hold the scraper with its face perpendicular to the specimen surface and parallel to the scribe, moving it back and forth across the scribe to remove the coating that has been undercut and has suffered loss of adhesion only, not to remove the coating that still has adhesion. Complete the scraping within 15 min of specimen removal from the exposure cabinet. If scraping cannot be completed within the prescribed time, immerse the speci-

TABLE 1 Rating of Failure at Scribe (Procedure A)

Representative Mean Creepage From Scribe		
Millimetres	Inches (Approximate)	Rating Number
Over 0	0	10
Over 0 to 0.5	0 to 1/64	9
Over 0.5 to 1.0	1/64 to 1/32	8
Over 1.0 to 2.0	1/32 to 1/16	7
Over 2.0 to 3.0	1/16 to 1/8	6
Over 3.0 to 5.0	1/8 to 3/16	5
Over 5.0 to 7.0	3/16 to 1/4	4
Over 7.0 to 10.0	1/4 to 3/8	3
Over 10.0 to 13.0	3/8 to 1/2	2
Over 13.0 to 16.0	1/2 to 5/8	1
Over 16.0 to more	5/8 to more	0

TABLE 2 Rating of Unscribed Areas (Procedure B)

Area Failed, %	Rating Number
No failure	10
0 to 1	9
2 to 3	8
4 to 6	7
7 to 10	6
11 to 20	5
21 to 30	4
31 to 40	3
41 to 55	2
56 to 75	1
Over 75	0

mens in water at room temperature or store in a plastic bag to avoid any drying effect.

NOTE 2—Some specimens exposed to natural weathering do not require rinsing, air blasting, or scraping to evaluate corrosion. Alternative methods may be used if agreed upon between the producer and the user.

7.3 *Rating*—Rate the corrosion or loss of paint extending from a scribe mark as prescribed in Table 1. Record the representative mean, maximum, and minimum creepage from the scribe, and note whether or not the maximum is an isolated spot. Also, rate in accordance with Table 2 the prevalence of corrosion on areas removed from the scribe.

8. Procedure B—Evaluation of Unscribed Areas

8.1 Rinse the specimen after completion of the exposure period (Section 6), using a gentle stream of water at a temperature up to 100°F (40°C). Dry the surface of the specimen with paper towels or compressed air. Drying should be done in such a manner that the corrosion on the specimen surface is not disturbed.

8.2 Evaluate unscribed specimens for corrosion spots, blisters, and any other types of failure that may occur. Depending upon the contour of the specimen, the use of a grid, as described in 4.7, is recommended as an aid in evaluating this type failure (Fig. 1). The percent of surface failure, excluding rust staining or run down, can be estimated by counting the number of squares that have points of failure and relating this number to the total number of squares covering the test area. Discount corrosion within 1/2 in. (12.7 mm) of edges.

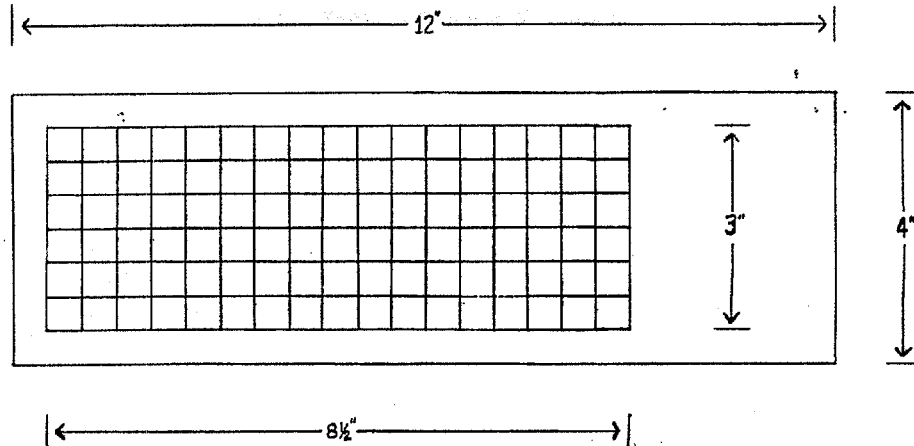
8.3 *Rating*—Convert percent failure to the rating number in accordance with Table 2. In some instances, the rating number may be used as a factor with the corresponding exposure time intervals to produce a performance index number.

NOTE 3—Formation of under-film corrosion may be evaluated and reported in accordance with Table 2 if the film is first carefully stripped with a neutral stripper. Exercise care to avoid alteration of the corrosion pattern or pretreatment.

NOTE 4—Where the character of the failure permits, the photographic blister standards given in Test Method D 714 may be used to describe the results of the exposure test, in respect to size of blisters or corroded areas, while Method D 610 may serve to describe the frequency and distribution, if desired.

9. Report

9.1 Report the ratings of the test specimens, the procedures, and, for scribed panels, the method followed. Also



NOTE—1 in. = 25.4 mm.

FIG. 1 Typical Grid with 1/2-in. (13-mm) Squares

report the exposure test to which the specimens were subjected.

10. Precision and Use

10.1 Since this is a method of evaluation based on measurements after various tests, the statement of precision

applicable to each specific method of exposure to corrosive atmospheres applies. The preferred methods of measurement, using ruled plastic sheets, are at least equal in precision to the various methods of exposing test specimens to corrosive environments.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting¹

This standard is issued under the fixed designation D 1730; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Section 2 was added and subsequent sections were renumbered editorially in September 1984.

1. Scope

1.1 These practices cover four types of treatment for preparation of aluminum and aluminum-alloy surfaces for painting, as follows:

1.1.1 Type A—Solvent Cleaning.

1.1.2 Type B—Chemical Treatments.

1.1.3 Type C—Anodic Treatments.

1.1.4 Type D—Mechanical Treatments. These four types cover a number of procedures, as described herein.

1.2 Variations in surface treatment produce end conditions which differ, and which do not necessarily yield identical results when paints are applied. Service conditions will dictate the type of surface preparation that should be selected, although the quality produced by any individual method may vary with different alloys.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

3. Type A—Solvent Cleaning

3.1 Solvent cleaning does not disturb the natural oxide film on the metal and may prove adequate for some applications, such as ambient indoor or very mild service conditions. Three methods may be employed, as follows:

3.1.1 *Method 1, Manual Swabbing or Dip-Washing*, with a solvent such as mineral spirits or high-flask solvent naphtha. With this method it is extremely difficult to prevent accumulation of contaminants on the swab or in the solvent. This method is only recommended when other treatments are impractical.

3.1.2 *Method 2, Solvent Spray Cleaning*, in accordance with Method A, Procedure 1 of Methods D 609.

3.1.3 *Method 3, Vapor Degreasing*, in special equipment employing trichloroethylene vapor, in accordance with Method A, Procedure 2 of Method D 609.

4. Type B—Chemical Treatments

NOTE 1—Materials and procedures employed in these methods of treatment are available from a number of sources as proprietary compounds or methods. Selection may be made from available sources.

4.1 *Method 1, Alkaline Cleaners*—Alkaline solutions, such as caustic soda, etch the metal, thus destroying the natural oxide film. They are followed by an acid treatment, preferably nitric acid or phosphoric acid. They shall not be used on assembled structures. Inhibited alkaline cleaners are sometimes employed as a pretreatment to remove grease and oil prior to an acid treatment. Inhibited alkaline cleaners do not etch the surface. They are not generally recommended unless followed by a conversion treatment, such as described in Methods 4, 5, 6, or 7.

4.2 *Method 2, Sulfuric Acid, Chromium Trioxide Etch*—This treatment provides a clean and uniform surface without undue etching, and is effective for removing oil and water stains and any film formed during heat-treatment. The etching solution is prepared by adding 1 gal (3.78 L) of concentrated sulfuric acid and 45 oz (1.28 kg) of chromium trioxide to 9 gal (34 L) of water. It is used at a temperature of 160 to 180°F (71 to 82°C) (depending on the alloy and the amount of film) for about 5 min and is followed by a water rinse. This treatment produces a passive surface suitable for painting under mild to intermediate exposure conditions and where clear finishes are to be applied.

4.3 *Method 3, Alcoholic Phosphoric Acid Cleaner*—This treatment involves the use of an aqueous solution of phosphoric acid (10 to 15 volume %) with alcohol or other organic solvents, together with wetting agents, emulsifying agents, etc. The solution may be applied by swabbing or dipping at room temperature (70 to 90°F (21 to 32°C)), and should be allowed to remain on the surface for several minutes, followed by thorough rinsing with clean water. A very thin phosphate film is formed which tends to protect the metal and promote paint adhesion under mild to intermediate exposure conditions.

NOTE 2—U.S. Military Specification MIL-M-10578B describes a treatment of this type.

4.4 *Method 4, Crystalline Phosphate Treatment*—This surface-coating method consists in reacting the aluminum surface in a zinc-acid-phosphate solution containing ox-

¹ These practices are under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings.

Current edition approved Sept. 8, 1967. Published November 1967. Originally published as D 1730-60. Last previous edition D 1730-66.

² *Annual Book of ASTM Standards*, Vol 06.01.

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dizing agents and other salts for accelerating the coating action. The aluminum surface is converted to a finely crystalline, phosphate coating of the proper texture adapted to inhibit corrosion and increase the adherence and durability of any applied paint film. It is recommended for product finishes. The phosphate coating process may be carried out by immersion or spray application. The aluminum surface is converted to the phosphate coating by immersion in the processing solution for 30 s to 4 min at 125 to 140°F (51 to 60°C), or by spraying the solution for 10 s to 2 min at 125 to 160°F (51 to 71°C).

NOTE 3—Before applying the treatments according to Methods 4, 5, 6, and 7 of Type B, the aluminum surfaces should be freed of grease, oil, or other foreign material by means of the procedure described in Method 3 of Type A, Method 1 of Type B, or any other suitable method.

NOTE 4—This treatment complies with the requirements of U.S. Federal Specification TT-C-490.

4.5 Method 5, Amorphous Phosphate Treatment—This process comprises treatment of clean aluminum surfaces in a warm (95 to 130°F (35 to 54°C)) aqueous solution comprising phosphoric, and hydrofluoric acids, chromium trioxide or a solution thereof. The aluminum surface is converted to a thin, adherent, amorphous coating, iridescent green to gray-green in color, depending upon the aluminum alloy treated, which inhibits corrosion and increases the adherence and durability of applied paint films. This method is recommended for use under the more severe conditions of service, and for product finishes. The coating process may be carried out by immersion or spray application. The time of treatment will vary from 15 to 45 s for the spray process, and from 30 s to 3 min for the immersion application (see Notes 3 and 5).

NOTE 5—Most of the treatments conforming to Methods 5, 6, and 7 of Type B comply with the requirements of U. S. Military Specification MIL-C-5541.

4.6 Method 6, Carbonate Chromate Treatment—This process comprises treatment in a hot (180 to 190°F (82 to 88°C)) dilute solution of sodium carbonate and potassium chromate for periods from 2 to 20 min, followed by a water rinse. The surface is then given a final treatment in hot 5% potassium dichromate solution, followed by a final rinse. The treatment produces a thin, adherent, conversion coating on the surface, increasing the corrosion resistance of the metal and promoting paint adhesion. This method is recommended for use under the more severe conditions of service and for product finishes (see Notes 3 and 5).

4.7 Method 7, Amorphous Chromate Treatment—This process comprises treatment of clean aluminum surfaces in aqueous chromium trioxide solutions containing suitable accelerating agents such as fluoride-containing materials. The aluminum surface is converted to an adherent, amorphous, mixed metallic oxide coating, iridescent golden to light-brown in color, which increases the corrosion-resistance and the adherence and durability of any applied paint film. This method is recommended for use under the more severe conditions of service and for product finishes. The coating process may be carried out by immersion, spray, or brush application, at room temperature (70 to 90°F (21 to 32°C)), in from 15 s to 5-min contact time (see Notes 3 and 5).

4.8 Method 8, Acid-Bound Resinous Treatment (see Note 6)—This surface treatment involves the use of a suitably

applied acid-bound resinous film of approximately 0.3 to 0.5 mils (7.6 to 12.7 μ m) thickness. The treatment is based on three primary components: a hydroxyl-containing resin; a pigment capable of reacting with the resin and an acid; and an acid capable of insolubilizing the resin by reacting with the resin, the pigment, and the metal substrate. The aluminum surface should be prepared by Methods 1, 2, or 3 of Type A (see Section 3) or chemical treatments, Type B, 4.1 or 4.2 prior to the application of this treatment. The film may be applied by brush, spray, or dip. Under normal conditions it should dry sufficiently for recoating within 30 min; and within 8 h it should not be softened by organic solvents commonly used in paint coatings. The film has good adhesion to the metal substrate and promotes good adhesion of most subsequent organic coatings to itself. This method is recommended for severe service conditions, particularly on fabricated structures, either in the shop or in the field.

NOTE 6—Materials meeting the requirements of U.S. Military Specification MIL-P-15328b may be used to apply Method 8 of Type B.

5. Type C—Anodic Treatments (see Note 7)

5.1 Anodic treatment in either sulfuric acid or chromium trioxide electrolyte will provide a protective and inert oxide coating which increases the corrosion-resistance of the metal and promotes paint adhesion. It is recommended where maximum corrosion-resistance by the treatment itself is desired. Anodic treatments should not be used on assemblies of dissimilar metals. The two procedures are as follows:

NOTE 7—Treatments conforming to Methods 1 and 2 of Type C comply with the requirements of U.S. Military Specification MIL-A-8625.

5.2 Method 1, Sulfuric Acid Anodic—This treatment consists in making the part the anode in a 15% sulfuric acid electrolyte (by weight) and applying current at a potential of about 16 to 18 V and a current density of 12 A/ft² until desired thickness of coating is obtained. After rinsing in cold water, the coating is sealed for 10 min by treatment in a boiling solution of 5% potassium dichromate. This method is not recommended where danger of acid entrapment is encountered.

5.3 Method 2, Chromium Trioxide Anodic—This treatment consists in making the part the anode in a 10 weight % chromium trioxide electrolyte at a temperature of 100°F (38°C) and applying a potential of 40 V for a period of 30 to 60 min. After first rinsing in cold water, the coating is finally rinsed in water at 150°F (66°C) minimum, and dried; an optional final sealing treatment to obtain maximum protection is obtained by immersion in a solution containing 0.01 g chromium trioxide per litre of water (0.0013 or 1 gal) at a temperature of 208 to 212°F (98 to 100°C) for 5 min.

6. Type D—Mechanical Treatments

6.1 Method 1, Hand or Power Wire-Brushing, or other abrasive treatment. Wire-brushing, either by hand or power, roughens the surface of the metal and mechanically improves the anchorage for superimposed paint films. The disadvantages are that the natural oxide film on the metal is disrupted and oil or grease films and other foreign matter are not completely removed.

6.2 Method 2, Sandblasting, where employed on aluminum or its alloys, must be carried out at relatively low pressures and with a fine silica sand. It roughens the surface

and mechanically improves the anchorage for paint films, but destroys the natural oxide film on the metal. Where used, it should be followed by an inhibitive chemical treatment.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting¹

This standard is issued under the fixed designation D 1731; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Section 2 was added and subsequent sections were renumbered editorially in September 1984.

1. Scope

1.1 These practices describe procedures for the preparation of hot-dip aluminum surfaces for painting.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

3. Types of Surface Preparations

3.1 Two types of surface preparations are covered as follows:

3.1.1 Solvent Cleaning, and

3.1.2 Chemical Treatments.

3.2 These two types cover several methods as described in Section 5. Variations in surface treatment produce end conditions which differ and which do not necessarily yield identical results when paints are applied. Service conditions will dictate the type of surface preparation that should be selected.

4. Solvent Cleaning

4.1 The surfaces may be prepared for painting by any of the methods for solvent cleaning described in 4.2 and 4.3.

4.2 *Method 1—Manual swabbing or dip washing with a solvent such as mineral spirits or high flask solvent naphtha. It is extremely difficult to produce a clean metal surface by this method. Accumulated contaminants in the solvent or on the swab are frequently redeposited on the surface being prepared.*

4.3 *Method 2—Solvent spray cleaning in accordance with Method A, Procedure 1 of Methods D 609.*

4.4 *Method 3—Vapor degreasing in special equipment in accordance with the latest revision of Method A, Procedure 2 of Method D 609, employing trichlorethylene or perchloro-*

ethylene vapor. Solvent cleaning does not disturb the natural oxide film on the metal and may prove adequate for some applications.

5. Chemical Treatment

5.1 The surfaces may be prepared for painting by any of the methods for chemical treatment (Note 1) described in 5.2 to 5.9:

NOTE 1—Materials and procedures employed in these methods of treatment are available from a number of sources as proprietary compounds or methods. Selection may be made from available sources.

5.2 *Method 1, Alkaline Cleaners—Etching alkaline solutions, such as caustic soda, should be avoided because of the possibility of dissolving away the aluminum coating thereby exposing the base metal. Since inhibited alkaline cleaners do not etch the surface, they can be used to remove grease, oil, and other organic solids from hot dip aluminum. After alkali cleaning, the surface should be thoroughly rinsed with clean water. Most often alkaline-cleaned surfaces are subsequently processed by one of the following methods, described in 5.3 to 5.9:*

5.3 *Method 2, Alcoholic Phosphoric Acid Cleaners—This treatment involves the use of an aqueous solution of phosphoric acid with alcohol or other organic solvents, together with wetting agents, emulsifying agents, etc. The solution may be applied by swabbing or dipping (preferably at a temperature of 70 to 90°F (21 to 32°C)). The solution should remain on the surface for several minutes, followed by thorough rinsing with clean water. A very thin phosphate film is formed which tends to protect the metal and promote paint adhesion.*

NOTE 2—Materials meeting the requirements of the latest revision of U.S. Military Specification MIL-M-10578B may be used.

5.4 *Method 3, Acid Fluoride Deoxidizer—This treatment includes the use of nitric or sulfuric acid with chromium trioxide and a fluoride. It provides a clean and uniform surface without undue roughening and is effective for removing inorganic soils and any film formed during the production of the hot dip aluminum. The deoxidizing solution is generally used at a temperature of 70 to 130°F (21 to 54°C) (depending on the alloy and the amount of film) and is followed by a water rinse.*

NOTE 3—Before applying the treatments according to Chemical Treatment Methods 3, 4, 5, 6 and 7, the aluminum surfaces should be freed of grease, oil or other foreign material by means of the procedure described in Solvent Cleaning Method 3, Chemical Treatment Method 1 or any other suitable method.

¹ These practices are under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings.

Current edition approved Sept. 8, 1967. Published November 1967. Originally published as D 1731-60. Last previous edition D 1731-66.

² Annual Book of ASTM Standards, Vol 06.01.

5.5 Method 4, Crystalline Phosphate Treatment—This surface-coating method consists in reacting the aluminum surface in a 130 to 145°F (54 to 63°C) (approximately) zinc acid-phosphate solution containing oxidizing agents and other salts for accelerating the coating action. The aluminum surface is converted to a crystalline, phosphate coating of the proper texture adapted to inhibit corrosion and increase the adherence and durability of any applied paint film. The phosphate coating process may be carried out by immersion or spray application. The aluminum surface is converted to the phosphate coating by immersion in the processing solution for 30 s to 4 min, or by spraying the solution for 10 s to 2 min (Note 3).

5.6 Method 5, Amorphous Phosphate Treatment (see Note 4)—This process comprises treatment of clean aluminum surfaces in a 90 to 125°F (32 to 51°C) aqueous solution comprising phosphoric, hydrofluoric acids and chromium trioxide. The aluminum surface is converted to a thin, adherent, amorphous coating, iridescent green to gray green in color, depending upon the aluminum alloy treated, which inhibits corrosion and increases the adherence and durability of applied paint films. The coating process may be carried out by immersion or spray application. The time of treatment will vary from 15 to 45 s for the spray process and 30 s to 3 min for the immersion application (Note 3).

5.7 Method 6, Carbonate Chromate Treatment (see Note 4)—This process comprises treatment in a 160 to 180°F (71 to 82°C) dilute solution of sodium carbonate and potassium chromate for periods from 2 to 20 min, followed by a water rinse. The surface is then given a final treatment in a 150 to 160°F (66 to 71°C), 5% potassium dichromate solution followed by a final rinse. The treatment produces a thin, adherent, conversion coating on the surface, increasing the corrosion resistance of the metal and promoting paint adhesion (Note 3).

5.8 Method 7, Amorphous Chromate Treatment (see Note 4)—This process comprises treatment of clean aluminum surfaces in aqueous chromium trioxide solutions containing suitable accelerating agents such as fluoride-containing materials. The aluminum surface is converted to an adherent, amorphous, mixed metallic oxide coating, iridescent golden to light brown in color, which increases the corrosion resistance and the adherence and durability of any applied paint film. The coating process may be carried out by immersion, spray or brush application, at 70 to 130°F (21 to 54°C), in from 15 s to 5 min contact time. It may be necessary to precede this treatment with a deoxidizer as described in Method 3 (see Note 3).

NOTE 4—Most of the treatments conforming to Chemical Treatment Methods 5, 6, and 7, comply with the requirements of the latest revision of U.S. Military Specification MIL-C-5541.

5.9 Method 8, Acid-Bound Resinous Treatment (see Note 5)—This surface treatment is based on the deposition of an acid-bound resinous film of approximately 0.3 to 0.5 mils (7.6 to 12.7 μ m) in thickness on the aluminum surface in such a manner as to enhance the adhesion of subsequently applied paint coatings. The treatment is based on three primary components: a hydroxyl-containing resin; a pigment capable of reacting with the resin and an acid; and an acid capable of insolubilizing the resin by reacting with the resin, the pigment and the metal substrate. The aluminum surface should be pretreated by Solvent Cleaning Methods 1, 2 or 3 or Chemical Treatment Methods 1 or 2 prior to the application of this treatment. The film may be applied by brush, spray, or dip. Under normal conditions it shall dry sufficiently for recoating within 30 min. The film has good adhesion to the metal substrate and promotes good adhesion of most subsequent organic coatings to itself.

NOTE 5—Materials meeting the requirements of the latest revision of U.S. Military Specification MIL-C-15328 may be used to apply to Chemical Treatment Method 8.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Practices for Preparation of Magnesium Alloy Surfaces for Painting¹

This standard is issued under the fixed designation D 1732; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These recommended practices have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These practices cover two classes of treatment for preparation of magnesium alloy surfaces for painting, as follows:

Class I—Chemical Treatments. Class II—Anodic Treatments.

In general, the latter treatments are the more protective of the two classes. Mechanical (abrasive) treatments, solvent cleaning, alkaline solution treatments, and acid pickles not resulting in protective conversion coatings are suitable preliminary treatments only for metal to be exposed under mildly corrosive (indoor) exposures. When a high degree of corrosion protection and paint adhesion are desired, as in many outdoor environments, surface preparation by one of the above conversion-coat classes is necessary.

NOTE 1—*Testing of Coatings*—Quality control tests of coatings are frequently desirable, and these generally consist of exposures, with or without paint, to salt spray, humidity, or natural environments, with suitable procedures for assessing the degree of breakdown suffered after fixed time intervals. It is recommended that quality control tests of coatings shall be made as far as possible with high-purity material (for example AZ31A alloy),² the inherent corrosion rate of which is relatively consistent from batch to batch) and that precautions shall be taken to remove surface contamination before coatings are applied. Such contamination shall be removed by acid pickling to a depth of at least 0.001 in. (25 μ m) per side.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. (See Note 11.)*

PRELIMINARY TREATMENT OF SURFACES

2. Procedure

2.1 Certain anodic treatments simultaneously produce conversion coatings on, and remove contamination from, magnesium alloy surfaces. In general, however, apply con-

version coatings only to surfaces previously freed from all contamination, including oxide, rolling-scale, corrosion product, burned-on drawing and forming lubricant, and the contamination introduced by blast cleaning and fabrication operations. Contamination in or under surface conversion coatings seriously reduces their protective values (Note 2). For the removal of tenacious surface contamination, such as rolling-scale or casting skin, an acid pickle to dissolve some of the actual surface is essential. When organic contamination, such as grease or oil, is also present, an initial degreasing operation in solvent or in an alkaline degreasing solution is usually necessary to allow the subsequent acid to wet the surface. These matters are discussed in more detail under the headings of the specific cleaners or treatments (Note 3), as follows:

2.2 *Alkaline Cleaners*—Oil, grease, and old (but not baked) chrome-pickle coatings are readily removed by most commercially available heavy-duty alkaline cleaners; but such cleaners are not suitable for removing oxide and the like, for which purpose use acid pickles, preceded by alkaline cleaners. Remove graphite lubricant and also baked chrome-pickle coatings by a solution conforming to the following composition:

Caustic soda (NaOH)	12 oz (90 g)
Wetting agent	0.1 oz (0.74 g)
Water (Note 4)	1 gal (1 litre)

Soak the parts in the above cleaner for 10 to 20 min at boiling-point, and a treatment shall follow either in the chromic acid-nitrate pickle described under 2.3.3 or, for parts machined to fine tolerances, in the chromium trioxide solution described under 2.3.1. After alkaline cleaning, rinse in water very thoroughly. Alkaline cleaners may be held in plain steel tanks.

2.3 *Acid Cleaners* (Note 1)—Acid picking removes mill-scale, oxide, corrosion product, and the like. Use as a preliminary treatment for surface conversion coatings when the highest degrees of surface uniformity and protective values are required. Acid cleaners are as follows:

2.3.1 For Sand and Permanent Mold Castings:

2.3.1.1 *Nitric-Sulfuric Acid Solution*—Use a solution of 8 volume % of concentrated nitric acid (HNO_3) plus 2 volume % of concentrated sulfuric acid (H_2SO_4) in water (see Note 1(a)) at 70 to 90°F (21 to 32°C) as a preliminary treatment for new sand castings and to remove the surface-contaminating effects of blast cleaning. Immerse for about 10 to 15 s, or until 0.002 in. (51 μ m) per surface is removed. The solution may be held in ceramic, rubber, synthetic rubber, or vinyl-lined tanks.

¹ These practices are under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings.

Current edition approved Sept. 8, 1967. Published November 1967. Originally published as D 1732-60. Last previous edition D 1732-66.

² For information concerning magnesium and aluminum alloys, see ASTM Specification B 80, B 90, B 91, B 93, B 107, and B 209 covering these alloys, in the section on Aluminum and Magnesium and Their Alloys, *Annual Book of ASTM Standards*, Vol 02.02. See also ASTM Practice B 275, for Codification of Certain Nonferrous Metals and Alloys, Cast and Wrought.

2.3.1.2 Chromic Acid Solution—Use a boiling 20 weight % solution of chromic anhydride (CrO_3) in water to remove old chemical and anodic treatments, corrosion product, and oxide layers, without significant dissolution of metal and hence without changing the dimensions of machined parts. Immersion time varies from 1 to 5 min, depending upon the condition of the surface. The solution may be held in lead-lined steel or ASTM alloy No. 990A or its Aluminum Association equivalent, alloy No. 1100 aluminum tanks.

2.3.2 For Die Castings:

2.3.2.1 Chromium Trioxide-Nitric-Hydrofluoric Acid Solution—This solution is used to produce a smut-free surface on die castings, without violent attack of the metal. The solution shall conform to the following composition:

Chromium trioxide (CrO_3)	37.5 oz (280 g)
Hydrofluoric acid (60% HF)	1 fl oz (8 ml)
Nitric acid (70% HNO_3)	3.25 fl oz (25 ml)
Water (Note 4)	to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for 30 s to 2 min, or until a bright, clean surface is obtained. The solution may be held in tanks lined with synthetic rubber or vinyl-base materials.

2.3.3 For Wrought Products:

2.3.3.1 Acetic Acid-Nitrate Solution—This solution rapidly removes surface contamination to 0.001 in. (25.4 μm). Use for wrought parts subsequently to be finished for the maximum protective value. The solution shall conform to the following composition:

Glacial acetic acid	25.5 fl oz (199 ml)
Sodium (NaNO_3)	6.6 oz (49.5 g)
Water (Note 4)	to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for 30 s upwards, or until a bright, clean surface is obtained. When heavy surface contamination, such as hot-rolled mill-scale is to be removed, immersion times shall be sufficient to remove at least 0.001 in. (25 μm) per surface. The solution may be held in No. 990A aluminum, ceramic, or rubber-lined tanks.

2.3.3.2 Chromium Trioxide-Nitrate Solution—Use this solution following the use of the method described in 2.2 for the removal of burned-on graphite lubricants from hot-formed parts. The solution shall conform to the following composition:

Chromium trioxide (CrO_3)	1.5 lb (180 g)
Sodium nitrate (NaNO_3)	2 oz (15 g)
Water (Note 4)	to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for approximately 5 min, for the removal of graphite.

2.4 Abnormally slow reaction in the above solution indicates that it is depleted and that the pH has risen to 1.7 or higher. Revivify the solution by the addition of chromium trioxide to bring the pH back to 0.5 to 0.7. Attempt no more than four revivifications. The solution may be held in ceramic, No. 990A aluminum, 18-8 stainless steel, or synthetic rubber-lined tanks.

NOTE 2—Removal of Contamination by Welding Fluxes—When a part to be painted has been welded by an operation involving the use of flux, such flux shall be removed before the subsection of the part to any surface preparation process. Such removal shall be made immediately by immersing the part in hot water with scrubbing, and finally by immersing it for 1 h in a boiling 5% solution of sodium dichromate,

after which the part shall be well rinsed.

NOTE 3: Suspension of Articles for Treatment—The use of magnesium alloy suspension wires is preferred for use in acid pickles in order to avoid objectionable contamination of the solutions through dissolution of the wire materials. Heavy metal contamination, particularly of copper, may deposit on the magnesium surface and lead to seriously reduced corrosion resistance. Copper suspension wires in the hot dichromate solutions are not objectionable.

NOTE 4: Quality of Water—In the preparation and make-up of acid pickles, dichromate solutions, and hot-water rinses, precautions shall be taken against the use of water contaminated with heavy-metal impurities, or excessive chlorides or sulfates. No upper limits can be specified at this stage for soluble impurities in the water, but powdering of coatings and poor resistance to corrosion are known to result from the use of contaminated water. Thus, when a choice exists, water from steam condensate or ion-exchange-treated water shall be employed in preference to well water or hard tap water.

SURFACE PREPARATION PROCEDURES—CHEMICAL

3. Class I, Type I (Chrome Pickle)³

3.1 Scope—Class I, Type I treatment is applicable to all forms and alloys of magnesium except certain special alloys containing silver, but since it may remove as much as 0.0006 in. (15 μm) of metal per surface, it shall not be used on parts machined to fine tolerances. When properly applied, the process constitutes a good paint base, but rigid control is required at each step. The treatment is applicable to magnesium alloy containing inserts of, or attached to, other metals.

3.2 Procedure—For wrought parts the bath shall conform to the following composition:

Sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	1.5 lb (180 g)
Nitric acid (HNO_3) (sp gr 1.42)	1.5 pt (187 ml)
Water (Note 4)	to 1 gal (1 litre)

3.2.1 For die-, sand- and permanent-mold castings the solution shall conform to the following composition:

Sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	1.5 lb (180 g)
Nitric acid (HNO_3) (sp gr 1.42)	1.5 pt (187 ml)
Sodium potassium, or ammonium acid fluoride (NaHF_2 , KHF_2 , or NH_4HF_2)	2 oz (15 g)
Water (Note 4)	to 1 gal (1.0 litres)

For wrought products, sand, and permanent-mold castings the above solutions operate at 70 to 90°F (21 to 32°C). The immersion times shall be from 1 to 2 min, the necessary time increasing with use of the solution. For die-castings give the parts a 15 to 30 s dip in water at 160 to 180°F (71 to 82°C), followed immediately by a 10-s dip in the second of the above baths, operated at 120 to 140°F (49 to 60°C). Failure to preheat the castings results in no coating in 10 s.

3.2.2 Following immersion remove the parts, allow to drain for not less than 5 s nor more than 30 s, then wash thoroughly in cold running water, followed by a dip in hot water at 160 to 180°F (71 to 82°C) to facilitate drying. Do not allow the parts to drain following the chromate treatment for more than the specified 30 s; excessive drainage times result in powdery coatings of poor value as paint bases. Such coatings also result from the use of hot-water rinses, the temperatures of which are in excess of 180°F (82°C). Paint the parts preferably immediately after they are dry. The solution may be held in Type 316 stainless steel, or ceramic tanks or in steel tanks lined with synthetic rubber or

vinyl-base material. Aluminum minimum are pickle.

3.3 Revivify metal, associates that the plished by the the dichromate free nitric acid when the free 0.5 pt/gal (6 procedure) a ZK60A alloy if good paint solution or lustrous coat for the best Revivify in a

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- (4) Metal
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³ Conforming No. 10 process.

vinyl-base materials. Tanks of ASTM alloy No. 990 A, or its Aluminum Association equivalent, Alloy No. 1100 aluminum are satisfactory for the nonfluoride-containing pickle.

3.3 *Revivification of Solutions*—Sluggish reaction with the metal, associated with pale yellow, lustrous coatings, indicates that the solution is depleted. Revivification is accomplished by the addition of dichromate and nitric acid, to raise the dichromate again to 1.5 lb (180 g)/gal (1 litre) and the free nitric acid to levels indicated in the table below. Revivify when the free nitric acid content of the solution is depleted to 0.5 pt/gal (62.3 ml/litre) (see Section 11 for analytical procedure) and shall take place once only for M1 and ZK60A alloys,³ and not more than six times for other alloys if good paint-base properties are desired. Excessive use of the solution or too many revivifications result in smooth, lustrous coatings not possessing the degree of etch necessary for the best paint adhesion with conventional primers. Revivify in accordance with the following table:

Revivification Number	Adjust Concentration of HNO ₃ to:
1	1.3 pt/gal (162 ml/litre)
2	1.1 pt/gal (137 ml/litre)
3 to 6	0.9 pt/gal (113 ml/litre)

4. Class I, Type II (Sealed Chrome Pickle)⁴

4.1 *Scope*—Class I, Type II treatment is applicable to all types and forms of magnesium-base alloys, subject only to the limitations of the Class I, Type I treatment, since it is essentially the same process as the latter, followed by sealing. In protective qualities and as a paint base, this treatment is somewhat superior to that of Class I, Type I treatment under severe exposure conditions (Note 5).

4.2 *Procedure*—Following chrome pickling as specified under Section 3 and rinsing in cold water, transfer the parts immediately to a boiling solution conforming to the following composition:

Sodium dichromate (Na ₂ Cr ₂ O ₇ ·2H ₂ O)	1.5 lb (180 g)
Calcium or magnesium fluoride (CaF ₂ or MgF ₂)	½ oz (2.5 g)
Water (Note 4)	to 1 gal (1.0 litre)

Boil the parts in the above solution for 30 min, after which rinse them in cold running water, followed by a rinse in hot water at a temperature of not less than 160°F (71°C) nor more than 180°F (82°C) to facilitate drying. Preferably apply the paint coating immediately after the parts are dry. The solution may be held in a steel tank.

NOTE 5: *Causes of Defective Coatings*—The following information is intended to provide guidance on the causes of the most usual defects arising in the application of either Class I, Type I or Class I, Type II coatings:

(a) *Spotted Coatings* are caused by ineffective preliminary degreasing, or by the presence of excessive surface contamination not removed prior to or during the chrome pickling treatment, or both.

(b) *Nonadherent Powdery Coatings* are caused by:

- (1) Too long an interval between removal from the chrome pickle and rinsing,
- (2) Ratio of acid to sodium dichromate too high,
- (3) Temperature of the solution or of the hot-water rinse too high,
- (4) Metal improperly degreased, or
- (5) Solution revivified too many times.

⁴ Conforming to Class I, Type II treatments are the AMC "L" process, the Dow No. 10 process, and Type II treatment of Military Specification MIL-M-3171A.

5. Class I, Type III⁵

5.1 *Scope*—Class I, Type III treatment is applicable to all types and forms of magnesium-base alloys except M1 alloy² and certain rare-earth alloys similar to EK30A.² It produces in itself no appreciable dimensional change and is, therefore, applicable to parts machined to fine tolerances. The treatment is applicable to magnesium containing inserts of, or attached to, other metals, but in such cases the preliminary treatment (5.2) shall be in the bifuoride bath (Solution No. 2) described in 5.2.2.

NOTE 6: *Causes of Defective Coatings*—The following information is intended to provide guidance on the causes of the more usual defects arising in the application of coatings from the Class I, Type III treatment.

(a) *Nonadherent Powdery Coatings* are caused by:

- (1) Over-dilution of the hydrofluoric acid or acid fluoride solution,
- (2) Low pH (less than 4.0) of the dichromate solution,
- (3) Insufficient precleaning of the metal surface, and
- (4) Direct contact between the steel tank containing the dichromate solution and the article being treated.

(b) *Failure to Coat and Nonuniform Coatings* are caused by:

- (1) High pH of the dichromate solution,
- (2) Low concentration of the dichromate,
- (3) Insufficient precleaning of the metal surface,
- (4) Omission of fluoride treatment,
- (5) Use of an unsuitable alloy for the treatment, for instance, M1 alloy,
- (6) Excessive immersion time in the hydrofluoric acid solution or use of an H₂F₂ concentration in the dichromate solution in excess of 0.2%,
- (7) Insufficient rinsing after the hydrofluoric acid dip, and
- (8) Insufficient heating of the dichromate solution (minimum temperature should be 200°F (93°C)).

5.2 *Procedure*—Following cleaning as prescribed in Section 2, treat the parts first by immersion at 70 to 90°F (21 to 32°C) in one or other of the following solutions:

5.2.1 Solution No. 1:

Hydrofluoric acid (60% H ₂ F ₂)	24 fl oz (187 ml)
Water (Note 4)	to 1 gal (1 litre)

5.2.2 Solution No. 2:

Sodium, potassium, or ammonium acid fluoride (NaHF ₂ , KHF ₂ , or NH ₄ HF ₂)	6½ oz (50 g)
Water (Note 4)	to 1 gal (1 litre)

Solutions No. 1 and No. 2 may be held in tanks consisting of steel lined with lead or rubber. In solution No. 1, immerse AZ31A and AZ31B alloy parts³ for from 30 s to 1 min; immerse all other alloys for 5 min. In solution No. 2, immerse all the alloys for 5 min. Solution No. 2 is suitable for use with all forms of magnesium alloys except those castings which have not been acid-pickled after blasting; these castings shall be treated in the hydrofluoric acid solution (solution No. 1). Following one or other of the above treatments, the parts shall be rinsed thoroughly in cold running water and transferred to a solution conforming to the following composition:

Sodium dichromate (Na ₂ Cr ₂ O ₇ ·H ₂ O)	1 to 1.5 lb (120 to 180 g)
Calcium or magnesium fluoride (CaF ₂ or MgF ₂)	½ oz (2.5 g)
Water (Note 4)	to 1 gal (1 litre)

⁵ Conforming to Class I, Type III treatments are the AMC "G" process, the Dow No. 7 process, and the Type III treatment of Military Specification MIL-M-3171A.

Operate the above solution at boiling point and immerse the parts therein for 30 min, following which thoroughly rinse them in cold running water, followed by a hot-water dip to facilitate drying. Preferably, apply the paint coating as soon as possible thereafter. A mild steel tank is suitable for holding the above solution.

5.3 Maintenance of Solutions:

5.3.1 *Fluoride Solutions*—Keep the concentration of free hydrofluoric acid in the solutions of 5.2.1 and 5.2.2 constant by the addition, as required, of either hydrofluoric acid or acid fluoride, respectively. See Section 9 for the analytical procedure.

5.3.2 *Dichromate Solution*—Keep the pH of the dichromate solution within the limits 4.0 to 5.5, or 4.0 to 4.8 in the case of AZ31A or AZ31B alloy,² by addition of chromium trioxide as required. Maintain the level by the addition of water. Maintain the solution saturated with respect to either magnesium or calcium fluoride by continuous immersion of a cloth bag containing excess of one or other of these compounds.

SURFACE PREPARATION PROCEDURES—ANODIC

6. Class II, Type I (Galvanic Dichromate Treatment)⁶

6.1 *Scope*—This treatment produces black coatings of good protective and paint-base qualities, and is applicable to all alloys and forms of magnesium, including M1 alloy,² with or without attachments of other metals. No appreciable dimensional change results, and hence the treatment is also applicable to articles machined to fine tolerances.

6.2 *Procedure*—Following cleaning as prescribed in Section 2, treat the articles in fluoride solution No. 1 or No. 2 as prescribed in 5.2. Use the latter solution when other metals are attached to the magnesium. After rinsing, immerse the articles in a solution conforming to the following composition:

Ammonium sulfate ((NH ₄) ₂ SO ₄)	4 oz (30 g)
Sodium dichromate (Na ₂ Cr ₂ O ₇ · 2H ₂ O)	4 oz (30 g)
Ammonia (NH ₄ OH) (sp gr 0.880)	1/2 fl oz (2.6 ml)
Water (Note 4)	1 gal (1 litre)

Operate the above solution at 120 to 140°F (49 to 60°C). The articles shall be made the anodes in the solution, with the tank, if of mild steel, acting as cathode, or with separate steel cathodes if the tank is lined with nonmetallic materials. No separate generator is necessary, but the magnesium parts must be electrically connected with the tank, or with the separate cathode plates through an external connection, taking care that the parts do not make direct contact with the cathode material.

6.2.1 The time of treatment shall be such that a uniform black coating is obtained on the articles. This takes from 10 to 30 min, and the consumption of from 70 to 150 A·min/ft² (929 cm²). An anodic current density of not more than 10 A/ft² (929 cm²) is desirable.

6.2.2 Following the treatment, rinse the parts thoroughly in cold running water, followed by a hot-water dip to facilitate drying.

⁶ Conforming to Class II, Type I treatment are the AMC "K" process, the Dow No. 9 process, and Type IV treatment of Military Specification MIL-M-3171A.

6.3 Maintenance of Solutions:

6.3.1 *Fluoride Solutions*—Maintain the fluoride solutions as under 5.3.1. See Section 9 for the analytical procedure.

6.3.2 *Sulfate-Dichromate Solution*—Maintain the pH of this solution between 5.6 and 6.2 by periodic additions of a solution containing 5 weight % of both chromic anhydride (CrO₃) and concentrated sulfuric acid (H₂SO₄, sp gr 1.84).

7. Class II, Type II⁷

7.1 *Scope*—This treatment is applicable to all forms and alloys of magnesium. Many aluminum alloys will anodically polarize in the treatment solution, and hence magnesium parts with aluminum attachments or inserts can be anodized. With aluminum alloys containing copper, however, the coating of the magnesium will proceed only if the area of the aluminum alloy is small compared to that of the magnesium. Metals other than aluminum or its base alloys shall not be in contact with the magnesium.

7.2 *Coating Properties*—The anodic coating consists of two phases: the first-formed phase is greenish-tan in color and about 0.0002 in. (5 μm) thick; whereas the second phase, formed only at higher voltages following the production of the tan coating, is fused and vitreous in nature, dark-green in color, and of thickness about 0.0012 in. (30 μm), causing a dimensional increase of about 0.001 in. (25 μm) per surface. The corrosion-resistance and paint-base characteristics are similar, and excellent for both the above phases. The coating possesses high abrasion resistance, but spalls under compression deformation. The dark-green coating can be partially impregnated with low-viscosity, penetrating, organic coating materials, a procedure that considerably reduces its spalling tendency.

NOTE 7—The dark-green coating of Class II, Type II treatments is recommended in preference to the thin tan coating only when:

- (a) Preliminary removal of surface contamination is not convenient.
- (b) The highest degree of abrasion-resistance is required from the coating.
- (c) A dimensional increase (see above) can be tolerated.
- (d) The article will not be subjected in service to impact, deformation, or flexing likely to cause spalling of the coating.

7.3 *Procedure*—The thin tan coating is formed with a current consumption and in a time approximately one quarter of that required to form the dark-green coating. The latter forms only after the voltage across the solution has reached the spark potential. During the ensuing coating formation, surface contamination, including graphite, is concurrently removed. Thus, articles intended to receive the dark-green coating need not first be cleaned. On the other hand, the formation of the thinner tan coating does not concurrently remove surface contamination. Since such contamination can seriously reduce corrosion resistance, it is mandatory that a preliminary cleaning, including acid pickling (2.2) shall be given to articles intended to receive the lower-voltage tan coating.

7.3.1 Rack and clean parts as appropriate (see above), and anodize either by direct or alternating current in an electrolyte conforming to one of the following compositions, respectively:

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ZK60

⁷ Conforming to Class II, Type II treatment is the Dow No. 17 process.

	Direct Current	Alternating Current
Ammonium acid fluoride (NH_4HF_2)	32 oz (240 g)	40 oz (300 g)
Sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	13.3 oz (99 g)	13.3 oz (99 g)
Phosphoric acid (85% H_3PO_4)	11.5 fl oz (90 ml) to 1 gal (1 litre)	11.5 fl oz (90 ml) to 1 gal (1 litre)
Water (Note 4)		

The temperature of the solution shall preferably be from 160°F to 180°F (71 to 82°C). It will not operate below 140°F (60°C), but temperatures higher than 180°F (82°C) do not deleteriously affect the results. After anodizing rinse the parts in running cold water, followed by a short immersion in hot water, or by treatment with hot air, to facilitate drying. Preferably, apply the paint coating immediately after the parts are dry.

7.4 Power Requirements—A current consumption of from 50 to 500 A·min/ft² (929 cm²) is necessary, according to both the composition of the alloy being treated and the type of coating desired. Thus, in the case of the dark-green coating on AZ31 alloy, for a 10-min treatment with alternating current, a current density of 30 A/ft² (929 cm²) is applied; but greater or lesser currents may be utilized by varying the time of treatment as, for example, application of 50 A/ft² (929 cm²) for 6 min. On the other hand, for the same alloy, the thin tan coating may be applied with alternating current, with a current consumption of 75 A·min/ft² (929 cm²), equivalent to a treatment time of only 1½ min at 50 A/ft² (929 cm²). As the coating forms, the resistance of the circuit increases, and consequently the voltage must be increased to maintain a constant current density. Normally the voltage across the bath will reach from 75 to 95 for the dark-green coating (according to alloy composition) and from 60 to 75 for the thin tan coating. In carrying out the treatment, a constant current control is a considerable advantage, inasmuch as the total treatment time can be preset and manual control of the voltage and current then become unnecessary.

7.4.1 As guides to the current consumptions with various alloys, some data are presented in Table 1. It will be appreciated that rigid current consumptions in the formation of the thin tan coating cannot be specified, since this coating can be varied in thickness considerably between say 0.0001 in. (2.5 µm) and 0.0005 in. (12.7 µm), and still be formed below the critical spark potential at which the dark-green vitreous coating begins to develop.

7.5 Solution Control—With depletion of the bath upon use, the final voltage required to impress the above currents on the article will slowly rise, but even for the full green coating at least 20 ft² (50 dm²) of surface can be treated per

gallon (1.0 litre) of solution before any change in the final voltage is experienced; and up to 40 ft² (98 dm²)/gal (1 litre) can be treated before revivification of the solution becomes necessary.

7.5.1 Prior to revivification, analyze for dichromate and phosphoric acid (see Sections 10 and 12), and make up differences from the original composition by additions of sodium dichromate and phosphoric acid.

7.5.2 A simple method for the determination of fluoride in the solution is not yet available. The fluoride concentration, is not critical, provided it always exceeds minimum concentration, below which pitting of the articles may ensue during their treatment. When revivifications are made by additions of sodium dichromate and phosphoric acid, make a concurrent addition of ammonium acid fluoride, equal to three times the weight of sodium dichromate added. Secondly, if pitting of the articles should occur under treatment, it is an indication that the fluoride content of the solution is too low, and about 5 oz (38 g)/gal (1 litre) of ammonium acid fluoride shall then be added in order to relieve the trouble.

7.6 Tank and Rack Materials—Mild steel is suitable for tanks and heating coils. Other metals, such as copper, aluminum, lead, zinc, Monel, and stainless steels are attacked by the electrolyte. When direct current is used the tank itself can be the cathode, but, when alternating current is used, at least two magnesium parts are necessary, of area ratios not more than 3 to 1, each one serving as electrode. The articles shall not contact the tank, otherwise pitting will ensue.

7.6.1 Racks shall be normally made of magnesium alloy, but aluminum alloys containing magnesium, such as ASTM alloy No. GR20A, or its Aluminum Association equivalent, alloy No. 5052, can be used if desired, provided they are not allowed to be in the solution on open circuit. Use a spring clip with magnesium ends for providing contact with thin sheet material, the liquid level not being allowed to extend beyond the limit of the magnesium ends of the clip. No solution-line attack is experienced on magnesium. Magnesium racks may be stripped by immersion for 2 min in hot 20% chromium trioxide solution or by immersion at room temperature for about 8 min in the chromium trioxide-nitrate solution bath described in Section 2.2.3.

8. Class II, Type III^a

8.1 Scope—This treatment is applicable to all forms and alloys of magnesium, free from attachments or inserts of other metals.

8.2 Coating Properties—The anodic coating consists of two phases: The first-formed subcoating phase is a light tan in color, and causes a dimensional increase of about 0.0002 in. (5 µm) per side; the second and thicker phase, formed at higher voltages, is dark brown and causes a dimensional increase of from 0.001 in. to 0.0012 in. (25 µm to 30 µm) per surface. The paint-base characteristics are similar, and excellent for both the above phases.

8.2.1 The dark-brown coating is hard and highly abrasion-resistant, but it spalls under compression deformation and its

TABLE 1 Current Consumption with Various Alloys

Alloy	Alternating Current				Direct Current			
	Voltage		A·min/ft ² (929 cm ²)		Voltage		A·min/ft ² (929 cm ²)	
	Dark-Green	Thin Tan	Dark-Green	Thin Tan	Dark-Green	Thin Tan	Dark-Green	Thin Tan
AZ31A or B	90	70	300	75	95	75	190	50
AZ61A	90	68	400	...	95	72	300	...
AZ63A	80	68	600	100	84	72	400	70
AZ91A	75	64	480	...	80	69	280	...
AZ92A	75	64	600	...	80	69	400	...
EK30A	90	70	400	...	95	75	300	...
ZK60A	85	70	300	...	90	75	190	...

^a Conforming to Class II, Type III treatment is the HAE process.

formation can cause a loss of fatigue strength of the metal. Such loss is negligible with metal of thickness 0.1 in. (2.5 mm) or over, rising to 3% and 9% for thicknesses of 0.094 in. (2.4 mm) and 0.040 in. (1 mm), respectively, all under reversed bending and corrected for coating thickness. No loss of fatigue strength arises from application of the thin tan coating, and neither is this coating subject to spalling.

8.3 Procedure—Rack and clean with hot alkaline cleaner (2.1) or chromium trioxide (2.2.1), or both, as appropriate, and anodize with alternating current in an electrolyte conforming to the following composition:

Potassium hydroxide (KOH)	18 to 20 oz (135 to 150 g)
Aluminum hydroxide (Al(OH) ₃) soluble in hot KOH solution (Note 8)	4 oz (30 g)
Potassium fluoride (K ₂ F ₂)	4.5 oz (34 g)
Trisodium phosphate (Na ₃ PO ₄)	4.5 oz (34 g)
Potassium manganate (K ₂ MnO ₄) (Note 9)	2.5 oz (19 g)
Water (Note 4)	to 1 gal (1 litre)

Dissolve the constituents of the electrolyte in the above order. Maintain the temperature of the electrolyte between 77 and 100°F (25 to 38°C).

NOTE 8—If an exceptionally hard, abrasion-resistant dark-brown coating is desired, the aluminum hydroxide in the above electrolyte shall be increased to 6 to 7 oz (45 to 52 g)/gal (1 litre).

NOTE 9—Potassium manganate (K₂MnO₄) in the above electrolyte may be replaced with an equal weight of potassium permanganate (KMnO₄), but in such case the permanganate shall be completely dissolved in water before it is added to the solution and an additional 1½ oz (11 g) of potassium hydroxide shall also be added. During operation of the bath, the permanganate is slowly reduced to manganate, complete conversion taking place after a metal area of approximately 2 ft² (18.6 dm²)/gal (1 litre) of electrolyte has been treated.

8.4 Post-Treatment—After the electrolytic treatment, rinse the parts well in water and immerse for 1 min at room temperature in a solution consisting of 2.7 oz (20 g) of sodium dichromate (Na₂Cr₂O₇·2H₂O) and 13.3 oz (99 g) of ammonium bifluoride (NH₄HF₂)/gal (1 litre) of solution. Following the immersion, remove the parts and dry without rinsing. Paint at any convenient time after the parts have dried.

8.5 Power Requirements—The practical range from current density is from 15 to 20 A/ft² (9.3 dm²) with voltages rising to from 70 to 86 (depending on alloy) for the hard brown coating and to 58 V for the thin tan coating. In this current density range, the terminating voltage is reached in from 45 to 60 min for the hard brown coating and in 10 min or less for the thin tan coating. Raise the voltage quickly from zero to 40 or more, and adjust the current and retain at the density desired. Normally the voltage will rise to 55 or more within 1 min, but as the coating forms, the resistance of the circuit increases and the voltage must be raised continuously to maintain the desired current density.

NOTE 10—If, at low initial voltages, the current remains high and coating formation does not proceed, a "surge" current shall be imposed by rapidly raising the voltage to 40, followed by immediately lowering it to zero. The voltage shall then be adjusted in the normal manner to maintain the desired current density.

8.6 Solution Control—The operating life of the electrolyte is extremely long, and under normal conditions of use it need never be discarded. Manganate and aluminate deplete slowly with use, whereas the depletion of fluoride and phosphate is extremely slow.

8.6.1 In the case of the dark-brown coating, a lightening of the normal color indicates a depletion of manganate in the solution, and additions shall then be made of 1 oz (7.5 g) of potassium manganate plus 1½ oz (11 g) of aluminum hydroxide for each gal (1 litre) of solution. Add the aluminum hydroxide after first dissolving it in a solution containing a weight of potassium hydroxide equal to that of the aluminum hydroxide to be added.

8.6.2 A rough coating indicates either a low, or an excessively-high, alkalinity in the electrolyte. The alkalinity shall be maintained between 10 and 12% free KOH, as determined by the method described in Section 13.

8.7 Tank and Rack Materials—Plain black iron is suitable for tanks and cooling coils. Suspend the parts by magnesium-base alloy clips or the like and protect them at the solution level by electroplaters' vinyl tape. Magnesium racks may be stripped of coating when necessary by immersion in hot 20% chromium trioxide solution (Note 11).

8.7.1 Divide parts to be treated into two batches each of approximately the same surface area. Rack each of the batches and connect electrically to one or other of the inlet terminals of the power supply so that the magnesium parts constitute both electrodes of the electrolysis bath (Note 12).

NOTE 11: Safety Precautions—Consideration should be given to the health hazards associated with the procedures covered by this specification. Precautions must be taken to avoid skin contact with any of the solutions involved, and to avoid inhalation of vapors, fumes, or spray arising therefrom. Baths containing dichromate or fluoride shall be fitted with exhausts to remove all spray arising therefrom.

NOTE 12: Repair Treatments—Pretreatment films that have been damaged shall be repaired before painting. Repair shall be effected by one of the treatments below. The phosphate touch-up solution of 8.2 below necessitates special preparation, whereas a chrome pickle solution is often already available. Nevertheless, for brush-on treatment, the phosphate solution possesses the advantages of being nontoxic and nonirritant.

(a) **Chrome Pickle Repair Treatment**—The chrome pickle solution for Class I, Type I treatments (for wrought parts) shall be copiously and continuously applied to the damaged area by brush for at least 1 min. The treated surface shall then be thoroughly flushed with water and, when dry, rubbed vigorously with a clean dry rag to remove loose powdery matter which, if allowed to remain, would deleteriously affect the adhesion of superimposed paint.

(b) **Phosphate Repair Treatment**—A solution shall be used conforming to the following composition:

Monoammonium phosphate (NH ₄ H ₂ PO ₄)	16 oz (84 g)
Ammonium sulfite ((NH ₄) ₂ SO ₃ ·H ₂ O)	4 oz (30 g)
Ethylene glycol monobutyl ether	13 to 20 fl oz (101 to 156 ml)
Water (see Note 4)	1 gal (1 litre)

The above solution shall be applied copiously to the damaged area by brush in sufficient quantity to keep the surface wet for at least 1 min, or until a medium to dark-gray continuous coating is formed. The treated metal surface shall then be well flushed with water and allowed to dry before the paint is applied.

ANALYTICAL PROCEDURES

9. Fluoride Solutions

9.1 This method is applicable to the relevant baths of Class I, Type III (Section 5) and Class II, Type I (Section 6) treatments. The concentrations of both hydrofluoric acid (HF) and bifluoride shall be determined by titration with 1 N sodium hydroxide (NaOH) with 1 N sodium hydroxide (NaOH) solution, using phenolphthalein as indicator.

9.2 Maintain the hydrofluoric acid solution described in 5.2 by additions of fresh acid to give a titration of between 10 and 20 ml of 1 N NaOH solution per 2 ml of sample. Maintain the bifluoride solution described in 5.2 so that 10 ml thereof gives a titration of 4.5 to 5.5 ml of 1 N NaOH solution.

9.3 For the hydrofluoric acid the sample shall be drawn into a wax-lined pipet, discharged therefrom into at least 100 ml of distilled water, and titrated immediately. A rubber bulb, or a length of rubber tubing fitted to the suction end of the pipet, shall be used in drawing the sample into the pipet.

10. Dichromate Solutions

10.1 This method is applicable to solutions of the following treatments: Class I, Types I, II, and III; and Class II, Types I and II (Sections 3, 4, and 5; and Sections 6 and 7, respectively).

10.2 Add 1 ml of the dichromate solution concerned to 150 ml of distilled water, and mix therewith 5 ml of concentrated hydrochloric acid (HCl, sp gr 1.19) and 5 g of potassium iodide (KI). After at least 2 min, titrate the liberated iodine in the solution with 0.1 N sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution using starch as an internal indicator.

10.3 Calculation:

$$A \times 0.0415 = B$$

$$A \times 4.96 = C$$

where:

A = millilitres of 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution,
B = pounds per gallon of sodium dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$), and
C = grams per litre of sodium dichromate.

11. Nitric Acid Solution

11.1 This method is applicable to Class I, Types I and II treatment solutions.

11.2 Mix 50 ml of distilled water with 1 ml of the solution in question, and titrate the mixture with 0.1 N sodium hydroxide (NaOH) solution to a pH of 4.0 to 4.05, using a pH meter with a glass electrode.

11.3 Calculation:

$$A \times 0.0505 = B$$

$$A \times 6.34 = C$$

where:

A = millilitres of 0.1 N NaOH solution, and
B = pints per gallon of nitric acid (HNO_3),
C = millilitres of 70% nitric acid per litre.

12. Phosphoric Acid Solution

12.1 This method is applicable to the Class II, Type II treatment solution. The phosphoric acid is precipitated as ammonium phosphomolybdate, which is dissolved in an

excess of sodium hydroxide, such excess being titrated with standard acid. Interference from fluoride is prevented by converting it to fluoborate, by addition of boric acid.

12.2 *Molybdate Reagent*—Prepare as follows: mix 118 g of 85% molybdic acid (H_2MoO_4) with 400 ml of distilled water, add 80 ml of ammonium hydroxide (NH_4OH) solution (sp gr 0.880) with stirring, and filter when solution is complete. Slowly pour the filtrate, with stirring, into a cold mixture of 400 ml of concentrated nitric acid (HNO_3 , sp gr 1.42) and 600 ml of water. Allow the solution to stand over night, and filter.

12.3 *Procedure*—Dilute a 14-ml weighed sample of the solution under examination to 500 ml, and then further dilute 50 ml of this diluted solution to 500 ml. Then again dilute 25 ml of the latter solution to 100 ml, and add 10 g of ammonium nitrate (NH_4NO_3), 1 g of boric acid (H_3BO_3), and 15 ml of concentrated nitric acid (HNO_3 , sp gr 1.42). Heat the mixture to 104°F (40°C) and slowly add 40 ml of molybdate reagent. Allow the resulting precipitate of ammonium phosphomolybdate to stand for 30 min, and then filter through a fine-texture, low-ash paper. Wash the precipitate free from acid by means of a 1% solution of potassium nitrate (KNO_3), and mix, together with the filter paper, with an excess of 0.1 N sodium hydroxide (NaOH) solution, using phenolphthalein as indicator. Titrate the excess of NaOH with 0.1 N hydrochloric acid (HCl).

12.4 Calculation:

$$\text{Phosphoric acid } (\text{H}_3\text{PO}_4), \% = [(A - B)/C] \times 0.426$$

where:

A = millilitres of 0.1 N NaOH solution,
B = millilitres of 0.1 N HCl, and
C = grams of sample in aliquot.

13. Free Alkali

13.1 This method is applicable to the Class II, Type III solution. Manganate, fluoride, and phosphate are precipitated by barium nitrate addition, and the filtrate therefrom is titrated with standard acid.

13.2 Pipet a 2-ml sample of the solution under examination into a 25-ml glass-stoppered graduated cylinder, followed by addition of 10 ml of 0.25 N sodium hydroxide (NaOH) solution and 0.5 g of barium nitrate ($\text{Ba}(\text{NO}_3)_2$) crystals. Shake this mixture vigorously for 1 min, filter through a medium-texture, low-ash paper, and wash the precipitate free from alkali, preserving all filtrate. Using a pH meter with a glass electrode, titrate the free alkali in this filtrate to pH 10.5, with 0.25 N hydrochloric acid (HCl).

13.3 Calculation:

$$\text{Potassium hydroxide}, \% = 2.805 \times [(A/4) - 2.5]$$

where:

A = millilitres of acid used.

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Standard Method of Making and Preparing Concrete and Masonry Panels for Testing Paint Finishes¹

This standard is issued under the fixed designation D 1734; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

^{e1} Note—Editorial changes were made in Sections 2.1 and 5.3 on October 1980.

1. Scope

1.1 This method describes the procedures and materials required in making concrete and masonry panels for use in testing paint finishes designed for either portland cement concrete or stucco surfaces.

2. Apparatus

2.1 *White Asbestos Cement Shingles*, 12 by 24 in. (305 by 610 mm) in size, or asbestos cement sheet, $\frac{1}{8}$ in. (3.12 mm) thick, of the required size.

2.2 *Forms*, wooden or rustproof metal, to make panels of the required size.

2.3 *Container*, of wood, stainless steel, or other rustproof metal of sufficient size to mix the mortars.

2.4 *Notched or Serrated Applicator*, similar to those used in applying cement for tile or linoleum flooring.

2.5 *Miscellaneous Equipment*—Wooden or cork trowel, wooden screed, steel trowel and 3-in. (76-mm) wide stiff-bristle paint brush.

NOTE 1—Wooden trowels, screeds or containers may be made of seasoned oak which may be rendered nonabsorptive by immersion for 15 min in paraffin at 220 C (392 F) in accordance with ASTM Method C 227, Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method).²

3. Materials

3.1 *Portland Cement*, conforming to the requirements of Type I of ASTM Specification C 150, for Portland Cement.²

3.2 *Masonry Cement*, conforming to the requirements of Type II of ASTM Specification C 91, for Masonry Cement.²

3.3 *Natural Sand*, conforming to the requirements of ASTM Specification C 144, for Aggregate for Masonry Mortar,³ modified as follows:

3.3.1 The standard sand may provide too coarse a texture on portland cement concrete panels. If smoother texture is desired, the requirement of the specification that not more than 35 % shall pass the No. 50 (300- μ m) sieve should be waived, and the sand should be required to contain from 50

to 60 % passing the No. 50 sieve, and from 5 to 20 % passing the No. 100 (150- μ m) sieve, with the other provisions of grading remaining unchanged.

3.3.2 The standard sand will provide aggregate too large for a satisfactory masonry cement (stucco) mortar mixture. Satisfactory sand may be obtained by requiring that 100 % pass the No. 16 (1.18-mm) sieve, from 45 to 80 % pass the No. 30 (600- μ m) sieve, from 65 to 75 % pass the No. 50 sieve, and from 10 to 30 % pass the No. 100 sieve.

3.4 *Water*, of sufficient purity as to be potable.

3.5 *Resin Emulsion*.⁴

4. Size of Test Panels

4.1 For use on outdoor testing racks, the panels shall be 8 by 12 by $\frac{3}{8}$ in. (203 by 305 by 9.5 mm) in size.

4.2 For machine exposure tests, the panels shall be of a size to fit the apparatus to be used for such tests. The panels shall be $\frac{1}{8}$ in. (9.5 mm) in thickness and should have a surface area of not less than 18 in.² (116 cm²).

5. Application of Bond Coat

5.1 Prepare forms of correct size for panels to be made and place upon a wooden platen or table. The forms should be oiled or waxed before use.

5.2 For ease in removing the finished test panel, make the forms easily demountable, or equip the table upon which panels are made with several plungers, operated by foot pressure, which may be used to force the completed panel up and out of forms easily.

5.3 Cut the asbestos-cement shingles or the asbestos-cement sheet in rectangles $\frac{1}{32}$ in. (0.8 mm) under the required panel size to provide a slip fit within the forms. (Caution—Use vacuum dust collector for removal of asbestos-cement particles during cutting.)

5.4 For each square foot of panel to be made, make up a bond coat consisting of 60 g of sand, 20 g of cement, 8 g of water, and 20 g of emulsion.

5.5 In the mixing box, thoroughly mix the sand and cement (dry), then add the water. After thoroughly mixing, add the emulsion and mix until homogeneous.

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials.

Current edition accepted Sept. 30, 1963. Originally issued 1960. Replaces D 1734 - 60 T.

² Annual Book of ASTM Standards, Vols 04.01 and 04.02.

³ Annual Book of ASTM Standards, Vols 04.02 and 04.05.

⁴ Emulsion Rhoplex AC-33 obtainable from Rohm & Haas Co., Independence Mall W., Phila., Pa., or Resyn 12K51 obtainable from the National Starch and Chemical Corp., 1700 W. Front St., Plainfield, N. J., or equivalent resin emulsion are satisfactory for this purpose.

5.6 Wet surface of t about 5 mi
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5.8 Clear coating, so apply the c
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6. Preparation

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6.2 Place side up, wit on top of mixture int top with a trowel to le Finally, bru

NOTE 3—I may be wette water-cement and sloppy m

5.6 Wet the smooth or back side of the shingle, or the surface of the sheet to which the bond coat will be applied, about 5 min before applying the bond coat.

5.7 Distribute the required amount of bond coat upon the previously wetted shingle or asbestos cement sheet, using either a brush, or a notched applicator or scraper, having notches approximately $\frac{1}{8}$ in. (3.2 mm) deep.

5.8 Clean the edges of the panel immediately after coating, so that the panel will fit in forms when ready to apply the concrete or masonry mix.

5.9 Allow the bond-coated panel to stand overnight (minimum).

6. Preparation of Concrete Panels

6.1 In the mixing box, prepare the concrete, from 1 part of portland cement and 3 parts of sand by volume. Thoroughly mix these dry ingredients. Add water and mix thoroughly. Use only enough water to make the mortar sufficiently plastic as to be easily worked into the corners of the form.

NOTE 2—Using dry ingredients, a good portland cement concrete mix may be obtained by using sufficient water to give a ratio between weight of water and weight of cement of approximately 0.6 (± 0.1) to 1.0.

6.2 Place the previously coated shingle or sheet, coated-side up, within the forms. Place the wet concrete in the forms on top of the coated base with the steel trowel, work the mixture into the corners and next to the form. Strike off the top with a wooden screed and use a wooden or cork float or trowel to level off the surface and work to the desired texture. Finally, brush the surface evenly with the dry paint brush.

NOTE 3—If deemed desirable, the surface of the bond-coated base may be wetted prior to placing the concrete mix. If this is done, the water-cement ratio should be reduced to compensate, otherwise a wet and sloppy mix may result.

6.3 During the first hour of the setting period, gas may form between the bond coat and the concrete coat, causing bubbles to form upon the surface of the panel. These should be broken carefully or punctured by a slender, sharp instrument to permit the gas to escape. Any holes thus made may be smoothed over, or filled up, about an hour later.

6.4 After the concrete has set up sufficiently to permit removal of the forms from the panel, or the panel from the forms, smooth the edges to desired texture, and cover with a wet cloth.

6.5 Keep the panel covered with the wet cloth until seasoned to desired age, wetting the cloth thoroughly twice daily. To provide freedom from crazing or cracking, a minimum seasoning period of seven days is recommended.

6.6 Before using, bevel off top exposed edges with a carborundum stone, sufficiently to prevent edge-breakage in use.

7. Preparation of Masonry Panels

7.1 In the mixing box, prepare the masonry or stucco mix from 1 part of portland cement, $1\frac{1}{2}$ parts of masonry cement, and 3 parts of sand, by volume. Thoroughly mix these dry ingredients. Add water and mix thoroughly. Use only enough water to make the mortar sufficiently plastic as to be easily worked into the corners of the form.

NOTE 4—The addition of masonry cement to the stucco mix makes a more workable mix than using only portland cement, and the water-cement ratio may be reduced to 0.5 (± 0.1) to 1.0.

7.2 Proceed as described in Section 6.2 to 6.6.

8. Identification

8.1 Identify the back of each panel (shingle or asbestos cement sheet side) as to type of mix (concrete or masonry) and data of manufacture.

NOTE 5—For optimum reproducibility, panels used in the same test should be of the same age, cure and seasoning.

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Standard Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus¹

This standard is issued under the fixed designation D 1735; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the basic principles and operating procedures for testing water resistance of coatings in an apparatus similar to that used for salt spray testing.

1.2 This practice is limited to the methods of obtaining, measuring, and controlling the conditions and procedures of water fog tests. It does not specify specimen preparation, specific test conditions, or evaluation of results.

NOTE 1—Alternative practices for testing the water resistance of coatings include Practices D 870, D 2247, and D 4585.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- B 117 Method of Salt Spray (Fog) Testing²
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products³
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces³
- D 714 Test Method for Evaluating Degree of Blistering of Paints³
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion³
- D 1193 Specification for Reagent Water⁴
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁵
- D 2247 Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity³
- D 2616 Test Method for Evaluation of Visual Color Difference With a Gray Scale⁶

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Nov. 27, 1987. Published January 1988. Originally published as D 1735 - 60 T. Last previous edition D 1735 - 86a.

² Annual Book of ASTM Standards, Vols 03.02 and 06.01.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁵ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁶ Annual Book of ASTM Standards, Vol 14.02.

D 3359 Test Method for Measuring Adhesion by Tape Test³

D 3363 Test Method for Film Hardness by Pencil Test³

D 4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation³

3. Summary of Practice

3.1 Coated specimens are placed in an enclosed chamber where a water fog is sprayed on them. The temperature of the chamber is usually maintained at 100°F (38°C). The exposure condition is varied by selecting the duration of the test. Water permeates the coating at rates that are dependent upon the characteristics of the coating. Any effects such as color change, blisters, loss of adhesion, softening, or embrittlement are observed and reported.

4. Significance and Use

4.1 Water can cause the degradation of coatings, so knowledge of how a coating resists water is helpful in predicting its service life. Failure in water fog tests may be caused by a number of factors including a deficiency in the coating itself, contamination of the substrate, or inadequate surface preparation. The test is therefore useful for evaluating coatings alone or complete coating systems.

4.2 Water fog tests are used for research and development of coatings and substrate treatments, specification acceptance, and quality control in manufacturing. These tests usually result in a pass or fail determination but the degree of failure may also be measured. A coating system is considered to pass if there is no evidence of water-related failure after a specified period of time.

4.3 Results obtained from the use of water fog tests in accordance with this practice should not be represented as being equivalent to a period of exposure to water in the natural environment, until the degree of quantitative correlation has been established for the coating or coating system.

4.4 The test apparatus is identical to that used in Method B 117, and the conversion of the apparatus to water fog testing is not difficult. Corrosion tests of a scribed coating on a ferrous substrate are possible as the water that drips off of the specimens is not recirculated.

5. Apparatus

5.1 *Test Chamber, Test Specimen Supports, and Fog Collecting Devices*, as specified in Method B 117.

6. Test Specimens

6.1 This practice does not cover the preparation of test specimens. The substrate composition and surface preparation, specimen preparation, and the number of specimens

should be agreed upon.

NOTE 2—All substrates are Methods D 82 uniform films.

7. Procedure

7.1 Fill the Type III of S

7.2 Maintain 2°F (38 ± temperature

7.3 Support from the vertical the direction for flat specimens. In other, any acting as a one specimen

7.4 Place fog for measurement nozzle with place them: Glass funnels or dish mended.

7.5 Adjust of water/h i mm.

NOTE 3—T mL/h for a cc

7.6 Oper closed until

should be agreed upon prior to testing.

NOTE 2—Applicable methods for the preparation of test panels and substrates are given in Methods D 609 and Practices D 1730. Test Methods D 823 cover application techniques of the production of uniform films.

1. Procedure

7.1 Fill the reservoir with reagent water conforming to Type III of Specification D 1193.

7.2 Maintain the test chamber at a temperature of $100 \pm 2^\circ\text{F}$ ($38 \pm 1^\circ\text{C}$). Other temperatures may be used if the temperature is reported in Section 8.

7.3 Support or suspend specimens approximately 15° from the vertical with the plane of the specimen parallel to the direction of fog flow. Slotted wood supports are suitable for flat specimens. Provide for free settling of fog on all specimens. Space specimens so that they do not touch each other, any metallic material, or any material capable of acting as a wick. Arrange the specimens so that water from one specimen does not drip on other specimens.

7.4 Place two containers within the chamber to collect the fog for measurement. Locate one container near the fog nozzle with the other as far as possible from the nozzle and place them so that they collect only the fog from the nozzle. Glass funnels with the stem extending into graduated cylinders or dishes having a diameter of 100 mm are recommended.

7.5 Adjust the atomizing air supply so that 1.5 to 3.0 mL of water/h is collected in a collector with a diameter of 100 mm.

NOTE 3—The fog rate specified in Method B 117 is only 1.0 to 2.0 mL/h for a collector with a diameter of 100 mm.

7.6 Operate the test continuously with the test chamber closed unless otherwise specified. Short interruptions to

inspect or remove specimens or to replenish the water supply are permitted.

7.7 Conclude the test after a specified period of time or after effects from exposure to water fog are observed.

7.8 Wipe the test specimens dry. Rate specimens for changes in color, blistering, etc. Evaluate specimens no less than 5 min and no more than 10 min after removal from test, as the effects from water exposure can change within a short time. Remove only as many specimens as can be rated within the specified time.

NOTE 4—The 0 to 10 scale described in the ASTM STP 500⁷ is preferred for rating. Relevant procedures for evaluating water effects are described in Test Methods D 610, D 714, D 2616, D 3359, and D 3363.

7.8.1 If possible, rate the specimens again after they have been removed from the test for a recovery period long enough that moisture absorbed within the specimens dries out and the specimens reach moisture equilibrium with room air. A recovery period of 12 from 24 h is generally sufficient. The post-recovery rating allows evaluation of the permanent effects of the exposure as distinct from the transient effects, and is especially important for evaluation of color and gloss.

8. Report

8.1 Report the following information:

8.1.1 Sample identification.

8.1.2 Results of the evaluation(s).

8.1.3 Reference to Practice D 1735.

8.1.4 Hours of test duration.

8.1.5 Test temperature.

8.1.6 Rate of fog collection and pH of water collected.

8.1.7 Special conditions of test or any deviations in test procedure.

⁷ Paint Testing Manual, ASTM STP 500, ASTM, 1972.

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Standard Test Method for Efflorescence of Interior Wall Paints¹

This standard is issued under the fixed designation D 1736; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the tendency of an interior wall paint to effloresce under normal environmental conditions. Historically this phenomenon has been exhibited primarily with latex, or other water-based paints, and the method has been used in testing aqueous products.

1.2 This test method is limited to the determination of the efflorescence resulting from migration of soluble salts from within the paint film. It does not deal with the efflorescence attributable to certain substrates.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

- 2.1 *ASTM Standard:*
D 1193 Specification for Reagent Water²

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *efflorescence*—a condition that occurs when soluble salts in the dried paint film or in the substrate migrate to the film surface during exposure. It should not be confused with other causes of nonuniform appearance or whitening that may occur at the time of application. Efflorescence is seen as either a light, medium, or heavy deposits of crystals. It exists both in isolated patches and over wide areas. Another evidence of efflorescence is the development of a non-uniform appearance of flat or glossy areas during the cycling procedure of the test.

3.1.2 degrees of efflorescence:

3.1.2.1 *flattening and glossing*—A nonuniform decrease or increase in gloss noticeable when the surface is illuminated and viewed at near grazing angles. There is no apparent change in color when viewed perpendicularly to the surface.

3.1.2.2 *fine efflorescence*—A barely discernible whitening of the surface when viewed perpendicularly.

3.1.2.3 *medium efflorescence*—A readily noticeable whitening of the surface without a marked masking of the color.

3.1.2.4 *heavy efflorescence*—A white deposit sufficient to mask the color.

4. Summary of Test Method

4.1 Painted panels are exposed to controlled cycles of low temperature and high humidity, and to standard conditions of temperature and humidity, inducing a fine condensate on the surface. The test is run for a maximum of eight cycles.

5. Significance and Use

5.1 The test conditions are similar to conditions that produce efflorescence inside a home or building. The degree of efflorescence and the number of cycles required to produce it are a good indication of the paint's ability to resist efflorescence.

6. Apparatus

6.1 *Test Cabinet*—A cold-wall type of household refrigerator without exposed cooling coils. Additional humidity shall be introduced into the test area from a pan of water on the chamber floor, or by putting water in the butter conditioner and setting the conditioner at its highest temperature. Conditions in the test cabinet shall be maintained at $42 \pm 3^\circ\text{F}$ ($5.5 \pm 1.5^\circ\text{C}$) and at 75 to 80 % relative humidity. Any other type of cabinet used should be checked for reproducibility of test results by comparison with a recommended cabinet.

7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

8. Test Panels

8.1 A sealed substrate shall be used. One satisfactory surface is tempered hardboard sealed with one coat of shellac varnish (3 to 4-lb cut). The panel size shall be 6 by 18 in. (150 by 450 mm). If the panel does not fit the cabinet, the size may be changed, although the area should be kept approximately the same.

9. Preparation of Panels

9.1 Apply the shellac varnish to the test panel and allow to dry. Brush two coats of the test paint on both sides of the sealed panel. Apply the test paint at a temperature above 60°F (15.5°C) and allow approximately 1 h between coats or, if not a latex paint, allow an appropriate time for drying between coats. After applying the second coat, dry the panel for 1 h (or appropriate time) at the same temperature and observe for uniformity of appearance at all angles of viewing before placing in the test cabinet. If the panels are

nonuniform in panels. If the erred unaccept

10. Procedure

10.1 Hang spacing of 1 t for 23 h. Ther (25°C) and 50 (approximatel

10.2 Inspect an 85° angle c in surface app

10.3 Return 80 % relative steam treatme

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10.3.2 Rer over the pan soon as the f area return t should take c and the pane than 5 to 10

NOTE 1—Pl cabinet. The pa with the format test cabinet too

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved March 31, 1989. Published May 1989. Originally published as D 1736 - 60 T. Last previous edition D 1736 - 89(1984)¹.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

nonuniform in gloss or color, reject them and prepare new panels. If the condition persists, the paint should be considered unacceptable for testing.

10. Procedure

10.1 Hang the panels vertically in the test cabinet with a spacing of 1 to 2 in. (25 to 50 mm) between them and chill for 23 h. Then place them in a room at approximately 77°F (25°C) and 50 % relative humidity until the surface is dry (approximately 30 min).

10.2 Inspect for efflorescence by observing the panels at an 85° angle of incidence to a light source so that any change in surface appearance can readily be noted.

10.3 Return the panels to the test cabinet at 42°F (5.5°C), 80 % relative humidity for ½ h to chill before receiving a steam treatment as follows:

10.3.1 Heat ½ in. (13 mm) deep reagent water in a flat-bottom pan to boiling. Remove or reduce the heat until there is no bubbling.

10.3.2 Remove the panel from the cabinet and hold it over the pan until a fine condensate forms on the surface. As soon as the fine condensate covers about 75 % of the panel area return the panel immediately to the test cabinet. It should take only a few seconds to develop the condensate, and the panel should be replaced in the test cabinet in less than 5 to 10 s.

NOTE 1—Place the steam apparatus as near as possible to the test cabinet. The panel temperature will rise quite rapidly and will interfere with the formation and retention of condensate if the panel is out of the test cabinet too long. Formation of too large a condensate droplet will

cause water spots or rundown streaks and interfere with the development and rating of efflorescence.

10.4 Subject the panels to eight cycles, unless heavy efflorescence is observed in fewer cycles. The 23 h in the test cabinet followed by conditioning at standard temperature and humidity and by observation of the panels constitute the first cycle; the steaming, 23-h chilling, conditioning, and observation constitute subsequent cycles, with exposure in the test cabinet over the weekend constituting one test cycle. Start the test on Monday or Tuesday so that only one weekend cycle is included in the test series. Other end points may be established by agreement between the purchaser and the manufacturer.

11. Report

11.1 Report any glossing, flattening, light, medium, or heavy efflorescence, and the percent of the surface covered. When heavy efflorescence occurs in fewer than eight cycles, report the number of cycles required to produce this condition.

12. Precision

12.1 This test method was developed to provide a reliable qualitative and semiquantitative measure of efflorescence. Available information on the method is not sufficient to establish its reproducibility on a quantitative basis, so that a statistical statement of precision is not possible. However, the need for other than a reliable qualitative or semiquantitative rating is not apparent at this time; further studies will be undertaken if the need arises.

13. Keywords

13.1 efflorescence

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Standard Test Method for Elongation of Attached Organic Coatings With Cylindrical Mandrel Apparatus

This test method covers the determination by the cylindrical test apparatus of the elongation of attached organic coatings when applied to flat sheet metal of uniform surface texture.

Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials.

This test method was discontinued in 1988 and replaced by ASTM Test Methods D 522, for Mandrel Bend Test of Attached Organic Coatings.¹

¹ Annual Book of ASTM Standards, Vol 06.01.

1. Scope
1.1 This test method covers the determination of elongation of organic coatings that may be applied to flat sheet metal of uniform surface texture. Such procedures are described in this test method.

NOTE
failures
methods
linseed-o

2. Terminology
2.1 Definitions of terms relating to films and coatings are given in Terminology D 155.

¹ This test method was discontinued in 1988 and replaced by ASTM Test Methods D 522, for Mandrel Bend Test of Attached Organic Coatings. This test method was published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19104.



Standard Classification for Reporting Paint Film Failures Characteristic of Exterior Latex Paints¹

This standard is issued under the fixed designation D 1848; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This classification covers descriptions of properties that may be observed in weathered latex paint films. These descriptions should be used in reporting the condition of such paint films.

NOTE 1—In inspecting weathered exterior latex paints, defects or failures in the films may be observed that are not described in ASTM methods for evaluating the deterioration of exterior paints of the linseed-oil type.

2. Types of Defects

2.1 The following types of defects of exposed latex paint films are recognized:

2.1.1 *Efflorescence*, characterized by a commonly white, nonuniform powdery incrustation not readily removed by wiping. Usually it cannot be removed with water only but can be removed with dilute mineral acid.

2.1.2 *Mottling (or Blotching)*—The presence of irregularly shaped and randomly distributed areas of nonuniform appearance in color, gloss, or sheen.

2.1.3 *Water Spotting*—Nonuniformities in color, gloss, or sheen in the shape of spots or streaks, caused by drops or rivulets of water.

2.1.4 *Crawling*—Defect in which the wet film recedes from small areas of the substrate (usually caused by insufficient wetting) leaving those areas uncoated.

2.1.5 *Pinholing*—The presence of a series of fine holes or voids in the film.

2.1.6 *Mud-Cracking*, characterized by an irregular broken network of cracks in the film.

3. Index Terms

3.1 This standard is indexed under the following terms: exterior paints/coatings; latex paints.

¹ This classification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 1848 - 61 T. Last previous edition D 1848 - 63 (1984) ϵ 1.

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Standard Test Method for Package Stability of Paint¹

This standard is issued under the fixed designation D 1849; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 3018 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

¹NOTE—Editorial changes were made throughout in May 1987.

1. Scope

1.1 This test method covers the change in consistency and certain other properties that may take place when packaged paint of either the solvent-reducible or water-reducible type is stored at a temperature above freezing.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 562 Test Method for Consistency of Paints Using the Stormer Viscometer²

D 869 Test Method for Evaluating Degree of Settling of Paint²

3. Apparatus

3.1 *Spatula*, weighing 45 ± 1 g with square-ended blade $4\frac{3}{4}$ in. (120 mm) in length and approximately $1\frac{3}{16}$ in. (20 mm) in width as described in Method D 869.

3.2 *Viscometer*—A Stormer viscometer with the paddle-type rotor as described in Test Method D 562.

3.3 *Paint Brush*, 1-in. (25-mm) nylon.

3.4 *Test Surface*—A smooth-surfaced paper chart coated with a suitable varnish or lacquer so as to render the surface impervious and resistant to paint liquids.

4. Procedure

4.1 Obtain duplicate samples of the paint in original, unopened containers (Note 1). Examine one of the samples received for the characteristics listed in 4.2, using the procedures therein described. Weigh the other unopened sample to the nearest 1 g, then hold it undisturbed for specified periods of time and ranges of temperature, as agreed between purchaser and seller (Note 2). After storage, reweigh the sample without shaking to determine loss of weight resulting from faulty closure.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Aug. 1, 1980. Published September 1980. Originally published as D 1849 - 61 T. Last previous edition D 1849 - 79.

² Annual Book of ASTM Standards, Vol 06.01.

NOTE 1—Containers should preferably be no larger than 1 qt (1 L).

NOTE 2—Storage for 1 month at $125 \pm 2^\circ\text{F}$ ($52 \pm 1^\circ\text{C}$) simulates some of the effects of storage for 6 months to 1 year at $73 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$). However, it should be recognized that storage at 125°F may not simply accelerate changes occurring at 73°F ; with latex paints, for example, at 125°F the growth of some putrefying bacteria is inhibited.

4.2 Bring the stored sample to $73 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$). Note evidence of pressure or vacuum in the unopened container. Open the container and note skinning, corrosion and odor of putrefaction, rancidity or souring. Disregard other odors. If the sample is in a 1-qt (1-L) or smaller container, measure the character of the lower or settled layer with the spatula as described in Method D 869. If the sample is larger than 1 qt, omit this step. Hand stir the paint 300 stirs in 2 min with a spatula appropriate to the container, stirring so as to ensure uniform distribution of any settled material (Note 3). Immediately after stirring, measure the consistency of the paint as prescribed in Test Method D 562: without allowing the paint to reset after stirring.

4.3 Apply the stored paint to one test panel and the control paint to the other. After the brushed film has completely dried, examine it for grains $\frac{1}{32}$ in. (0.8 mm) in diameter, even larger gelatinous lumps, and streaks caused by such grains or lumps.

NOTE 3—Avoid the use of a mechanical shaker or syringe-type measuring device that may disperse these lumps.

5. Report

5.1 For the stored sample only, report the time of storage in days and the temperature of storage in degrees Fahrenheit or Celsius. Report the initial and final sample weight.

5.2 For both the sample tested as received and the stored sample, report the following:

5.2.1 Skinning, pressure, corrosion of the container, and odor of spoilage, each quality separately designated as follows:

- 10 = none
- 8 = very slight
- 6 = slight
- 4 = moderate
- 2 = considerable
- 0 = complete failure

5.2.2 Rigidity of the lower layer as determined in accordance with Method D 869, if performed;

5.2.3 Consistency as the weight in grams necessary to produce 200 r/pm on the Stormer viscometer;

5.2.4 (designate

10 =
8 =
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6. Preci

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5.2.4 Grains, lumps, or streaks in the brushed film, designated as follows:

10 = none	4 = moderate
8 = very slight	2 = considerable
6 = slight	0 = complete failure

6. Precision

6.1 *Empirical Ratings*—Reproducibility of empirical ratings by different operators cannot be predicted with reli-

ability because of the personal factor as, for example, in the judgement of odor.

6.2 *Lower Layer Rigidity*, ± 4 grades, within one laboratory.

6.3 *Stormer Consistency*, ± 60 g, within one laboratory.

7. Index Terms

7.1 This test method is indexed under the following terms: package stability; stability—package.

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Standard Guide for Analysis of Electrocoat Bath Samples¹

This standard is issued under the fixed designation D 1978; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the selection of test methods for determination of the important parameters that affect the performance of electrocoating paints.

1.2 The test methods involved are D 4370, D 4399, D 4584, and D 5145.

2. Referenced Documents

2.1 ASTM Standards:

D 4370 Test Methods for Acid and Base Milliequivalent Content of Electrocoat Baths²

D 4399 Test Method for Measuring Electrical Conductivity of Electrocoat Baths²

D 4584 Test Method for Measuring Apparent pH of Electrocoat Baths²

D 5145 Test Method for Nonvolatile and Pigment Content of Electrocoat Baths²

3. Significance and Use

3.1 This guide indicates test procedures recommended for the maintenance of acceptable performance of the paint in an electrocoating bath. Several critical parameters must be

determined throughout the operation of the bath. These parameters must be adjusted when deviations from the norm occur.

3.2 The test methods for electrocoat baths are unique, as the aqueous samples have a nonvolatile content between 8 and 25 %. Constant agitation must be present when the samples are taken and during the measurement of some of the parameters.

4. Test Methods

4.1 *Acid and Base Content*—Test Methods D 4370 covers the determination of acid and base milliequivalent content of electrocoat baths.

4.2 *Electrical Conductivity*—Test Method D 4399 describes the determination of the electrical conductivity of electrocoat baths.

4.3 *pH Determination*—Test Method D 4584 describes the measurement of the apparent pH of paints and ultrafiltrates of electrocoat baths.

4.4 *Nonvolatile and Pigment Content*—Test Method D 5145 covers the determination of nonvolatile and inorganic pigment content of electrocoat baths.

5. Precision

5.1 The referenced test methods have precision limits listed. Reference to the individual standards for precision statements is recommended.

6. Keywords

6.1 electrical conductivity; nonvolatile content; electrocoat baths; pH; pigment content

1. Scope

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¹ This test meth and Related Coatir Subcommittee D01 Current edition
² Annual Book
³ Paint/Coating: Coatings Technolog

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Materials.

Current edition approved Feb. 22, 1991. Published April 1991.

² Annual Book of ASTM Standards, Vol 06.01.

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Standard Test Method for Print Resistance of Architectural Paints¹

This standard is issued under the fixed designation D 2064; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers an accelerated procedure for evaluating the print resistance of architectural paints. It differs from print resistance Test Method D 2091 in that the latter is concerned with lacquer finishes under packaging, shipping, and warehousing conditions, whereas this test method is concerned with decorative coatings undergoing random on-site pressure contact.

NOTE 1—Printing should not be confused with blocking, which is measured in Test Method D 4946. The former relates to the indentation of a surface, and the latter, the sticking together of two surfaces.

2. Referenced Documents

2.1 ASTM Standards:

D 2091 Test Method for Print Resistance of Lacquers²
D 4946 Test Method for Blocking Resistance of Architectural Paints²

3. Terminology

3.1 Definitions:

3.1.1 *print resistance*—the ability of a paint film to resist forming a permanent impression in a semihardened film as a result of a pressure from an object placed on it.

3.2 For additional definitions of terms, refer to *Paints/Coatings Dictionary*.³

4. Summary of Test Method

4.1 A piece of cheesecloth is placed on the painted surface and topped with a No. 8 rubber stopper (position smaller diameter of stopper on the cheesecloth) and a 500-g weight to create a pressure of about 0.9 psi (6.2 kPa). This assembly is placed in a 140°F (60°C), or other agreed upon temperature, oven for 1 h. When cooled, the resulting paint surfaces are rated on the numerical scale of 0 to 10, which corresponds to a subjective rating of an impression resulting from their being in contact with the cheesecloth.

5. Significance and Use

5.1 The ability of a coating to resist printing is important because its appearance is adversely affected if the smoothness of the coating film is altered by contact with another surface, particularly one with a texture. Interior paint systems, particularly gloss and semigloss on window sills and other

horizontal surfaces, often have objects such as flower pots placed on them that may tend to leave a permanent impression. This tendency for a paint film to “print” is a function of the hardness of the coating, the pressure, temperature, humidity, and the duration of time that the object is in contact with the painted surface.

6. Apparatus

6.1 *Conditioning Room*, at 65 to 85°F (18 to 29.5°C) and 40 to 60 % relative humidity.

6.2 *Glass Slides*, approximately 3 by 6 in. (75 by 150 mm).

6.3 *Drawdown Applicator*, 3-in. (75-mm) wide with a clearance of 6 mils (150 μ m).

6.4 *Oven*, maintained at 140 $\pm$ 3.5°F (60 $\pm$ 2°C) or, other agreed upon temperature.

6.5 *Rubber Stoppers*, No. 8 (40-mm top diameter and 32-mm bottom diameter).

6.6 *Cheesecloth*, medium weave.⁴

6.7 *Scissors*.

6.8 *Weight*, 500-g.

7. Procedure

7.1 Cast the paints being tested on glass slides using a drawdown applicator with a clearance of 6 mils (150 μ m). Allow the coated glass slides to dry in the conditioned room for 7 days.

7.2 Cut out approximately 1½ by 1½-in. (40 by 40-mm) pieces of cheesecloth, one piece for each slide. Use cheesecloth that is usually supplied with 4 intact layers. Do not separate the layers.

7.3 Place the test slides on a horizontal tray, painted face upward, then place one piece of cheesecloth over each slide and top with the small diameter face of a No. 8 stopper and a 500-g weight, one weight and stopper for each specimen. (Weights and stoppers should be equilibrated in the oven prior to running the test.) It is recommended that control paints of known satisfactory and unsatisfactory performance be used in each test run and that the tests be run in duplicate.

7.4 Place the entire assembly in the oven and remove it after exactly 1 h. Immediately lift off the weights and stoppers without disturbing the cheesecloth and allow to cool.

7.5 After approximately ½ h, remove the cheesecloths and carefully examine the appearance of the films underneath. Note the depth and amount of cheesecloth pattern left in the paint film. If necessary examine the panel, for better

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and Devices and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved May 15, 1991. Published July 1991.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Paint/Coatings Dictionary*, available from the Federation of Societies for Coatings Technology, Blue Bell, PA, 1978.

⁴ Idealfold bleached cotton cheesecloth, Grade 20 B, Weave 24/20, 36-in. wide, 70 yd² available from Hermitage Industries, Inc., Camden, SC 29020, or an equivalent may be used.

perception of indentations, by viewing it at a low (grazing) angle of illumination.

8. Interpretation of Results

8.1 Rate print resistance is rated on a numerical scale of 0 to 10 corresponding to a subjective rating of the degree to which the paint film has been imprinted. Record the numerical value for print resistance in accordance with the following ASTM style rating or scoring system:

Numerical Rating	Qualitative Characterization (Resistance to Printing)	Quantitative Description (Amount of Printing)
10	perfect	none
8	very good	very slight
6	good	slight
4	fair	moderate
2	poor	considerable
0	very poor	very great

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9. Report

9.1 Report the print resistance rating determined in accordance with 8.1. Specify temperature used.

10. Precision

10.1 Data are unavailable for a conventional intra- and interlaboratory precision statement. However, based on actual laboratory experience, with experienced operators, the repeatability is estimated to be plus or minus one unit. The employment of common control paints with mutually agreed ratings should make it possible for interlaboratory agreement (reproducibility) to approach intralaboratory agreement (repeatability). Although numerical ratings may differ with different operators or at different times, ranking should be about the same. As in many tests the precision improves with practice.

11. Keyword

11.1 print resistance



Standard Test Method for Determination of Edge Performance of Composite Wood Products Under Surfactant Accelerated Moisture Stress¹

This standard is issued under the fixed designation D 2065; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method is intended to serve as a means for measurement of swelling and cracking of the coated or uncoated edge of a composite wood substrate that has been subjected to wetting by a test solution containing surface active agent.

1.2 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *crack rating*—a numerical value on a scale from 1 to 10, with 10 representing no evidence of cracking of the edge (see Test Method D 661).

3.1.2 *composite wood products*—boards or other structural or decorative materials manufactured from wood fibers, flakes or strands and various resin binders consolidated under heat, or pressure, or both.

4. Summary of Test Method

4.1 A surfactant solution is placed in a tray that contains a sponge. A preweighed and calipered panel with a coated edge is placed with the edge in contact with the surfactant saturated sponge for 2 h. The exposed panel is blotted dry, reweighed, recalipered, and inspected for edge cracking. Percentage weight and thickness changes are calculated and recorded. Edge cracking is rated in accordance with Test Method D 661 and recorded.

5. Significance and Use

5.1 This test method provides a measure of the dimensional stability and integrity of the coated edge of various composite wood products under accelerated moisture stress. It is widely used as an indicator or predictor of the anticipated performance of composite wood products during

exterior exposure. It may be used for developmental evaluation of coatings, substrates, or both. It may also be useful for quality control or monitoring of the production of coated or uncoated composite wood products.

6. Apparatus

6.1 *Wide Flat Tray or Pan*, suitable to contain the required number of sponges and test boards. The sides must be high enough to safely contain at least 1/4 in. (6 mm) of solution.

6.2 *Rack or Holder*, suitable to support the required number of test boards at the nominal 60° angle.

6.3 *Micrometer or Vernier Caliper*, of sufficient capacity for the board thickness to be measured and capable of 0.1 mm accuracy.

6.4 *Balance*, with sufficient capacity (typically 400 g) and 0.01 g accuracy.

6.5 *Suitable Saw*, for cutting of samples.

6.6 *Common Household Cellulose Sponges*, cut to suitable size.

7. Materials

7.1 A sufficient supply of test solution.

7.2 Appropriately identified test boards cut to proper size.

8. Hazards

8.1 When using saws, wear goggles, dust mask, and use proper machine safeguards to prevent injury.

9. Procedure

9.1 See Fig. 1.

9.2 Make the required test solution.³ Pour solution into the tray to saturate all sponges, and allow to reach room temperature. A 1/4-in. (6-mm) depth of solution in the tray works well.

9.3 Cut board specimens to a suitable size. A 4 (10.2) by 5-in. (12.7-cm) size has been found to be convenient. The 4-in. (10.2-cm) dimension should be the coated edge. Preparation of triplicate specimens is recommended.

9.4 Measure thickness of the samples along the edge to be tested. Three measurements at 1-in. (2.54-cm) intervals (Fig. 2) are recommended for a 4-in. (10.2-cm) specimen. Record results.

9.5 Weigh the samples to the nearest 0.01 g and record.

9.6 Place boards in the tray on sponges as noted in Fig. 1. If coated edges are to be tested, note that the sponges must be

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.52 on Factory-Coated Wood Building Products.

Current edition approved May 15, 1991. Published July 1991.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Common test solutions such as 1.0 % TRITON® X-100 available from Rohm and Haas Co., Independence Mall West, Philadelphia, PA 19105 or equivalent, is made using warm, (over 120°F (49°C)), distilled, or deionized water.

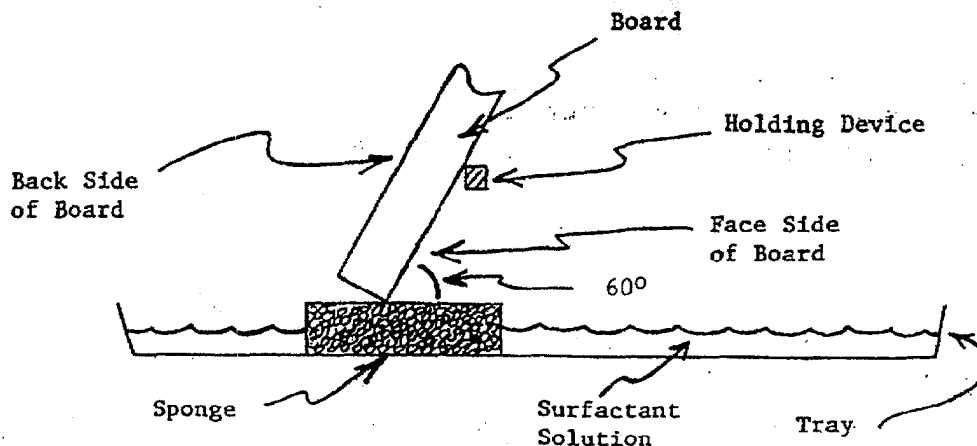


FIG. 1 Test Apparatus Side View

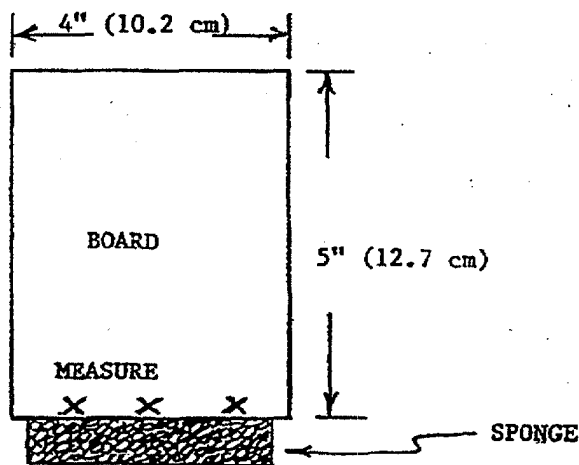


FIG. 2 Specimen Dimensions

narrower than the length (nominally 4 in. or 10.2 cm) of the contacting coated edges to prevent contact of the solution with the uncoated edges of the samples. Sponges 3.5-in. (9.0-cm) wide have been used successfully. For the same

reason, the coated face of the board must rest against the sponge at a 60° angle to prevent contact of the solution with the uncoated back of the board.

9.7 The test duration is 2 h. After 2 h, remove the boards and blot the edges dry with paper towels.

9.8 Observe and record changes as follows:

9.8.1 Visually inspect the edges for cracks and record the crack rating in accordance with Test Method D 661. Repeat the observation 48 h later.

9.8.2 Remeasure the thickness of the boards at the same spots measured in 4.4. Record the percent change at each spot.

9.8.3 Reweigh the boards and record the weight gain and percent weight gain, and

9.8.4 Calculate averages and standard deviations for the percent thickness change and percent weight gain.

10. Precision and Bias

10.1 The precision and bias of this test method have not been fully determined. Future round robins are planned.

11. Keywords

11.1 accelerated moisture stress; detergent test; wood or composite-wood

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Standard Test Methods for Relative Tinting Strength of Printing Ink Dispersions¹

This standard is issued under the fixed designation D 2066; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 These test methods cover procedures for determining the relative tinting strength of printing ink dispersions by visual or instrumental evaluation.

1.2 These test methods are applicable to paste-type printing inks, flushed pigments, and other pigment dispersions that are essentially nonvolatile under ordinary room conditions and for which there is a wet reference standard of the same pigmentation and consistency. With proper choice of tinting base, they are applicable to dispersions of any color, including black and white.

NOTE 1—The instrumental procedures for tinting strength are similar in principle to those described in Test Methods D 387, D 2745, and D 4838.^{2,3}

1.3 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products⁴

D 387 Test Method for Color and Strength of Colored Pigments with a Mechanical Muller²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates³

D 2745 Test Method for Instrumental Tinting Strength of White Pigments²

D 4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints³

E 284 Definitions of Terms Relating to Appearance of Materials⁵

E 1331 Test Method for Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry⁵

E 1347 Test Method for Color and Color-Difference Measurement of Object-Color by Tristimulus (Filter) Colorimetry⁵

E 1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry⁵

2.2 ANSI Standards:

PH 2.17 Geometric Conditions for Reflection Density⁶

PH 2.18 Spectral Conditions for the Measurement of Optical Density⁶

PH 2.30 Viewing Conditions for Graphic Arts and Photography—Color Prints, Transparencies and Photomechanical Reproductions⁶

3. Terminology

3.1 Definitions relating to color attributes and color differences are covered in Terminology D 16 and E 284.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *tinting strength*—the ability of a material to impart its color to a standard base; the reciprocal of the relative concentration required to match the reference material in a standard base.

3.2.2 *masstone (or masscolor)*—the color of a material that is thick enough to mask any background.

3.2.3 *undertone (or undercolor)*—the color of a thin film of a material.

4. Summary of Test Methods

4.1 Thin and thick films of the standard and unknown dispersions are drawn down in juxtaposition on bond and on coated paper. Visual evaluation of the relative undertone and masstone provides a check on color equivalency.

4.2 The standard and unknown dispersions are each reduced to the same concentration in a suitable tinting base. Thick wet drawdowns of the two tints are evaluated for tinting strength by Test Methods A or B.

4.2.1 *Test Method A—Visual Evaluation*: If the strength of the tints is judged unequal, aliquots of the stronger tint are further reduced until equivalence is obtained. The tinting strength of the unknown dispersion is calculated from the weight of extra tinting base added per unit weight of the stronger tint.

4.2.2 *Test Method B—Instrumental Evaluation*: Reflectance measurements are made on thick wet films of the original tints. The tinting strength of the unknown dispersion is calculated according to the Kubelka-Munk equation.

4.3 Preparation of a confirming tint is recommended as an unbiased method of verification. The preferred approach is to prepare a new tint of the unknown at a concentration calculated to match the standard tint.

5. Significance and Use

5.1 Tinting strength is an essential property of printing ink dispersions. Although test results on wet drawdowns and

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D1.56 on Printing Inks.

Current edition approved May 15, 1991. Published July 1991.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Available from American National Standards Institute, 13th Floor, 11 W 42nd St., New York, NY 10036.

tints do not guarantee equivalency of dry printed ink films, they provide useful parameters for quality assurance of established formulations, gaging relative degree of dispersion, and estimating the color value of colorants from different batches, sources, or grades.

6. Apparatus

6.1 *Laboratory Balance*, sensitive to at least 1.0 mg, preferably 0.1 mg.

6.2 *Spatulas*, (2) with flexible blades 80 to 120 mm in length (for weighing and mixing).

6.3 *Mixing Surface*, such as a glass or similar slab fixed to a work bench.

6.4 *Putty Knife*, with an 80–120 mm wide blade having a smooth straight edge (for use as a drawdown blade).

6.5 *Standard Daylight*, preferably a D50 light source conforming to ANSI Standard PH 2.30.

6.6 *Reflectance Measuring Instrument*, (for instrumental evaluation). Unless otherwise agreed upon, the instrument shall be a spectrophotometer with hemispherical (integrating-sphere) geometry conforming to Test Method E 1331, a spectrophotometer with bidirectional (45/0 or 0/45) geometry conforming to Test Method E 1349, or a tristimulus (filter) colorimeter with either geometry conforming to Test Method E 1347. Alternatively, a reflection densitometer conforming to ANSI Standard PH 2.17 and having a set of Status T or Status E filters⁷ (see 12.3.2), conforming to ANSI Standard 2.18 may be used for certain colors.

NOTE 2—The filter systems in typical densitometers are suitable only for use with black, white, and the three process colors (yellow, magenta and cyan). Instrumental evaluation of other colors requires a spectrophotometer or a colorimeter.

7. Materials

7.1 *Reference (Standard) Dispersion*, having the same pigmentation and consistency as the test (unknown) dispersion.

7.2 *Tinting Base*, as agreed upon between the producer and user, consisting of a suitable pigment well dispersed in a vehicle that is compatible with the vehicle in the test dispersion. The consistency of the base should not be appreciably lower than that of the test dispersion. Driers are not generally used because they may affect the color of the base and corresponding tints.

7.2.1 *White Base*,⁸ for testing colored and black dispersions. A suitable white base may contain by weight 60 % of either zinc oxide or titanium dioxide and 40 % vehicle.

7.2.2 *Black Base*, for testing white dispersions. A suitable black base may contain by weight 4 % black pigment (preferably non-flocculating), 43 % precipitated calcium carbonate, and 53 % vehicle. Alternatively, a neutral black nondrying printing ink such as a news ink.

7.2.3 *Dark Blue Base* (optional), for visual testing of white dispersions. A suitable dark blue base may contain by weight

42 % ultramarine blue, 18 % precipitated calcium carbonate, and 40 % vehicle.

7.2.4 *Light Blue Base* (optional), for visual testing of yellow dispersions. A suitable light blue base may contain by weight 1 % phthalocyanine blue dispersion and 99 % white base.

NOTE 3—Mixtures of a light blue base with yellow samples produce green tints, differences between which are more easily detected by eye than are mixtures of white and yellow. However, false results may be obtained. The use of a blue base is not recommended for visual tests on greenish-yellow colorants and is not permitted for instrumental evaluation of any yellow colorant.

7.3 *Weighing Substrate*, nonabsorbent, such as skin paper⁹ or small glass plates ca 75 to 100 mm square.

7.4 *Drawdown Substrates*, one consisting of white bond paper at least 50-mm wide and 150-mm long with a black bar at least 20-mm wide imprinted across the short dimension about half way down the length of the sheet, and a second one consisting of white coated paper.¹⁰

7.5 *Microscope Cover Glasses*¹¹ (optional, for instrumental measurements), made of finest optical glass, 50 by 45 mm, 0.13- to 0.17-mm thick.

7.6 *Standard Spacer* (optional, for instrumental measurements), such as cardstock the same size as the cover glass described in 7.5, about 1-mm thick, with a 35-mm diameter hole.

8. Sampling

8.1 These test methods do not include a method for preparation of dispersions. If colorants from different batches or sources are being evaluated, it is important that the standard and unknown samples be dispersed either in the identical manner or to the maximum degree, as agreed upon between the producer and the user.

8.2 Carefully select a dispersed sample that is free of skin and other contamination and representative of the lot being evaluated. Transfer to a clean container, protect with skin paper, close and seal.

9. Evaluation of Masstone and Undertone for Relative Color

9.1 Using the bond paper with the black bar, place small portions of the standard and unknown dispersions close together, but not touching, in the center at one end of the sheet in the long dimension.

9.2 Place the blade of the drawdown knife behind the pastes and, using heavy pressure, draw down a thin film of the pastes in juxtaposition. When the middle of the black bar is reached, raise the blade slightly and draw down the remaining pastes in a layer sufficiently thick that the black bar is not visible. Remove excess material.

9.3 Immediately examine the drawdowns under the standard D50 light or other agreed upon light source. Judge

⁹ Skin paper available from NAV Pack, 18 Monterey Lane, Englishtown, NJ 07726 or equivalent, has been found suitable for this purpose.

¹⁰ Bond paper with a black bar (Form 3NT-4) available from The Leneta Co., P.O. Box 86, Hahokus, NJ 07423 or equivalent, has been found suitable for this purpose.

¹¹ Microscope cover glasses available from Fisher Scientific Co. (catalogue no. 12-545H) or other scientific supply houses have been found suitable for this purpose.

⁷ Status T filters are available in the USA, Status E in other countries. The major difference is in the peak transmission of the blue filter.

⁸ A spectrophotometrically controlled universal white bleach, containing by weight 60 % zinc oxide (Florence White Seal) and 40 % Herculyn-D plasticizer, is available from National Printing Ink Research Institute (NPIRI), Lehigh University, Sinclair Laboratory 7, Bethlehem, PA 18015.

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the hue, depth, cleanliness, transparency and other properties of the unknown dispersion relative to the standard dispersion. Record qualitative observations of the thin film over white paper as the relative undertone, the thin film over the black bar as the relative transparency, and the thick film as the relative masstone.

9.4 Repeat 9.1 and make a tight drawdown on a sheet of coated paper. Make an immediate visual judgment of the relative undertone. Include relative gloss and bronzing in the evaluation.

NOTE 4—When the consistencies of the standard and unknown dispersions are significantly different, the film thicknesses of the tight drawdowns may not be comparable. In such cases, judgments regarding relative hue should be reserved until the tints are examined (see Note 8 in 11.6).

NOTE 5—If the hue or cleanliness of the test dispersion is significantly different from the standard dispersion, tinting strength cannot be tested by the procedures covered in this test method. A numerical assessment of such systems may be obtained by making color measurements according to Test Methods E 1331, E 1347, or E 1349 and calculating color differences by the 1976 CIELAB equations in accordance with Test Method D 2244.

10. Preparation of Tints

10.1 Select a tinting base appropriate to the sample being tested (see 7.2). Examine the base for uniformity. If there are signs of separation or settling, stir thoroughly in container. If necessary, transfer the quantity required for testing to a slab and mix to ensure that the same composition of base will be used for both the standard and the unknown samples.

10.2 Tare or counterbalance a weighing substrate. Using guidelines suggested in Table 1, weigh out the desired amount of the standard dispersion. The quantity of specimen need not be exactly as listed in Table 1 but must be weighed to at least three significant figures. Divide the actual weight by the desired decimal concentration to obtain the total tint weight. The difference between the total weight and the specimen weight represents the weight of bleaching base to be added.

10.3 Gently mix the specimen and tinting base on the weighing substrate until the tint is uniform. Use a circular stirring motion, periodically scraping all material from the surface of the substrate. *Do not use so much energy that further dispersion will result.* If necessary, transfer all mate-

TABLE 1 Suggested Tint Concentrations for Strength Testing of Printing Ink Dispersions^a

Type of Dispersion	Dispersion Concentration in Tint	Ratio Dispersion	Content of Tint, g		
			Dispersion	Tinting Base	Total
Flush or concentrate	0.01	1:99	0.05	4.95	5.0
Process color ink	0.02	1:49	0.10	4.90	5.0
Laked or low strength color	0.05	1:19	0.25	4.75	5.0
Titanium dioxide					
with lamp black base	0.85	6:1	4.25	0.75 ^b	5.0
with carbon black base	0.98	49:1	4.90	0.10 ^c	5.0

^a In NPIRI Universal Bleaching Base except where noted; Figures are given as a guide. It is recommended that standard batches be checked first to establish tint concentrations that give proper lightness levels.

^b Materials should be weighed to three significant figures. Increase weights by a factor contingent on the balance sensitivity.

^c Use double the quantity for instrumental tinting strength conducted by spectrophotometry and confirmed by aliquot reduction.

^d For white dispersions, weigh tinting base first.

rial to a glass slab and continue mixing with a gentle scraping and stirring motion until a uniform color *with no specks or streaks* is achieved. With a clean putty knife, push the tint to one side of the slab. Clean the putty knife and remainder of the slab.

NOTE 6—With flushes and other high viscosity dispersions, it is recommended that the tinting base be mixed into the specimen in small increments.

10.4 Repeat 10.2 and 10.3 with the unknown dispersion. Be sure the specimen concentration in the tint and the type of tinting base are identical to that used for the standard dispersion.

10.5 If there will be a delay in the evaluation process, transfer the tints to small clean containers and label appropriately. Always gently restir immediately before subsequent use in order to minimize problems of flooding or floating.

TEST METHOD A—TINTING STRENGTH BY VISUAL EVALUATION

11. Procedure

11.1 Using separate ink knives, gently stir the standard and the test tints. Place a small quantity of each tint close together, but not touching, at one end of a small glass plate or other drawdown substrate. Hold the drawdown knife at a low angle (5 to 15° from horizontal) and, using light pressure, draw down the tints in juxtaposition. The two films must be in contact with each other, smooth, and sufficiently thick so as to mask any background.

11.2 Immediately examine the drawdowns under the standard light. If the two tints appear equal, record the tinting strength of the unknown as 100 %. If the tints are unequal in strength, estimate the strength difference between the stronger and weaker color either from experience or from instrumental measurements (see Eq 4a or 4b in 13.2.2).

NOTE 7—With colored and black samples, the stronger tint will be darker. With white samples, the stronger tint will be lighter.

11.3 Weigh to three significant figures an aliquot of about 1 g (or a quantity representing about 10 to 20 %) of the stronger tint. Multiply the exact weight by the estimated strength difference in decimal units; add tinting base accordingly. For example, for an estimated 10 % difference, add 0.10 g base/g aliquot of the stronger tint.

11.4 Gently mix the adjusted tint until uniform. Gently remix the original tint of the weaker dispersion, make a thick drawdown versus the adjusted tint as in 11.1, and examine as in 11.2.

11.5 If the drawdowns are still unequal, *discard* the adjusted tint. Weigh out a new aliquot of the stronger tint and add more or less tinting base than in 11.3.

11.6 Repeat 11.3 and 11.4 until the drawdowns show that the adjusted tint equals the strength of the lighter tint. When equivalency is obtained, record whether the standard or unknown tint was stronger, the weight of the final aliquot, and the weight of added tinting base.

NOTE 8—If there is a difference in color between the unknown and standard dispersions, a situation will result wherein, as dilution progresses, the darker tint will revert to the lighter tint without obtaining a match. In such cases, this method cannot be used (see Note 5).

11.7 Compute the strength of the unknown dispersion (u)

as a percentage of a standard dispersion(s) as follows:

$$TS_u \% = \frac{1 + (b/a)_u}{1 + (b/a)_s} \times 100 \quad (1)$$

where:

TS_u = tinting strength of the unknown dispersion,
 b = weight of extra tinting base added to an aliquot of the stronger tint to obtain equivalence, g, and
 a = weight of the aliquot, g.

The term b/a represents the strength difference between the stronger and weaker colorant. For the weaker dispersion, $b/a = 0$ and drops out of Eq 1. When the unknown dispersion is stronger, Eq 1 reduces to:

$$TS_u \% = 1 + (b/a)_u \times 100 \quad (1a)$$

When the standard dispersion is stronger, Eq 1 reduces to:

$$TS_u \% = \frac{1}{1 + (b/a)_s} \times 100 \quad (1b)$$

NOTE 9—Tinting strength is always expressed as a decimal or a percentage of the unknown relative to the standard. The practice of expressing results as a strength difference may lead to erroneous calculations of the replacement concentration. See Eq 3 in 13.1.

11.8 Since replication of visual tinting strength tests inherently suffers from bias, prepare a confirming tint in accordance with the procedure given in 13.1.

TEST METHOD B—TINTING STRENGTH BY INSTRUMENTAL EVALUATION

12. Procedure

12.1 Set the instrument for the large area of view or illumination and standardize in accordance with Test Methods E 1331, E 1347, or E 1349, or, in the case of a densitometer, the manufacturer's instructions. If it is the intent to make measurements directly on wet tints, it may be useful to protect the instrument with a material such as plastic wrap with the porthole cut out.

12.2 Gently remix the standard tint prepared in Section 10. Place a sufficient quantity on a small glass plate or other rigid surface so that the material is at least 30 to 35 mm in diameter and thick enough to mask any background. Alternatively, use a standard spacer (see 7.6) to prepare a thick sandwich between two microscope cover glasses.

12.3 Measure the reflectance factor in one of the following manners:

12.3.1 *Spectrophotometer*: Following the procedure given in Test Method E 1331 or E 1349, quickly mount the tint on the porthole of the spectrophotometer and, within or at 45 s, measure the reflectance factor between 420 and 680 nm. If hemispherical geometry is used, the specular component may be either included or excluded, as long as the same condition is consistently used. Make a minimum of two measurements, moving or rotating the specimen between runs. Record the spectral reflectance factor in decimal units at the wavelength of maximum absorption (minimum reflectance) and compute the mean.

12.3.2 *Densitometer*: If the tint involves black, white or a process color, select the filter having the appropriate Status T or Status E response in accordance with ANSI Standard PH 2.18. The peak transmission of the visual response filter should be at 555 nm for blacks and whites; of the blue filter, at 460 nm (Status T) or 440 nm (Status E) for process

yellows; of the green filter, at 530 nm for magentas; of the red filter, at 600 nm for cyans. Make measurements as in 12.3.1 at two or three different locations. If the readout is density, convert to the reflectance factor as follows:

$$R = 10^{-D}$$

If the readout is percent reflectance, record in decimal units.

12.4 Alternatively, measure the CIE tristimulus values of the specimen on a spectrophotometer in accordance with Test Method E 1331 or Test Method E 1349 or on a tristimulus colorimeter in accordance with Test Method E 1347. Make the measurements as in 12.3.1. If hemispherical geometry is used, the specular component may be either included or excluded as long as the same condition is consistently used. The tristimulus values may be based on either the CIE 1964 (10°) supplementary standard observer and standard illuminant D₆₅ or the CIE 1931 (2°) standard observer and standard illuminant C, as long as the same basis is consistently used. Record in decimal units the lowest appropriate value, for example, X with blue colors, Y with reds, blacks, and whites, or Z with yellows.

12.5 Repeat 12.2 and 12.3 or 12.4 with the unknown tint.

12.6 Calculate the tinting strength of the unknown dispersion according to the Kubelka-Munk equation as follows:

$$TS_u \% = \frac{[(1 - R_{\infty})^2 / 2R_{\infty}]_u}{[(1 - R_{\infty})^2 / 2R_{\infty}]_s} \times 100 \quad (2)$$

where:

R_{∞} = spectral reflectance factor, expressed as a decimal fraction, of an infinitely thick layer of material (at the wavelength of maximum absorption), or, by mutual agreement, an appropriate tristimulus value.

NOTE 10—If the tint represents a white pigment, use Eq 2 in the inverted form.

NOTE 11—The term $[(1 - R_{\infty})^2 / 2R_{\infty}]$ represents K/S of the colorant, where K is the absorption coefficient and S is the scattering coefficient, both of which are specific to a colorant. Therefore, if the pigmentation in the unknown dispersion is different from that in the standard dispersion, that is, the dispersions are metameric, Eq 2 no longer applies. The equation is also reported to work best when the reflectance factor or tristimulus value of the tints used is about 0.40 and the tinting strength of the unknown is within 10 % of the standard.

12.7 If the tinting strength result for the unknown is not within 10 % of the standard, reduce an aliquot of the stronger tint by the procedure given in 13.2. Remeasure the adjusted tint and calculate a new tinting strength value (see 13.2.5).

12.8 Prepare a confirming tint (see 13.1) if the original tinting strength value was not within 10 % of the standard (see 13.1 or 13.2 if within 10 %).

13. Preparation of Confirming Tint

13.1 Replacement Concentration:

13.1.1 In this method of confirmation, a new tint of the unknown dispersion is prepared at a concentration calculated to match the standard tint.

13.1.2 Compute the replacement concentration, also called color value, as follows:

$$C_u = C_s / TS_u \quad (3)$$

where:

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C_u = concentration of the unknown dispersion required to match the standard dispersion,
 C_s = concentration of the standard dispersion in the original tint prepared in 10.2, and
 TS_u = tinting strength result for the known dispersion in decimal units.

13.1.3 Weigh out a quantity of the unknown dispersion similar to that employed in 10.2. Divide the actual weight by C_u to obtain the total tint weight. The difference between the total and the specimen weight represents the weight of tinting base to be added. Mix as in 10.3.

13.1.4 Gently remix the original tint of the standard dispersion. Make a thick drawdown of both tints as in 11.1. If the two tints match, the tinting strength result computed for the unknown dispersion is correct.

13.1.5 Failure of the two tints to match suggests a weighing error or inadequate mixing of the tints. Clean up and start over from Section 10.

13.1.6 Use Eq 3 to compute the color/money/value of a colorant from different sources or grades (optional). Multiply C_u and C_s by their respective unit costs. Add to each the costs of other components in a formulation, represented by $(1 - C_u)$ and $(1 - C_s)$. The lower total cost figure is the better value.

13.2 Reduction of the Stronger Tint:

13.2.1 In this method of confirmation, the instrumental tinting strength result is used to calculate the strength difference between the stronger and weaker dispersion. An aliquot of the stronger tint is reduced accordingly.

13.2.2 Compute the strength difference, b/a by re-arrangement of Eq 1a or 1b. If the test dispersion is the stronger, that is, TS_u in decimal units is greater than 1.0 then:

$$(b/a)_u = TS_u - 1 \quad (4a)$$

If the standard dispersion is the stronger, that is, TS_u is less than 1.0 then:

$$(b/a)_s = (1/TS_u) - 1 \quad (4b)$$

13.2.3 Weigh out an aliquot of the stronger tint as in 11.3. Multiply the exact weight by b/a to obtain the weight of bleaching base to be added. Mix the adjusted tint until uniform.

13.2.4 Evaluate the adjusted tint by either of the following procedures:

13.2.4.1 *Visual Evaluation*—Draw down the adjusted tint versus the original tint of the weaker color as in 11.1.4. Failure to obtain equivalency on the first cut may suggest a weighing error or the inapplicability of the Kubelka-Munk equation (Eq 2), or both. If necessary, reduce a new aliquot until equivalency is obtained. Calculate the correct tinting strength according to Eq 1a or 1b.

13.2.4.2 *Instrumental Evaluation*—Prepare a thick film of the adjusted tint, remeasure the reflectance, and calculate the adjusted tinting strength by Eq 2. If 100 %, the original tinting strength value is correct. If not 100 %, compute the correct tinting strength by multiplying the original tinting strength percentage by the adjusted tinting strength in decimal units.

14. Report

14.1 Report the following information:

14.1.1 The type and identification of the test dispersion, the reference standard dispersion, and the nature of the tinting base,

14.1.2 The results of the visual evaluation of the relative color difference (masstone and undertone) of the drawdowns on bond and coated paper,

14.1.3 The relative tinting strength and the method by which it was determined (visual or instrumental). If the instrumental method was used, the manufacturer and type of instrument, the geometry (including for hemispherical geometry whether the specular component was included or excluded) and, if used, the basis for the calculation of tristimulus values,

14.1.4 The method, if any, by which the tinting strength result was confirmed, and

14.1.5 Any deviation, by agreement or otherwise, from the procedures given in these test methods.

15. Precision and Bias

15.1 Precision:

15.1.1 An interlaboratory study of these test methods was conducted in which 3 sets of process color printing inks ranging in tinting strength from 75 to 85 % were tested by operators in 6 different laboratories. The tests were conducted as blind duplicates on each of 2 days. In addition to visually evaluated tinting strength, the same reductions were measured spectrophotometrically or densitometrically, or both. The estimated standard deviations and the degrees of freedom are given in Table 2. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

15.1.1.1 *Repeatability*—Two results obtained by the same operator on different days should be considered suspect if they differ by more than the maximum allowable difference indicated in Table 2.

15.1.1.2 *Reproducibility*—Two results, each the mean of results obtained on different days by operators in different laboratories, should be considered suspect if they differ by more than the maximum allowable difference indicated in Table 2.

15.2 *Bias*—The tinting strength results obtained in the interlaboratory study of these test methods were higher than the formulated values by the amounts shown in Table 3.

16. Keywords

16.1 colorimeter; densitometer; Kubelka-Munk equation; pigment dispersions; printing inks; relative tinting strength; spectrophotometer

TABLE 2 Precision of Tinting Strength Determinations

Method of Evaluation	Standard Deviation, % absolute	Degrees of Freedom	Maximum Allowable Difference, % absolute
Repeatability			
Visual	1.3	12	3.7
Spectrophotometer	1.8	8	5.0
Densitometer	3.6	10	10.1
Reproducibility			
Visual	2.8	12	8.0
Spectrophotometer	1.7	8	4.7
Densitometer	4.8	12	13.4

TABLE 3 Accuracy of Tinting Strength Results

Method of Evaluation	Difference Between Overall Mean Test Results and Formulated Tinting Strength, ^A
	% absolute
Visual	+0.73
Spectrophotometer ^B	+3.87
Densitometer ^B	+1.90

^A If the unknowns had been stronger than the standard, the plus signs would be minus signs.

^B Values are based on the original tints, not on adjusted tints as recommended in 12.8.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Print Resistance of Lacquers¹

This standard is issued under the fixed designation D 2091; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 6211 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the resistance of dried lacquer films to imprinting.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Base²

2.2 U.S. Federal Specifications:

CCC-C-419(f) Cloth, Duck, Cotton, Unbleached, Plied³

CCC-C-440(e) Cloth, Cheesecloth, Bleached and Unbleached³

3. Summary of Test Method

3.1 A weight presses a piece of fabric against the test surface. The surface is then examined and changes in appearance of the test surface are reported.

4. Significance and Use

4.1 An unsatisfactory appearance can result from pressure deformation of a film inherently too soft or containing residual solvent. This test method is primarily used to evaluate the resistance of a lacquer finish to printing under

the conditions of packaging, shipping, and warehousing.

5. Apparatus and Materials

5.1 *Test Panels*—A plane surface consisting of wood, metal, paper supported on plate glass, plastic, or other surface with an area at least 100 % greater than the base of the weight. The planeness, smoothness, and composition should be agreed upon by the purchaser and the seller.

5.2 *Imprinting Fabric*, 8-oz Army duck, Type III, conforming to Fed. Spec. CCC-C-419(f) or cheesecloth, Type I, conforming to Federal Specification CCC-C-440(e).

NOTE—With the cheesecloth only, use a pad of nonwoven felt cloth at least 0.05 in. (1.3 mm) thick, weighing 7 oz/yd² (0.24 kg/m²) and larger than the plane end of the weight.

5.3 *Weights*, consisting of metal cylinders cut from standard stock 2 in. (51 mm) in diameter, with plane ends perpendicular to the axis, and of a length to give a pressure of 1/2 or 1 lb/in.² (3.5 or 7.0 kPa).

5.4 *Automatic Application Equipment, (Optional)*, as described in Test Methods D 823.

5.5 *Film Thickness Measuring Apparatus*, as described in Test Methods D 1005, D 1186, and D 1400.

6. Preparation of Test Specimen

6.1 *Single Coats*—Prepare a specimen for test by applying the test lacquer to a panel with a film applicator or other specified method, as described in Test Methods D 823, to give a specified film thickness to be stated in the result. In the absence of a specified dry film thickness, 1 ± 0.1 mil (25 ± 3 μ m) is recommended.

6.2 *Multiple Coats*—Use the finish system as agreed upon between the purchaser and the seller. In the absence of a specified dry film thickness, 3 ± 0.3 mils (75 ± 8 μ m) are recommended.

6.3 *Conditioning*—Allow to dry as agreed upon between the purchaser and the seller, but run the test within 48 h after coating. In the absence of a specified drying schedule, a period of 24 h at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and a relative humidity of 50 ± 5 % is recommended.

7. Procedure

7.1 Place over a uniform area of the specimen either a smooth piece of duck cloth or a piece of cheesecloth with a felt pad cushion. Place one or more weights, each on top of the other, on the duck or cheesecloth and pad and maintain the weight in compliance with one of the following test conditions:

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved March 25, 1988. Published September 1988. Originally published as D 2091 - 62 T. Last previous edition D 2091 - 87.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

7.1.1 At $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity for an 18-h period,

7.1.2 At 120°F (50°C) for a 4-h period, or

7.1.3 At 140°F (60°C) for a 4-h period.

7.2 After the specified time, remove the weight, pad, and fabric. Remove lint and dust from the panel with a clean air stream. Examine the specimen immediately under bright, diffused illumination for impression of the fabric into the lacquered surface and compare to photographic standards (Fig. 1).

8. Report

8.1 Report the following information:

8.1.1 Surface involved,

8.1.2 Drying schedule,

8.1.3 Film thickness,

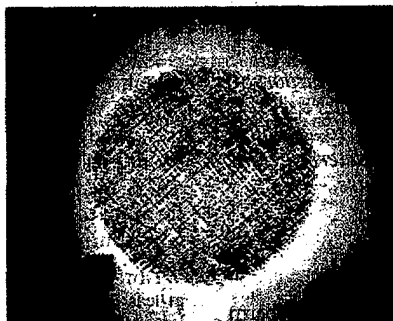
8.1.4 Weight used,

8.1.5 Test temperature used, time, and

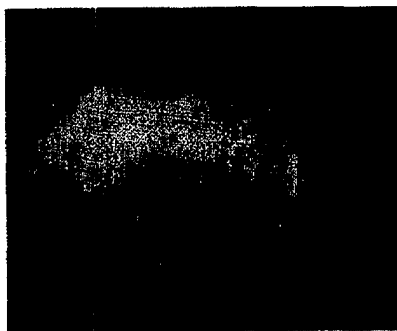
8.1.6 Conditions of test other than those primarily specified in this test method.

9. Precision

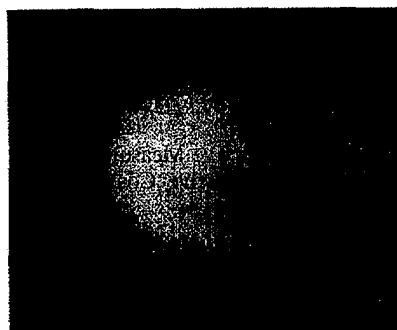
9.1 Results varying by more than 1° of print should be suspect, either with the same or different operators.



(a) Heavy



(b) Medium



(c) Slight

FIG. 1 Print Resistance

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Standard Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting¹

This standard is issued under the fixed designation D 2092; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice describes six methods of preparation for painting new zinc-coated (galvanized) surfaces produced by either the hot-dip method or by electroplating. This practice covers surfaces that have not been treated previously at the mill to provide temporary protection against staining by moisture other than by easily removed protective oils (see Appendix X1).

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 U.S. Federal Specification:

TT-P-641 Primer Coating, Zinc Dust Oxide (for Galvanized Surfaces)²

2.2 U.S. Military Specification:

DoD-P-15328 Primer (Wash) Pretreatment (Formula No. 117 for Metals)²

2.3 Canadian Standard:

CGSB, 1-GP-198 Primer, Cementitious for Galvanized Surfaces³

2.4 Steel Structures Painting Council Specification:

Paint No. 27 Basic Zinc Chromate-Vinyl Butyrol Wash Primer⁴

3. Summary of Practice

3.1 This practice describes methods of preparation that provide galvanized surfaces suitable for painting, specifically so that an applied coating system can develop the adhesion necessary for satisfactory service life.

3.2 Six methods of surface preparation (Note 1) are covered as follows:

3.2.1 Method A—Crystalline Zinc Phosphate Treatment.

3.2.2 Method B—Chromate Treatment.

3.2.3 Method C—Aqueous Chromic-Organic Treatment.

3.2.4 Method D—Acid-Bound Resinous Treatment.

3.2.5 Method E—Annealing Heat Treatments.

3.2.6 Method F—Amorphous Complex-Oxide Treatment.

NOTE 1—Materials employed in these methods of treatment are available from a number of sources as proprietary compounds or methods. Selection may be made from available sources.

3.3 Variations in surface preparation produce end conditions that differ and that do not necessarily yield identical results when paints are applied. Service conditions will dictate the type of surface preparation to be selected, although the quality produced by any individual method may vary with different zinc coatings.

3.4 Galvanized surfaces are treated by using various methods and apparatus; satisfactory application may be made at the following locations:

	Mill	Plant	Field
Method A	Y	Y	Y
Method B	Y	Y	...
Method C	Y	Y	...
Method D	Y	Y	Y
Method E	Y	...	...
Method F	Y	Y	...

3.5 This practice does not describe the cleaning necessary to provide a zinc-coated (galvanized) surface suitable for the application of the treatments. Many cleaning methods are applicable and should be agreed upon between the purchaser and the supplier.

NOTE 2—Most producers of zinc-coated (galvanized) steel sheets and coils have adopted the practice of applying an inhibitor to the zinc surface to give temporary protection against staining by moisture during shipping or storage. Some of these inhibitors will interfere with proper reaction of most of the treatments described in these methods, and an unsatisfactory surface for painting will result. It is strongly recommended that the purchaser consult the supplier of the chemical treatment to be used as to the suitability of the zinc surfaces for treatment by any of these methods (see Appendix X2).

4. Significance and Use

4.1 This practice describes procedures that can be used to prepare new zinc-coated surfaces for painting and improve the bond of paint to the zinc surface.

5. Processes

5.1 *Method A, Crystalline Zinc Phosphate Treatment*—This conversion-coating method consists of reacting the zinc surface in a zinc acid phosphate solution containing oxi-

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

Current edition approved Sept. 26, 1986. Published November 1986.

² Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

³ Available from Canadian Government Publishing Center, Supply and Services, Ottawa, Ontario K1A 0S9 Canada.

⁴ Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

dizing agents and other salts for accelerating the coating action. The zinc surface is converted to a crystalline phosphate coating of the proper texture to inhibit corrosion and increase the adherence and durability of the paint film. Such treatments are recommended for product finishes and may be carried out by immersion, spray, or brush application.

5.2 Method B, Chromate Treatment—This treatment consists of a dip or spray with a dilute solution of a mixture of chromium trioxide and other acids, with the proper accelerator, for a period from 5 to 30 s at room temperature to 130°F (55°C) to provide a thin amorphous chromate coating that increases corrosion resistance and paint adhesion.

5.3 Method C, Aqueous Chromic-Organic Treatments—Certain water-soluble resins, when properly formulated with chromium compounds, may be applied to zinc surfaces by roller coat or other suitable means, such as dip and squeegee rolls. This may be done over a wide temperature range provided the film is properly baked or cured, or both, as required by the paint system to be applied. The resultant coating provides a corrosion-resistant film that increases the adhesion of applied paint films.

5.4 Method D, Acid-Bound Resinous Treatment (Vinyl Wash Primer) (See DoD-P-15328 and SSPC-Paint No. 27)—This surface treatment is based on the application of an acid-bound resinous film of approximately 0.3 to 0.5 mil (8 to 13 μ m) thickness. The treatment is based on three primary components: a hydroxyl-containing resin, a pigment capable of reacting with the resin and an acid, and an acid capable of

insolubilizing the resin by reacting with the resin, the pigment, and the zinc surface. The film is usually applied by spray, but may be applied by brush, dip, or roller coater. Under normal conditions it will dry sufficiently for recoating within 30 min, and within 8 h it will not be softened by organic solvents commonly used in paint coatings. The film has good adhesion to the metal substrate and promotes good adhesion of most subsequent organic coatings to itself. Two types of this treatment are available: (1) two-package material to be used the day it is mixed and (2) one-package material that has package stability and does not require daily preparation.

NOTE 3—It may be difficult to control the dry film thickness within the parameters of this specification when applied by brush, roller, or dip coater.

5.5 Method E, Annealing Heat Treatments—Under the controlled conditions obtainable in a mill, hot-dip galvanized surfaces may be converted and alloyed with the base metal to change the surface character of the zinc coating and make it more receptive to paint. This surface can be further improved by treating in accordance with Methods A, B, C, or D.

5.6 Method F, Amorphous Complex-Oxide Treatment—This surface treatment method consists of reacting the zinc surface in an alkaline solution containing heavy metal ions for a period of 5 to 30 s at 115 to 160°F (45 to 70°C). The surface of the zinc is converted to a nonmetallic, amorphous, complex-oxide coating that inhibits corrosion and increases the adhesion and durability of paint finishes. The treatment can be carried out by immersion or spray application.

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the treat
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Appendixes

(Nonmandatory Information)

X1. CLEANING OF ZINC SURFACES

X1.1 When zinc surfaces treated by Methods A through F have been soiled in fabrication, they should be cleaned before finishing. Alkali and acid cleaners should not be used without consulting the supplier of the treatment. Organic solvents will remove most soils but will not remove water-soluble salts unless specially selected. Proprietary solutions designed to clean zinc surfaces are available.

X1.2 New zinc-coated steel surfaces that cannot be treated in accordance with these methods prior to painting

can be exposed to the weather for at least 6 months prior to painting to allow the surface to oxidize.⁵

NOTE X1.1—There are special primers that obviate the need for weathering—U.S. Fed. Spec. TT-P-641 and CGSB, 1-GP-198 are examples.

⁵ Literature on painting zinc-coated steel surfaces may be obtained from the American Zinc Institute, 292 Madison Ave., New York, NY 10017, or from the National Paint, Varnish, and Lacquer Assoc., 15 Rhode Island Ave., N.W., Washington, DC 20005.

X2. IDENTIFYING THE PRESENCE OF AND REMOVING CHROMATE TREATMENTS USED AS WET-STORAGE (ALSO CALLED HUMID-STORAGE) STAIN INHIBITORS

X2.1 One of the inhibitors used by producers of zinc-coated steel is a hexavalent chromium solution. This treatment prevents Method D from working properly.

X2.2 If zinc-coated steel to be painted is galvanized to order, the order should prohibit the use of hexavalent chromium humid-storage stain treatments.

X2.3 Hexavalent chromium treatment can be removed

from galvanized surfaces by one of the following three methods:

X2.3.1 Weathering the surfaces for six months.

X2.3.2 Abrading the surfaces by sanding.

X2.3.3 Brush-off abrasive blast cleaning.

X2.4 The presence of hexavalent chromium on galvanized surfaces can be determined by spot testing with

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diphenylcarbohydrazide solution. The spot test can also be used to evaluate the effectiveness of preparation to remove the treatment.

X2.5 Make the spot test solution as follows:

X2.5.1 Dissolve 0.5 g of 1,5-diphenylcarbohydrazide powder in a mixture of 20 mL acetone and 20 mL denatured ethanol. Heat the mixture in a warm water bath if necessary.

X2.5.2 Dilute 20 mL of concentrated phosphoric acid to 40 mL by slowly adding to 20 mL of distilled or deionized water.

X2.5.3 Add the dilute phosphoric acid to the acetone-alcohol solution.

X2.5.4 Store the solution away from light. Discard if it

becomes discolored. Preferably make fresh solution as needed using proportionally smaller amounts of ingredients.

X2.6 Conduct the spot test as follows:

X2.6.1 Degrease the test spot on the galvanized surface.

X2.6.2 Place several drops of the test solution on the galvanized surface.

X2.6.3 If no color develops in the solution within 10 s, hexavalent chromium is not present.

X2.6.4 If solution droplets turn a pink to purple color, then hexavalent chromium is present.

X2.6.5 Conduct the spot test on several representative spots on each individual piece of galvanized steel.

X2.6.6 Test every piece of galvanized steel that is to be treated for painting.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Method for Softening of Organic Coatings by Plastic Compositions

This method covers the determination of the relative degree of surface softening of organic coating by plastic compositions under specified exposure conditions.

Formerly under the jurisdiction of Committee D-20 on Plastics, this test method was discontinued in 1990.

1. Scope

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Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer¹

This standard is issued under the fixed designation D 2196; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace Method 4287 of Federal Test Method Standard No. 141A. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Section 24 was added editorially in August 1990.

1. Scope

1.1 These test methods cover the determination of the apparent viscosity and the shear thinning and thixotropic properties of non-Newtonian materials in the shear rate range from 0.1 to 50 s⁻¹.

1.2 This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Document

2.1 ASTM Standard:

E 1 Specification for ASTM Thermometers²

3. Summary of Test Methods

3.1 Test Method A consists of determining the apparent viscosity of coatings and related materials by measuring the torque on a spindle rotating at a constant speed in the material.

3.2 Test Methods B and C consist of determining the shear thinning and thixotropic (time-dependent) rheological properties of the materials.³ The viscosities of these materials are determined at a series of prescribed speeds of a rotational-type viscometer. The agitation of the material immediately preceding the viscosity measurements is carefully controlled.

4. Significance and Use

4.1 Test Method A is used for determining the apparent viscosity at a given rotational speed, although viscosities at two or more speeds better characterize a non-Newtonian material than does the single viscosity measurement.

4.2 With Test Methods B and C, the extent of shear

thinning is indicated by the drop in viscosity with increasing viscometer speed. The degree of thixotropy is indicated by comparison of viscosities at increasing and decreasing viscometer speeds (Test Method B), viscosity recovery (Test Method B), or viscosities before and after high shear (combination of Test Methods B and C). The high-shear treatment in Test Method C approximates shearing during paint application. The viscosity behavior measured after high shear is indicative of the characteristics of the paint soon after application.

5. Apparatus

5.1 Rotational-type viscometers having at least four speeds, such as:

5.1.1 Brookfield Viscometer,⁴ Model LVF, having four rotational speeds, or Model LVT having eight rotational speeds, with set of four spindles; or

5.1.2 Brookfield Viscometer, Model RVF, having four rotational speeds, or Model RVT having eight rotational speeds, with set of seven spindles.

5.2 Thermometer—ASTM thermometer having a range from 20 to 70°C and conforming to the requirements for Thermometer 49C as prescribed in Specification E 1.

5.3 Containers, round 1-pt (0.5-L) can, 3³/₈ in. (85 mm) in diameter, or 1-qt (1-L) can, 4 in. (100 mm) in diameter.

5.4 Shaker,⁵ or equivalent machine capable of vigorously shaking the test specimen.

6. Materials

6.1 Standard Oils,⁶ calibrated in absolute viscosity, millipascal seconds.

7. Calibration of Apparatus

7.1 Select at least two standard oils of viscosities differing by at least 5 P (0.5 Pa·s) within the viscosity range of the material being measured and in the range of the viscometer. Condition the oils as closely as possible to 25.0°C (or other

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved Aug. 29, 1986. Published October 1986. Originally published as D 2196 - 63 T. Last previous edition D 2196 - 81.

² Annual Book of ASTM Standards, Vol 14.03.

³ Pierce, P. E., "Measurement of Rheology of Thixotropic Organic Coatings and Resins with the Brookfield Viscometer," *Journal of Paint Technology*, Vol 43, No. 557, 1971, pp. 35-43.

⁴ Brookfield viscometers are available from the Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

⁵ A reciprocating shaker may be obtained from the Red Devil Tools, 2400 Vauxhall Rd., Union, NJ 07083.

⁶ Absolute viscosity standards are available in 1-pt samples from The Cannon Instrument Co., P.O. Box 16, State College, PA 16801, or Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

agreed-upon temperature) for 1 h in a 1-pt (0.5-L) can, 3/8 in. (85 mm) in diameter. Measure the viscosities of each oil as described in Test Method B (Section 13) taking readings only at increasing speeds (13.7). Make certain that the spindle is centered in the container prior to taking measurements.

NOTE 1—The Brookfield LV and RV series viscometers are equipped with a spindle guard leg. The spindle/speed multiplying factors (Table 1) are designed for use with the guard leg in place except for the following conditions: RV series when the factors are the same with or without the guard leg for spindles No. 3 through 7; or LV series when the factors are the same with or without the guard leg for spindles No. 3 and 4.

7.1.1 Calibration in a 1-pt (0.5-L) can is always possible with the LV series viscometer with the guard leg attached. Calibration of the RV series viscometer in the 1-pt can must be done with spindles No. 3 through 7 without the guard leg. If the No. 1 or No. 2 spindles are to be used, calibration is carried out in the 1-qt (1-L) can with the guard leg attached.

7.2 Combining the tolerance of the viscometer ($\pm 1\%$, equal to the spindle/speed factor) and the tolerance of the temperature control (typically $\pm 0.5^\circ\text{C}$ at 25°C) it is reasonable to assume that a viscometer is calibrated if the calculated viscosities are within $\pm 5\%$ of the stated values (see Table 2 for examples of the considerable change in viscosity with temperature exhibited by standard oils). If measurements are not made at 25°C , then the stated viscosities should be corrected to the temperature at which they are measured. If the viscosities determined in 7.1 differ from the stated values of the viscosity standard by more than 5%, calculate new factors for each spindle/speed combination as follows:

$$f = V/s \quad (1)$$

where:

f = new factor for converting scale reading to viscosity, cP (mPa·s),

V = viscosity of standard oil, mPa·s, and

s = scale reading of the viscometer.

TABLE 1 Factors for Converting Brookfield Dial Readings to Centipoises (Millipascal Seconds)

NOTE—M = 1000.

Speed, rpm	RV Series Factors Spindles						
	1	2	3	4	5	6	7
0.5	200	800	2000	4000	8000	20M	80M
1	100	400	1000	2000	4000	10M	40M
2	50	200	500	1000	2000	5M	20M
2.5	40	160	400	800	1600	4M	16M
4	25	100	250	500	1000	2.5M	10M
5	20	80	200	400	800	2M	8M
10	10	40	100	200	400	1M	4M
20	5	20	50	100	200	500	2M
50	2	8	20	40	80	200	800
100	1	4	10	20	40	100	400

Speed, rpm	LV Series Factors Spindles			
	1	2	3	4
0.3	200	1000	4000	20M
0.6	100	500	2000	10M
1.5	40	200	800	4M
3.0	20	100	400	2M
6	10	50	200	1M
12	5	25	100	500
30	2	10	40	200
60	1	5	20	100

7.3 Prepare a table of new factors similar to that furnished with the viscometer (Table 1) for the spindle/speed combinations worked out in 7.2. Spindle/speed factors vary inversely with speed.

8. Preparation of Specimen

8.1 Fill a 1-pt or 1-qt can with sample to within 1 in. (25 mm) of the top with the sample and bring it as close as possible to a temperature of 25°C or other agreed-upon temperature prior to test.

8.2 Vigorously shake the specimen on the shaker or equivalent for 10 min, remove it from the shaker, and allow it to stand undisturbed for 60 min at 25°C prior to testing (Note 2). Start the test no later than 65 min after removing the can from the shaker. Do not transfer the specimen from the container in which it was shaken.

NOTE 2—Shake time may be reduced if necessary, or as agreed upon between the purchaser and manufacturer, but, in any case, should not be less than 3 min.

TEST METHOD A—APPARENT VISCOSITY

9. Procedure

9.1 Make all measurements as close as possible to 25°C , or other agreed-upon temperature.

9.2 Place the instrument on the adjustable stand. Lower the viscometer to a level that will immerse the spindle to the proper depth. Level the instrument using the attached spirit level.

9.3 Tilt the selected spindle (Note 3), insert it into one side of the center of the surface of the material, and attach the spindle to the instrument as follows: Firmly hold the upper shaft coupling with thumb and forefinger; screw left-hand thread spindle coupling securely to the upper shaft coupling being very careful when connecting to avoid undue side pressure which might affect alignment. Avoid rotating the dial so that pointer touches the stops at either extreme of the scale.

NOTE 3—Select the spindle/speed combination that will give a minimum scale reading of 10 but preferably in the middle or upper portion of the scale. The speed and spindle to be used may differ from this by agreement between user and producer.

9.4 Lower the viscometer until the groove (immersion mark) on the shaft just touches the material. Adjust the viscometer level if necessary. Move the container slowly in a horizontal plane until the spindle is located in approximately the center of the container so that the test will be run in a region undisturbed by the lowering of the spindle.

9.5 Turn on the viscometer. Adjust the viscometer to the rpm selected (Note 3) for the material under test. Allow the viscometer to run until the pointer has stabilized (Note 4). After the pointer has stabilized, depress the clutch and switch

TABLE 2 Viscosity Variation of Cannon Viscosity Standards About the 25°C Temperature Point

Cannon Viscosity Standard	Viscosity at 25°C , cP (mPa·s)	Viscosity Change With $\pm 1^\circ\text{C}$ at 25°C , cP (mPa·s)
S-600	1 400	87.7 (6.26 %)
S-2000	4 900	332 (6.77 %)
S-8000	20 000	1462.3 (7.31 %)

off the motor so that when it stops, the pointer will be in view (Note 5).

NOTE 4—In thixotropic paints, the pointer does not always stabilize. On occasion it reaches a peak and then gradually declines as the structure is broken down. In these cases, the time of rotation or number of revolutions prior to reading the viscometer should be agreed to between user and manufacturer.

NOTE 5—Always release the clutch while the spindle is still immersed so that the pointer will float, rather than snap back to zero.

10. Calculation

10.1 Calculate the apparent viscosity at each speed, as follows:

$$V = fs$$

where:

V = viscosity of sample in centipoises, mPa·s,

f = scale factor furnished with instrument (see Table 1), and

s = scale reading of viscometer.

11. Report

11.1 Report the following information:

11.1.1 The Brookfield viscometer model and spindle,

11.1.2 The viscosity at the spindle/speed utilized,

11.1.3 The specimen temperature in degrees celsius, and

11.1.4 The shake time and rest period, if other than specified.

12. Precision and Bias

12.1 *Precision*—See Section 23 for precision, including that for measurement at a single speed.

12.2 *Bias*—No statement of bias is possible with this test method.

TEST METHOD B—VISCOSITY UNDER CHANGING SPEED CONDITIONS, DEGREE OF SHEAR THINNING AND THIXOTROPY

13. Procedure

13.1 Make all measurements with the Brookfield viscometer as close as possible to 25°C, or other agreed upon temperature.

13.2 Adjust the instrument and attach the spindle as in 9.2 through 9.4.

13.3 Set the viscometer at the slowest rotational speed (Notes 5 and 6). Start the viscometer and record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

NOTE 6—When the eight speed viscometers (RVT and LVT) are used, lower or higher speeds than that permitted by the four speed viscometers may be used upon agreement between producer and user.

13.4 Increase the viscometer speed stepwise and record the scale reading after ten revolutions (or equivalent time for each spindle/speed combination) at each speed. After an observation has been made at the top speed, decrease the speed in steps to the slowest speed, recording the scale reading after ten revolutions (or equivalent time) at each speed.

NOTE 7—It is preferable to change speed when the motor is running.

13.5 After the last reading has been taken at the slowest speed, shut off the viscometer and allow it and the specimen to stand undisturbed for an agreed-upon rest period. At the

end of the rest period, start the viscometer at the slowest speed and record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

14. Calculations and Interpretation of Results

14.1 Calculate the apparent viscosity at each speed as shown in Section 9.

14.2 If desired, determine the degree of shear thinning by the following method:

14.2.1 *Shear Thinning Index* (sometimes erroneously called the thixotropic index)—Divide the apparent viscosity at a low rotational speed by the viscosity at a speed ten times higher. Typical speed combinations are 2 and 20 rpm, 5 and 50 rpm, 6 and 60 rpm but selection is subject to agreement between producer and user. The resultant viscosity ratio is an index of the degree of shear thinning over that range of rotational speed with higher ratios indicating greater shear thinning.

14.2.2 A regular or log-log plot of viscosity versus viscometer speed in rpm may also be useful in characterizing the shear-thinning behavior of the material. Such plots may be used for making comparisons between paints or other materials.

14.3 If desired, estimate the degree of thixotropy (under conditions of *limited* shearing-out of structure) by one of the following methods:

14.3.1 Calculate the ratio of the slowest speed viscosity taken with increasing speed to that with decreasing speed. The higher the ratio, the greater the thixotropy.

14.3.2 Calculate the ratio of the slowest speed viscosity taken after the rest period to that before the rest period. The higher the ratio, the greater the thixotropy.

15. Report

15.1 Report the following information:

15.1.1 The Brookfield viscometer and spindle,

15.1.2 The viscosities at increasing and decreasing spindle speeds,

15.1.3 The rest period time and the viscosity at the end of that time,

15.1.4 The specimen temperature in degrees celsius, and

15.1.5 The shake time if other than that specified.

15.2 *Optional Reporting*:

15.2.1 *Degree of Shear Thinning*—Shear thinning index and speeds over which it was measured (14.2).

15.2.2 *Estimated Degree of Thixotropy* (under conditions of shearing-out of structure)—Ratio of the lowest speed viscosities, for both increasing and decreasing speeds; or ratio of the lowest speed viscosities before and after the rest period, and speed at which they were measured (14.3).

16. Precision and Bias

16.1 *Precision*—See Section 23 for precision, including that for measurement of the shear thinning index (ratio of viscosity at 5 r/min to that at 50 r/min). It has not been possible to devise a method for determining precision for viscosities at increasing and decreasing speeds other than as individual measurements. No attempt was made to determine the precision of the measurement of the degree of thixotropy because this parameter is dependent on the material, the time of the test, and other variables.



16.2 *Bias*—No statement of bias is possible with this test method.

TEST METHOD C—VISCOSITY AND SHEAR THINNING OF A SHEARED MATERIAL

17. Apparatus

17.1 High-speed laboratory stirrer with speeds of at least 2000 rpm and equipped with a 2-in. (50-mm) diameter circular dispersion blade.⁷

18. Preparation of Specimen

18.1 Insert the 2-in. (50-mm) blade into the center of the can (4.3) so that the blade is about 1 in. (25 mm) from the bottom. Run the mixer at 2000 rpm (Note 8) for 1 min.

NOTE 8—Materials may be sheared at other speeds using other size blades upon agreement between producer and user.

19. Procedure

19.1 Immediately insert the same spindle used in Test Method B into the sheared material in the same manner as in Section 9.

19.2 Start the viscometer and adjust to the highest speed used in Test Method B (13.5). Record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

19.3 Decrease the viscometer speed (Note 7) step-wise and record the scale readings at each speed down to the lowest speed used in Test Method B, recording the scale reading after ten revolutions at each speed (or other agreed-upon number of revolutions).

20. Calculations and Interpretation of Results

20.1 As in Test Method B, calculate the viscosities at each decreasing speed.

20.2 If desired, calculate the degree of shear thinning by the method given in Test Method B, 14.2. The measured viscosity behavior after shearing is essentially that of the paint immediately after application (disregarding changes in solids).

20.3 If desired, estimate the degree of thixotropy (under conditions of complete shearing-out of structure) by calculating the ratio of the lowest speed viscosities before and after shear. The lowest speed before-shear viscosity is taken from Test Method B, 14.1, at the lowest increasing speed. The lowest speed after-shear viscosity is taken from 20.1. The higher the ratio, the greater the thixotropy.

21. Report

21.1 Report the following information:

- 21.1.1 The Brookfield viscometer model and spindle,
- 21.1.2 The viscosities at decreasing spindle speeds,
- 21.1.3 The specimen temperature in degrees celsius, and
- 21.1.4 The speed of the high-speed mixer, size of blade, and time of mixing if different from method.

21.2 Optional Reporting:

21.2.1 *Degree of Shear Thinning*—Shear thinning index and speed over which it was measured (14.2).

21.2.2 *Estimated Thixotropy*—Ratio of lowest speed viscosities before and after shear and the speed at which they were measured.

22. Precision and Bias

22.1 *Precision*—The precision for individual viscosity measurements is the same as for Test Method A in Section 23. No attempt has been made to determine the precision of the shear thinning index or degree of thixotropy for Test Method C for the reasons given in 16.1.

22.2 *Bias*—No statement of bias is possible with this test method.

23. Summary of Precision

23.1 In an interlaboratory study of Test Methods A and B, eight operators in six laboratories measured on two days the viscosities of four architectural paints comprising a latex flat, a latex semi-gloss, a water-reducible gloss enamel, and an alkyd semi-gloss, that covered a reasonable range in viscosities and were shear thinning. Measurements at increasing speeds of 5, 10, 20, and 50 r/min (equivalent to eight operators testing 16 samples) were used to obtain the precision of Test Method A. The within-laboratory coefficient of variation for Test Method A (single speed) was found to be 2.49 % with 121 degrees of freedom and for Test Method B (Shear Thinning Index) 3.3 % with 31 degrees of freedom. The corresponding between-laboratories coefficients are 7.68 % with 105 degrees of freedom and 7.63 % with 27 degrees of freedom. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

23.1.1 *Repeatability*—Two results obtained by the same operator at different times should be considered suspect if they differ by more than 7 % relative for single speed viscosity and 9.5 % relative for shear thinning index.

23.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 21.6 and 22.1 % relative, respectively, for the same two test methods.

24. Keywords

24.1 Brookfield viscometer; non-Newtonian; rheological properties; rheology; rotational; shear thinning; thixotropic; thixotropy; viscometer; viscosity

⁷ Cowles or Shar type mixer/disperser.

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Standard Test Method for Adhesion of Organic Coatings by Scrape Adhesion¹

This standard is issued under the fixed designation D 2197; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense to replace Method 6303 of Federal Test Method Standard No. 141C and for listing in the DoD Index of Specifications and Standards.

^{e1} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This test method covers the determination of the adhesion of organic coatings such as paint, varnish, and lacquer when applied to smooth, flat (planar) panel surfaces.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²

D 1186 Test Methods for Nondestructive Measurement of Dry-Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

D 1400 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

3. Summary of Test Method

3.1 The materials under test are applied at uniform thickness to flat panels, usually sheet metal of uniform surface texture. After drying, the adhesion is determined by pushing the panels beneath a rounded stylus or loop that is loaded in increasing amounts until the coating is removed from the substrate surface.

4. Significance and Use

4.1 Coatings to perform satisfactorily must adhere to the substrates on which they are applied. This test method has been found useful in differentiating the degree of adhesion of coatings to substrates. It is most useful in providing relative

ratings for a series of coated panels exhibiting significant differences in adhesion.

5. Apparatus

5.1 *Application Equipment*, as described in Test Methods D 823.

5.2 *Film-Thickness Measuring Apparatus*, as described in Test Methods D 1005, D 1186, or D 1400.

5.3 *Balanced-Beam, Scrape-Adhesion Tester* (Fig. 1),³ consisting of a balanced beam to which is secured a platform for supporting weights, and a rod at an angle of 45° that holds the scraping loop. The rod shall be set so that the scraping loop contacts test surfaces directly below the weights. The loop shall be 1/16-in. (1.6-mm) diameter rod, bent into a "U" shape with an outside radius of 0.128 ± 0.002 in. (3.25 ± 0.05 mm) and hardened to Rockwell HRC 56 to 58, and shall be chromium plated and polished.

6. Preparation of Specimens

6.1 Apply the materials under test to panels of the composition and surface condition on which it is desired to determine adhesion. The panel material (6.1.1), surface preparation, thickness, and number of coats shall be specified or agreed upon by the seller and the purchaser. Apply uniform coatings and air dry or bake under conditions of humidity and temperature mutually agreeable to the seller and purchaser. Either mask the panel or remove material after application, so that 1/2 in. (13 mm) at one end of the panel is uncoated.

6.1.1 The surface of the panel must be hard enough that it will not be damaged by the scraping loop. If no panel material is specified, use 0.032-in. (0.8-mm) cold-rolled carbon steel prepared in accordance with Methods B or C of Methods D 609.

7. Conditioning and Number of Tests

7.1 Condition the test panels for at least 48 h at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, and test in the same environment, or immediately on removal therefrom, unless otherwise specified or agreed by the seller and the purchaser. Test at least two replicate specimens of each material.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Aug. 29, 1986. Published October 1986. Originally published as D 2197 - 63 T. Last previous edition D 2197 - 85.

² Annual Book of ASTM Standards, Vol 06.01.

³ The tester is available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910. Other apparatus of demonstrated equivalency may be used.

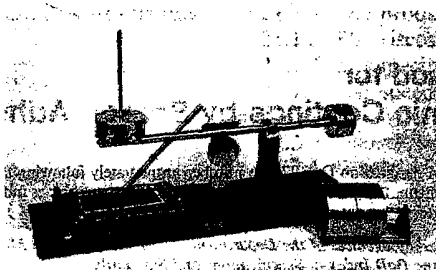


FIG. 1 Balanced-Beam Scrape-Adhesion Tester

8. Procedure

8.1 When using the instrument shown in Fig. 1, level the base plate of the apparatus and place it so that the weight holder is toward the operator. This places the beam release on the operator's right and allows freedom to move the test specimen manually under the weighted scraping element (loop). Adjust the main bearing support so that the beam is balanced in the horizontal plane when the loop is just touching the specimen surface.

8.2 Raise the beam and lock it. Wipe the loop with clean cloth or chamois. Place a test panel on the sliding platform so that it may be moved away from the operator and the uncoated portion is toward the main beam support. Place weights on the weight support using an initial amount that is estimated to be appropriate for the particular coating. Carefully lower the beam until the loop rests on the uncoated portion of the test specimen and the full load is applied, then slowly (1 to 2 s/in.) push the sliding platform away from the operator for a distance of at least 3 in. (75 mm). If the coating is removed, continue the testing, using successively smaller loads (0.5-kg increments) until the coating is not removed. If the coating is not removed by the initial scrape, continue the testing, using successively larger loads (0.5-kg increments) until the coating is removed or until the maximum load of 10 kg has been applied. Use a new area of the test surface each time a scrape is made.

8.3 When the critical load has been approximately located, repeat the test five times at each of three loadings: above, below, and at the load determined in the first trial. Apply the different loads in random fashion so that all scrapes at one load are not made in succession or on one panel.

8.3.1 Periodically examine the loop to ensure that the

original smooth surface is intact. If the contacting surface is worn, reverse the loop. When both sides are worn, replace with a new loop.

8.4 For each applied load, tabulate the number of times the coating was removed or adhered. The load where the scrape results change from mainly adhering to mainly removed, ignoring the first ½ in. (13 mm) of the scratch if the coating was removed, is the adhesion failure end point.

9. Report

9.1 Report the following information:

- 9.1.1 Load in kilograms at the adhesion failure end point.
- 9.1.2 Panel material and surface preparation.
- 9.1.3 Dry-film thickness.
- 9.1.4 Any deviation from the specified procedure.

10. Precision

10.1 *Correlation*—This method was developed when correlation with other methods of assessing adhesion was considered to be of equal importance to the agreement between results obtained in the same or different laboratories. It was established that when materials differing widely in hardness and adhesion were evaluated by a number of experienced personnel in several laboratories, the adhesion results obtained using this method correlated well with those obtained with several other methods.

10.2 *Precision*—If sufficient cooperators can be obtained, an interlaboratory study will be conducted to establish precision.

11. Keywords

11.1 adhesion, scrape; balanced-beam scrape adhesion tester

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Standard Test Method for Stain Removal From Multicolor Lacquers¹

This standard is issued under the fixed designation D 2198; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout in April 1989.

1. Scope

1.1 This test method covers a procedure for the removal of pencil, crayon, and grease stains from multicolor lacquer films that have been applied to primed steel panels.

NOTE—Due to the nature of the test, comparable results can be obtained only when exactly the same materials and apparatus are used. In this test method it has not been found possible to describe the materials and apparatus adequately in more than general terms.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- A 109 Specification for Steel, Strip, Carbon, Cold-Rolled²
- A 366/A 366M Specification for Steel, Sheet, Carbon, Cold-Rolled, Commercial Quality²
- D 209 Specification for Lampblack Pigment³
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁴

2.2 U. S. Federal Specifications:⁵

- Fed. Spec. EE-0-451C Specification for Oleo (Margarine)
- Fed. Spec. TTV-121C Specification for Varnish, Spar, Water Resisting
- Fed. Spec. VV-0-551 Specification for Oil, Lubricating, Marine and Engine, Mineral
- Fed. Spec. VV-P-236 Specification for Petrolatum

2.3 U. S. Military Specification:⁵

- MIL-P-11414A Specification for Primer Coating, Lacquer, Rust Inhibiting

3. Summary of Test Method

3.1 Three 6½ by 17-in. (165 by 430 mm) primed steel panels are sprayed with multicolor lacquer and allowed to dry for 24 h. Varnish is then applied to the ends of the panels

and allowed to dry 72 h. The three stains are applied by a mechanical apparatus and removed by washing.

4. Significance and Use

4.1 The procedure described in this test method is intended as an aid in evaluating the ease of removal for surface stains encountered by surfaces coated with multicolor lacquers.

5. Apparatus and Materials

5.1 *Steel Panels*, 6½ by 17 in. (165 by 430 mm), 20-gage cold-rolled steel conforming to Specification A 109 or Specification A 366.

5.2 *Washability Machine*.⁶

5.3 *Lacquer Primer*, conforming to MIL-P-11414A.

5.4 *Drafting Pencil*.

5.5 *China Marking Crayon*.

5.6 *Wetting Agent*.⁷

5.7 *Amorphous Silica*.

5.8 *Diaper Cloth*.

5.9 *Varnish*, clear and waterproof, conforming to Fed. Spec. TT-V-121C or equal cut in mineral spirits.

5.10 *Carriage*, for mounting pencil and crayon.

5.11 *Arm*, extendible, for mounting mohair roller.

5.12 *Mohair Roller*, 1½ in. (40 mm), for application of grease stain.

5.13 *Glass Plate*, 4 by 7 in. (100 by 175 mm), for holding grease stain.

5.14 *Grease*—The grease shall consist of the following ingredients:

5.14.1 *Lanolin*, anhydrous, 50 g,

5.14.2 *Petrolatum*, 50 g, conforming to Fed. Spec. VV-P-236,

5.14.3 *Margarine*, 30 g, conforming to Fed. Spec. EE-0-451C,

5.14.4 *Mineral Oil*, 10 g, conforming to Fed. Spec. VV-0-551, and

5.14.5 *Lampblack*, 5 g, conforming to Specification D 209.

6. Preparation of Panels

6.1 Prepare three 6½ by 17-in (165 by 430-mm) steel panels in accordance with Methods D 609. Spray a 0.5-mil

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Aug. 31, 1984. Published January 1985. Originally published as D 2198 - 63 T. Last previous edition D 2198 - 68 (1979)^{e1}.

² *Annual Book of ASTM Standards*, Vol 01.03.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁶ Gardner Model AG8100, available from Byk-Gardner, Inc., Gardner Laboratory, 2435 Linden Ln., Silver Spring, MD 20910, has been found suitable for this purpose.

⁷ Triton X-100, available from Rohm and Haas Marketing Services, Independence Mall West, Philadelphia, PA 19105, has been found suitable for this purpose.

(13- μ m) film of the lacquer primer and air dry for 24 h at normal room temperature. With the primer reduced 100 % with the recommended thinner, one wet coat will give approximately 0.5 mil.

6.2 Spray two coats of multicolor lacquer over the primer with the atomization pressure adjusted to get complete covering and allow 1 h between coats for flash off.

6.3 To protect the end of the panels in handling and scrubbing, brush one coat of varnish on the 5 in. (125 mm) of the end of each panel after 24 h of drying of the multicolor. Leave 7 in. (175 mm) of exposed test lacquer in the center of the panels and allow the varnish to dry 72 h before staining the panels. Test within 1 to 2 h after staining.

7. Procedure

7.1 Application of Stains:

7.1.1 *Pencil and China Marking Crayon*—Make both the pencil lead and the crayon flat by rubbing over fine sandpaper before application of pencil and crayon stains. Mount the pencil vertically in the carriage so that it rests with its own weight on the surface to be stained. Add an additional weight so that the total is 40 g. Move the pencil over the panel 5 strokes perpendicular to the abrasion boat path. Repeat this procedure with the crayon. Stain three panels.

7.1.2 *Grease Stain*—Apply 1 g of grease to a 4 by 7-in (100 by 175-mm) glass plate and smooth with a rubber roller. Pass a 1½-in. (40-mm) mohair roller mounted on the extendible arm over the greased plate making three stripes and removing the grease down to the glass on each stripe so that the entire surface of the roller is uniformly coated. Then pass the roller over the panels coated with the lacquer under test. Make one stripe on the glass panel between the staining of each subsequent panel under test to maintain a uniform

amount of grease on the roller.

7.2 Removal of Stains:

7.2.1 Cut the diaper cloth into a strip 5⅞ by 6¾ in. (150 by 170 mm) and fold to three thicknesses to give 2 by 6¾ in. (50 by 170 mm), keeping the embossed side out and the raw edges in. Soak the folded cloth in water, squeeze out the excess water, and place on the 2-lb (900-g) abrasion boat (load boat with weight if necessary). Place 2 g of No. 22 silica wet with 2 mL of a 5 % wetting agent solution and smear this paste uniformly on the scrubbing area of the cloth with a spatula.

7.2.2 Start the scrubbing test using the Gardner Washability Machine and record the number of cycles required to remove completely each of the three stains. If any of the stain is not removed after 100 cycles, add 1 mL of 5 % wetting agent solution to rewet the cloth and continue the test. Repeat at 100-cycle intervals stopping after 500 cycles. Rinse the panel in running water and allow to dry.

8. Report

8.1 Record the results as the number of cycles (double strokes) needed to remove each stain completely. Describe any stain that is not removed after 500 cycles as the percent of stain remaining on each of three panels and record the mean of the three panels.

9. Precision

9.1 Because of the variations that can arise from the application of the stains, meaningful estimates of precision cannot be given. In round robin tests fairly good agreement was obtained by the cooperators when the method of application of stains was the same.

10. Keywords

10.1 stain removal; multicolor lacquers; lacquer

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1. Scope

1.1 This standard covers the method for determining the relative resistance of a fabric to contact.

NOTE—reliable results are obtained.

1.2 This standard covers the method for determining the relative resistance of a fabric to contact.

2. Summary

2.1 A method is described for determining the relative resistance of a fabric to contact for marking.

3. Significance and Use

3.1 This standard is used to determine the relative resistance of a fabric to contact for marking.

4. Apparatus

- 4.1 Diaper cloth
- 4.2 Plate
- 4.3 Mohair roller
- 4.4 Silica
- 4.5 Wetting agent

This is a Related Content Document. It is not a standard and is not published by ASTM.



Standard Method for Measurement of Plasticizer Migration From Vinyl Fabrics to Lacquers¹

This standard is issued under the fixed designation D 2199; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers an accelerated test for the measurement of a tendency for plasticizers in finished vinyl fabric to be transferred to coatings with which they come in contact.

NOTE—Age of fabric sample may affect results of test. To ensure most reliable results, test with fabric sample closest in age to what will be coated.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Method

2.1 A small sample of vinyl fabric is placed on a conditioned coating film under a pressure of $\frac{1}{2}$ psi (3.45 kPa). The assembly is placed in an oven for 3 days at elevated temperature. At the end of that time, the coating is examined for marring or softening. For coatings that may block or print at 50°C or lower, a lower test temperature and a longer test time may have to be used.

3. Significance and Use

3.1 Plasticizers in finished vinyl fabric can be transferred to coatings with which they come in contact. When this takes place, objectionable marring and softening occur. This method covers an accelerated test for measurement of this tendency.

4. Apparatus

4.1 *Drawdown Blade*, 5-mils (125- μ m) clearance to provide a wet film of approximately 2.5 mils (63 μ m) in thickness.

4.2 *Plate Glass Panels*.

4.3 *Window Glass*, 2 by 2-in. (51 by 51-mm) square, double strength.

4.4 *Sponge Rubber*, 2 by 2-in. (51 by 51-mm) square by $\frac{1}{4}$ in. (6.3 mm) thick.

4.5 *Weights*, flat-bottom, sufficient to place a total weight of 2 lb (910 g) on each test sample.

4.6 *Forced-Convection Oven*, thermostatically controlled to $\pm 2^\circ\text{C}$.

4.7 *Photograph Roller*.²

4.8 *Aluminum Foil*.

5. Procedure

5.1 Apply the coating to the glass panel with the drawdown blade to provide a uniform film with an area of at least a 2-in. (51-mm) square and a dry thickness of at least 1 mil (25 μ m) or as agreed upon. Dry the coating in accordance with the recommendations of the manufacturer or for a minimum of 24 h at room temperature and 2 h at 50°C.

5.2 Preheat the glass plate bearing the coating, the 2 by 2-in. glass square, and the weights for 30 min at 50°C. Place the square of vinyl fabric on the coating with care and ensure intimate contact by rolling with the photographic roller. Cover the fabric with foil, sponge rubber, glass, and weight in that order. The total weight shall be 2 lb (910 g).

5.3 Place the assembly in the forced convection oven at a temperature of 50°C for 72 h. After removal from the oven and cooling, remove the weight, sponge rubber, and aluminum foil, and carefully remove the vinyl fabric. Note and report any resistance to removal. Wipe the surface of the fabric with a soft rag dampened with heptane and examine for removal of exuded plasticizer.

6. Rating

6.1 Rate the degree of migration by viewing the surface of the coating at a low angle against the light within 2 h after termination of heating and within 15 min of removal of the fabric.

7. Report

7.1 Report marring or softening, or both, on the scale of no change, faint imprint, severe imprint, or marring.

7.2 If plasticizer is evident when the surface is wiped, this shall be reported.

8. Precision

8.1 This method does not result in a numerical value for precision but repeated tests have demonstrated that several laboratories will assign the same rating to various coating fabric combinations. Also, replicate tests by one laboratory are in good agreement.

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 Factory-Applied Coatings on Preformed Products.

Current edition approved Oct. 29, 1982. Published January 1983. Originally published as D 2199 - 63 T. Last previous edition D 2199 - 78.

² A squeegee-type roller is satisfactory.

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1. Scope

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Standard Pictorial Surface Preparation Standards for Painting Steel Surfaces¹

This standard is issued under the fixed designation D 2200; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 The visual surface preparation standards consist of a series of color prints available as separate publications. Two different sets of photographs are described in this standard, designated as Method A (ISO/Swedish Standard²) and Method B (SSPC Standard³). The two methods differ in the depiction of the initial surface, in the definition and depiction of the cleaning conditions, and in the number of cleaning methods included. Because of these differences, the specifier should state whether Method A or Method B should be used.

The colored visual surface preparation standards represent different conditions of hot rolled steel before and after surface preparation. Prior to cleaning, there are four rust grades, A to D, that cover the range from intact mill scale to 100 % rusted and pitted steel. The standards then depict the appearance of the four grades after cleaning by one or more methods (for example, blast cleaning) to various degrees of thoroughness.

2. Referenced Documents

- 2.1 *Adjuncts:*
Pictorial Surface Preparation Standards²
Surface Cleanliness Definitions³

3. Terminology

3.1 Definitions:

3.1.1 The cleanliness definitions for the Method A visual surface preparation standard appear in the text of the pictorial surface preparation standards publication.² The definitions for Method B are found in a separate publication.³

¹ This standard is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

The pictorial standards described were prepared by the Swedish Corrosion Inst. and have been jointly approved by ASTM, the Steel Structures Painting Council (Vis 1), and the Swedish Standardizing Commission.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 2200 - 66. Last previous edition D 2200 - 85 (1989).

² The pictorial surface preparation standards are available from ASTM Headquarters (request Adjunct No. 12-422000-00); the Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213; and Sveriges Standardiseringskommission, Box 3295, Stockholm 3, Sweden.

³ The Visual Standards Method B and surface cleanliness definitions are available from the Steel Structures Painting Council, 4400 Fifth Avenue, Pittsburgh, PA 15213.

4. Significance and Use

4.1 The appearance of the various degrees of blast cleaning are influenced by the initial rust grades of the steel being cleaned. The standards aid visually in judging and evaluating the degree of rusting before cleaning and the degree of cleaning of steel surfaces prior to painting.

4.2 Two methods for visual standards have evolved because of differences in the practice of using visual standards throughout the world. In Europe, the visual standards (Method A) are used as the primary means of determining the degree of cleaning. In the US, the SSPC written definitions take precedence with the visual standards used as a supplement. The visual standards of Method B comply with the SSPC definitions.

5. Procedure and Interpretation

Method A—ISO/Swedish Standard

5.1 Determine the method of cleaning to be used (for example, hand/power tool cleaning, abrasive blast cleaning, or flame cleaning).

5.2 Determine the initial condition of the steel in accordance with four initial grades (A, B, C, or D).

5.3 Following the cleaning operation, compare the surface prepared with the photographs showing the degree of thoroughness for that particular initial condition. Select the degree that most closely corresponds to the prepared surface.

5.4 Repeat the procedure for representative areas of structure and record all three items (initial condition, method of cleaning, and degree of thoroughness achieved).

Method B, SSPC Visual Standard

5.5 Determine the degree of blast cleaning to be employed.

5.6 Determine the initial condition of steel in accordance with photographs A, B, C, and D.

5.7 Following the cleaning operation, compare the prepared surface with the photographs showing the degree of thoroughness for that particular initial condition. Select the degree that most closely corresponds to the prepared surface.

5.8 Repeat for all representative areas of structure and record for each area the initial condition and degree of thoroughness achieved.

5.9 When abrasives other than silica sand are used for

blast cleaning, consult the photographs in the appendix of the SSPC Standard for possible variations in appearance created by the abrasive type.

NOTE 1—Different steel surfaces show differences in shade, color, tone, pitting, flaking, mill scale, etc. To some extent, these differences

between the actual steel surface and the visual standard can be reconciled between the painting contractor and the inspector.

6. Keywords

6.1 blast cleaning; flame cleaning; photographic standards for surface preparation; power tool cleaning; surface preparation; surface preparation standards

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Standard Test Method for Preparation of Hot-Dipped Nonpassivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products¹

This standard is issued under the fixed designation D 2201; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon, (e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Footnote 1 and Note 4 were corrected editorially in October, 1987.

1. Scope

1.1 This test method describes the preparation of a type of hot-dipped, nonpassivated, galvanized steel panel to be used for testing paint, varnish, lacquer, conversion coatings, and related products.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

A 525 Specification for General Requirements for Steel Sheet, Zinc-Coated (Galvanized) by the Hot-Dip Process²

D 2092 Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting³

2.2 U.S. Federal Test Specifications:

O-T-634a Type II Trichloroethylene⁴

O-P-191a Perchloroethylene⁴

3. Test Panels

3.1 The test panels shall be prepared from galvanized sheet that has not been subject to any kind of treatment for the purpose of passivating (Note 1) and shall be completely free from any visible signs of storage stain or white rust. The gage shall be No. 20 (0.0396 in.) (1 mm) unless otherwise agreed upon between the manufacturer and the purchaser, but in no case lighter than No. 24 gage (0.0276 in.) (0.7 mm). The galvanizing shall be of the 1.25-oz class conforming to Specification A 525, known as "commercial (full spangle)."

NOTE 1—This test panel is designed for referee testing and is not typical of galvanized material normally stocked in commercial ware-

houses. Specified panels may be obtained from standard panel suppliers.

3.2 The zinc coating shall have been applied by a continuous galvanizing method, using an aluminum-bearing zinc. Galvanized steel is customarily shipped from the mills uncoiled; however, galvanized steel that has been oiled with a nonreactive rust preventative oil shall be acceptable under this specification.

3.3 After cutting, the panels shall be cleaned in organic solvents or vapor degreased and wrapped in clean paper having a neutral pH and sealed in vaporproof bags or envelopes. The use of alkali cleaners shall not be permitted. The panel size shall be not less than 2¾ by 5⅞ in. (70 by 150 mm) and all corners and edges shall be smooth and uniformly rounded.

4. Solvents and Reagents

4.1 *Solvent Mixture*—Prepare a solvent mixture consisting of either equal volumes of Varnish Makers' and Painters' (VM&P) naphtha and xylene or of 3 volumes of VM&P naphtha and 1 volume of ethylene glycol monoethyl ether (2-ethoxyethanol).

4.2 *Stabilized Trichloroethylene or Perchloroethylene.*

NOTE 2—Perchloroethylene and trichloroethylene shall conform to and be maintained in accordance with the Federal Specification for Trichloroethylene (O-T-634a Type II) or Perchloroethylene (O-P-191a).

5. Procedure A—Galvanized Steel Test Panels Without Chemical Treatment

5.1 Wipe the corrosion-free panels vigorously on both sides with a cloth wet with the solvent to remove the gross film of oil. Clean the panels by either solvent spray or vapor degreasing, as described in 4.2 and 4.3.

5.2 *Solvent Spray Cleaning*—Spray the solvent mixture (3.1) downward at an acute angle to the panel surface using a spray gun operated with the fluid tip wide open and the atomizing tip almost completely closed. Preferred practice is to support the panels on a rack at an angle of approximately 25° from the vertical and to hold the spray gun so that the jet is directed vertically downward. The bottoms of the panels should rest upon projections, such as nails, which permit the solvent to drain from the bottom edge. Direct the solvent over the surface of the panel in an oscillating rinsing manner, moving the spray progressively from the top to the bottom of the panel until all soluble and loosely adhering soil has been washed off. The minimum treatment shall consist of five

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Aug. 31, 1965. Originally published as D 2201 - 63 T. Last previous edition D 2201 - 65 (1981)^{e1}

² Annual Book of ASTM Standards, Vol 01.06.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

oven and uniformly heat at 350°F (177°C) for 10 min. After heating, remove the panels from the oven, and allow them to cool to room temperature and coat them immediately with the paint system (Note 3). Do not touch the panels with the fingers during the entire process of preparation.

6.1 Clean standard, corrosion-free panels, without sanding or abrasion, in accordance with Procedure A. Coat the panels with a phosphate coating or other conversion coating representative of production performance (Note 4). The conditions of treatment shall be agreed upon between the manufacturer and the purchaser. Paint the treated panels immediately or store in a vacuum desiccator until needed. If panels are stored in a vacuum desiccator, upon removal from the desiccator, place the panels in an oven and uniformly heat at 350°F (177°C) for 10 min. After heating, remove the panels from the oven, allow them to cool to room temperature, and coat them immediately with the paint system (Note 3). Do not touch the panels with the fingers during the entire process of preparation.

5.3 Vapor Degreasing—Suspend the panels in a vapor phase-type degreaser containing stabilized trichloroethylene or perchloroethylene and allow them to remain above the boiling solvent until they attain the temperature of the vapor, at which time condensation on the panels no longer occurs. Allow the panels to cool to room temperature and rub briskly with a clean, lint-free cloth. Place the panels in an

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Standard Guide for Selection of Tests for Traffic Paints¹

This standard is issued under the fixed designation D 2205; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Sections 12 and 13 were editorially changed in March 1990.

1. Scope

1.1 This guide covers the selection and use of procedures for testing traffic paints in the laboratory and in the field.

1.2 This guide covers the testing of a ready-mixed paint product of sprayable consistency that shall be suitable for use as a reflecting traffic guide on paved roadways.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- C 219 Terminology Relating to Hydraulic Cement²
- D 8 Terminology Relating to Materials for Roads and Pavements³
- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products⁴
- D 154 Guide for Testing Varnishes⁵
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints⁵
- D 215 Methods of Chemical Analysis of White Linseed Oil Paints⁵
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁵
- D 711 Test Method for No-Pick-Up Time of Traffic Paint⁵
- D 713 Practice for Conducting Road Service Tests on Fluid Traffic Marking Materials⁵
- D 868 Test Method for Evaluating Degree of Bleeding of Traffic Paint⁵
- D 869 Test Method for Evaluating Degree of Settling of Paint⁵
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion⁵
- D 913 Test Methods for Evaluating Degree of Resistance to Wear of Traffic Paint⁵

D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁵

D 969 Test Method for Laboratory Determination of Degree of Bleeding of Traffic Paint⁵

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁵

D 1309 Test Method for Settling Properties of Traffic Paints During Storage⁵

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁵

D 1644 Test Methods for Nonvolatile Content of Varnishes⁵

D 1647 Test Methods for Resistance of Dried Films of Varnishes to Water and Alkali⁵

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁷

D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁸

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁵

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁵

D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints⁵

D 4061 Test Method for Retroreflectance of Horizontal Coatings⁷

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁸

E 308 Method for Computing the Colors of Objects by Using the CIE System⁸

2.2. *U.S. Federal Test Methods Standard 141B.*⁹

4121 Dry Opacity

3. Terminology

3.1 *Definitions*—For definitions used in this guide, refer to Terminology C 219 and Definitions D 8 and D 16.

4. Summary of Guide

4.1 This guide consists of the following tests that, although not exhaustive, cover the areas normally of concern in traffic paint testing:

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved Dec. 13, 1985. Published March 1986. Originally published as D 2205 - 63 T. Last previous edition D 2205 - 75.

² *Annual Book of ASTM Standards*, Vol 04.01.

³ *Annual Book of ASTM Standards*, Vols 04.02 and 04.03.

⁴ *Annual Book of ASTM Standards*, Vols 06.01, 06.02, and 06.03.

⁵ *Annual Book of ASTM Standards*, Vol 06.01.

⁶ *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

⁷ *Annual Book of ASTM Standards*, Vol 14.02.

⁸ Discontinued; see 1988 *Annual Book of ASTM Standards*, Vol 06.01.

⁹ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

Liquid Paint Properties
Application and Appearance Properties
Properties of the Dried Film
Analysis of Paint
Field Evaluations

Sections
6 through 11
12 through 17
18 through 20
21 through 24
25 through 29

5. Conditions Affecting Traffic Paint

5.1 Practical requirements for traffic paint may vary with:

5.1.1 Substrate type, such as portland cement and asphaltic concretes, and the various coarse aggregates used therein.

5.1.2 Climatic conditions, both generally and specifically, at the time of paint application.

5.1.3 Service density, such as heavy traffic areas in cities versus lightly traveled rural highways and parking lots.

5.1.4 Traffic type, whether light passenger cars or heavy trucks and airplanes.

5.1.5 Presence of foreign matter on the road surface, such as oil, old paint, skid marks, sand, salt, concrete curing compound, etc.

5.2 New portland cement concrete surfaces have a greater degree of moisture and alkalinity than older surfaces and thereby adversely affect paint adhesion. Paint adhesion is also affected by the ratio of cement to fine aggregate, coarse aggregate, and mixing water, as well as by the surface character of the aggregate that can range from impervious smooth quartz to irregular, porous slag.

LIQUID PAINT PROPERTIES

6. Skinning

6.1 Paints containing a binder that dries by oxidation are subject to skin formation in a partially filled can or by diffusion of air into a filled can. Since skins are insoluble in the paint they must be removed before use. The referenced test employs a partially filled container to indicate the tendency of a paint to skin. A typical minimum time for skinning is 18 to 24 h.

6.2 Examine the original sample for skins both on the surface and in the mass. Using a well-mixed, skin-free portion of the sample, perform a skinning test in accordance with Guide D 154, except use a 1-pt (0.5-L) friction-top can instead of an 8-oz (0.25-L) jar.

7. Coarse Particles

7.1 Paints must be free of oversize particles and foreign matter to avoid clogging application equipment, a typical maximum being 1 % by weight of total paint. The referenced test with a 325-mesh (45- μ m) screen gives the percent of this material in the paint.

7.2 Determine coarse particles in accordance with Test Methods D 185.

NOTE 1—This test is not used for traffic paint containing pre-mixed glass beads.

8. Fineness of Dispersion

8.1 The more finely a pigment is dispersed, the more efficiently it is being used. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the material down a calibrated, tapered groove in a hardened steel block with the groove varying in

depth from 4 to 0 mils (100 to 0 μ m). The point at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the liquid is taken as the fineness reading. Lower readings in mils or micrometres or higher reading in Hegman units indicate better fineness of dispersion.

8.2 Fineness of grind is not generally specified for traffic paint but some application equipment may require a limit of 1 to 2 Hegman units (3 to 3.5 mils, 75 to 90 μ m). If additional assurance is needed that the paint will not clog application equipment, determine the fineness in accordance with Test Method D 1210 after reducing the traffic paint with mineral spirits, or compatible aromatic solvent with a similar evaporation rate, to keep the film wet long enough to determine the end point more easily. When a premix traffic paint is being tested, conduct the test on the paint before addition of the beads.

9. Density or Weight per Gallon

9.1 Density as measured by weight per unit volume is not a performance characteristic but is used to check product uniformity from batch to batch. A calibrated weight per gallon cup is used.

9.2 For an unbeaded paint, determine the density in accordance with Test Method D 1475.

9.3 For beaded paints, use a special weight-per-gallon cup¹⁰ having a modified cap so that the beads do not interfere with a snug fit of the cap to the cup. Proceed in accordance with Test Method D 1475.

10. Consistency

10.1 Paints of a given type should fall within a stated consistency range as agreed upon between the purchaser and the seller. Consistency is used mainly to ensure product uniformity. Improper consistency, however, can adversely affect application properties, and in turn, paint performance.

10.2 Determine consistency using the Stormer viscometer in accordance with Test Method D 562. If the requirement is in Krebs units, Table 1 of Test Method D 562 permits changing seconds to KU.

11. Package Stability

11.1 Since paints are not normally used immediately after manufacture, they must remain stable in the can for some time, which for traffic paints does not generally exceed 6 months. Although package stability can usually be determined by alternatively heating and cooling a specimen, occasionally the results do not coincide with storage at normal temperature. The referenced methods determine the degree of pigment settling after 2 weeks cycling or after 6 months storage at room temperature. These are usually sufficient as it is difficult to rate numerically the ease of redispersing an aged traffic paint.

11.2 Determine the degree of pigment settling in the accelerated test in accordance with Test Method D 1309. Determine the degree of pigment settling and ease of

¹⁰ A satisfactory modified cup is available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

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remixing a shelf-aged specimen in accordance with Test Method D 869.

APPLICATION AND APPEARANCE PROPERTIES

12. Drying Time

12.1 The drying time of a traffic paint is particularly important because it determines how quickly a lane can be opened to free flow of traffic without the paint being transferred to adjacent pavement.

12.2 Though no-pick-up time as determined by Test Method D 711 has no direct correlation with field application, it is employed as a quality control test.

13. Bleeding

13.1 Bleeding refers to the passage of colored matter such as bitumen from an asphalt pavement through the traffic paint film. It is a function of the age of the asphalt, its compatibility with the paint, and the speed of drying of the paint. Typical traffic paints give results of 6 to 10 on an arbitrary scale of photographic standards where 10 is no bleeding and 2 is considerable bleeding. Determine bleeding in accordance with Test Methods D 868 and D 969.

14. Hiding Power

14.1 Hiding power or opacity is a measure of the ability of a paint to hide the substrate. It varies, naturally, with the thickness of the applied film that may be influenced by the flow and application properties of the paint.

14.2 Determine the dry hiding power of traffic paints in accordance with Procedure A, Method 4121 of U.S. Federal Test Method Standard 141B. (This method is being rewritten in ASTM form.)

15. Color and Color Difference

15.1 The color of a paint may be determined precisely by means of a spectrophotometer. However, the exact color is not usually as important as how closely a paint matches a standard. Color difference between a product and a standard can be determined visually or with less elaborate instruments than for color measurement. Visual comparison of color is fast and often acceptable although numerical values are not obtained. Color difference instruments, while not more sensitive than the eye, provide numerical values that can be subsequently compared to later measurements.

15.2 If required, determine the color in terms of tristimulus values or chromaticity coordinates in accordance with Method E 308.

15.3 Determine color difference by visual comparison against standard color chips¹¹ in accordance with Practice D 1729. This practice covers the spectral photometric, and geometric characteristics of light source, illuminating and viewing conditions, size of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials.

15.4 Determine color difference instrumentally in accordance with Test Method D 2244. The method covers the

instrumental measurement of small color differences observable in daylight illumination between nonfluorescent, non-metameric, opaque surfaces.

16. Reflectance

16.1 Reflectance is a measure of the light reflected from the surface of a paint. It determines which of two specimens appears lighter when viewed in average daylight at an angle that eliminates gloss effects.

16.2 Determine the green filter reflectance in accordance with Test Method E 97.

17. Night Visibility or Retroreflectance of Beaded Paints at Low Angles

17.1 This property is important to traffic paint but visibility at night is not related to daylight reflectance. The retroreflectance evaluation of test panels coated with traffic paint should be in accordance with Test Method D 4061. Such panels can serve as controls in monitoring the application of traffic paint and can be helpful in correlating visual ratings with absolute values.

PROPERTIES OF THE DRIED FILM

18. Resistance to Wear

18.1 Resistance to wear is a measure of the ability of the dried film to withstand wear from traffic and from objects rolled or pulled across the surface. In the referenced method abrasive is poured onto a dry film on a glass panel until the paint is removed. A typical value for traffic paint is 65 L of sand for removal of a 3-mil (75- μ m) dry film. Determine resistance to wear in accordance with Test Method D 913.

18.2 Using unbeaded traffic paint, determine the abrasion resistance to falling sand or silicon carbide in accordance with Test Methods D 968.

19. Elongation

19.1 Elongation is a measure of the flexibility of a paint film. Traffic paints may have difficulty in meeting the referenced test if they are over-pigmented to obtain high reflectance.

19.2 Using unbeaded traffic paint, determine the flexibility in accordance with Test Method D 1737 but using 30-gage (0.32-mm) tin plate in place of the specified steel panel.

19.2.1 As the thickness and curing conditions are not specified in Test Method D 1737, one of the following alternatives should be used for testing traffic paint:

19.2.1.1 Apply a 15-mil (380- μ m) wet film, allow to air dry 18 h, bake 2 h at 50°C, and let cool before conducting the test with a 1/2-in. (12.7-mm) mandrel.

19.2.1.2 Apply a 10-mil (250- μ m) wet film, allow to air dry 24 h, bake 1 h at 65°C, cool, and use a 1/4-in. (6.4-mm) mandrel.

19.2.1.3 Apply a 6-mil (150- μ m) wet film, bake 6 h at 100°C, cool, and use a 1/4-in. (6.4-mm) mandrel.

20. Water Resistance

20.1 This property is important to traffic paints because they are frequently exposed to rain or condensation on bridges. The immersion test time is quite short in relation to

¹¹ Standard yellow color chips may be obtained from the Traffic Control Systems Div., HTO-20, Office of Traffic Operations, Federal Highway Administration, Washington, DC 20590.

actual exposure so that the test detects only paints with poor water resistance.

20.2 Using unbeaded paint, determine water resistance in accordance with either Practice D 870 or Test Methods D 1647.

20.3 As Practice D 870 specifies steel panels but not the immersion time, while Test Methods D 1647 requires tin plate and an immersion of 18 h, the following should be used for testing traffic paint: apply a 5-mil (130- μ m) wet film to a clean glass panel, allow to air dry for 72 h, immerse in reagent water for 24 h, and allow a recovery period of 2 h before examining.

ANALYSIS OF PAINT

21. Chemical Analysis

21.1 If a specification requires certain raw materials or certain components in a given amount, then chemical analysis is necessary to determine whether the specified materials are present in the required amounts. Analysis does not necessarily establish paint quality that can also be greatly affected by manufacturing techniques. Select test procedures from Methods D 215 and other ASTM methods that are pertinent to the components of traffic paints.

NOTE 2—No single schematic analysis is comprehensive enough to cover the wide variety of traffic paint compositions.

22. Nonvolatile Content (Paint)

22.1 The percent nonvolatile matter indicates the amount of material remaining after the solvent evaporates and is a measure of the film solids. Determine the nonvolatile content in accordance with Test Methods D 1644 using a larger specimen size in the case of beaded paint. It is suggested that the methods be selected as follows:

22.1.1 *Test Method A*—3 h at 105°C for paints where the nonsolvent components decompose at 149°C, and

22.1.2 *Test Method B*—10 min at 149°C for most paints where the nonsolvent components are reasonably stable at 149°C.

23. Pigment Content

23.1 Pigment gives paint its hiding and color and influences many other properties. Determine the percent pigment in accordance with Test Method D 2371.

24. Binder Content

24.1 The nonvolatile vehicle is that portion of the film-forming solids in a paint other than the pigment. It is not to be confused with the nonvolatile portion of the vehicle. Subtract the pigment content from the nonvolatile content to obtain the nonvolatile vehicle content. If desired, separate the vehicle for further analysis in accordance with Method D 2372.

FIELD EVALUATIONS

25. Road Service Test

25.1 Whereas numerous laboratory tests in the previous

sections indicate general suitability of traffic paint, and also batch-to-batch uniformity, these tests cannot predict performance under all possible end uses. Accordingly, the test paint should be applied in a repeatable manner under carefully stated conditions of end use and then tested, observed, and evaluated at stated times throughout the useful life of the paint.

25.2 Proceed in accordance with Practice D 713, being careful to record the value of each variable stated.

26. Retroreflectance

26.1 Since there is no acceptable instrumental method of evaluating the retroreflectance in the field of the glass spheres on (in) the traffic paint stripe (Note 4), the following two visual methods are extensively used: (1) rating longitudinal stripes from a car traveling at approximately 20 to 35 mph and requiring a test line 50 ft (15 m) long, and (2) rating transverse test stripes in the wheel tracks with tungsten illumination from the side of the road with eye and light source (held chest high) and separated by a distance that corresponds to the observation angle of a driver viewing the stripes on a highway.

26.1.1 These ratings are based on a scale of 10 (complete (100 %) retroreflectance) to 0 (no retroreflectance).

NOTE 3—The wheel track is the area extending 9 in. (230 mm) to each side of the point of greatest wear.

NOTE 4—An instrumental method with much greater precision is being developed.

27. Durability

27.1 The test line rating is based on the paint film remaining at the time of inspection when estimated by close observation with the unaided eye. The rating is on the scale from 0 to 10, the latter representing 100 % remaining, a rating of 9 representing 90 % remaining, etc.

28. Appearance

28.1 This is the complete impression conveyed when the test stripe is viewed at a distance of at least 10 ft (3 m). Any discoloration of the surface due to bleeding, dirt collection, darkening, fading, mold growth, etc. will affect the rating that is also on the scale from 10 to 0.

29. Length of Useful Life

29.1 The length of useful life is the length of time (in days) between application of the test lines and when the weighted rating first reaches a value of 4, or when any specific quality (appearance, durability, or night visibility) first reaches the numerical rating of 3, whichever is the lesser number of days.

29.2 When it is necessary to calculate the "length of useful life," calculate as follows (Practice D 713):

$$L = D \times (10 - 4) / (10 - R) = 6D / (10 - R)$$

where:

L = length of useful life,

D = number of days the test stripe has been on the road, and

R = weighted rating at time of calculation.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Freeze-Thaw Resistance of Water-Borne Coatings¹

This standard is issued under the fixed designation D 2243; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 3012 of Federal Test Method Standard No. 141 and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers the determination of the extent to which water-borne coating retain their original properties free of detrimental changes.

1.2 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 562 Test Method for Consistency of Paints Using the Stormer Viscometer²

D 2196 Test methods for Rheological Properties of Non-Newtonian Materials By Rotational (Brookfield) Viscometer²

3. Apparatus

3.1 **Test Chamber**—A suitable cabinet, room, or enclosure space large enough to contain the specimens to be tested permitting at least 1 in. (25 mm) of air space between the sides of adjacent cans and capable of being maintained continuously at a temperature of 0°F (−18°C).

NOTE 1—Although a variation of the test chamber temperature of $\pm 3.5^\circ\text{F}$ ($\pm 2^\circ\text{C}$) is allowed, the test chamber temperature should be maintained as near 0°F (−18°C) as practicable and the amount of variation should be recorded and reported.

3.2 **Viscometer**—A Stormer viscometer with paddle type rotor as described in Test Method D 562 or a Brookfield viscometer as described in Test Methods D 2196.

3.3 **Test Charts**—Smooth surface paper charts having adjacent black and white areas, and coated with a suitable varnish or lacquer to render the surface impermeable to paint liquids.

3.4 **Paint Brush**, 1 in. (25 mm).

4. Preparation of Sample and Specimens

4.1 Prepare specimens for testing by filling 1-pt (500 mL) resin-lined, friction-top cans two thirds full. Ensure that the bulk sample from which the cans are filled is well stirred and uniform, that the containers used are clean, and that the lids

are applied promptly to the cans to prevent evaporation losses. Two such specimens are required for each test.

5. Exposure to Test Conditions

5.1 Store one can at room temperature and identify this as the control specimen.

5.2 Place the second can, the paint under test, identified as the "test specimen", in the chamber maintained at 0°F (−18°C) in such a manner that it does not touch the walls or bottom of the chamber and so that free circulation of air around it is permitted. The placing of cans on racks that raise them off the bottom of the chamber or upon pieces of insulating board resting on the bottom is suggested. In the case of several test specimens, maintain a minimum of 1 in. (25 mm) of air space between adjacent cans and between cans and the chamber walls. Keep the test specimen in the chamber for 17 h and then remove and allow to stand for 7 h undisturbed at room temperature, adjacent to the control specimen, for a complete freeze-thaw cycle of 24 h.

5.3 Repeat 5.2 for additional freeze-thaw cycles, as many as agreed on between cooperating laboratories or buyer and seller (One to five cycles are usual.).

6. Examination

6.1 After completion of the agreed or specified number of cycles, before stirring, examine both specimens for condition in the can, observing and rating any evidence of settling, gelation, coagulation, lumpiness, etc. as slight, moderate or pronounced.

6.2 Then stir the specimens and determine their viscosities in accordance with Test Method D 562 or Test Methods D 2196, and record the temperature of measurement.

NOTE 2—Stir specimens by hand in their can using a stainless steel paint spatula with a blade measuring approximately 5 by $\frac{3}{4}$ in. (125 by 20 mm). Stir carefully so as to avoid air entrainment and foam.

NOTE 3—In carrying out consistency determinations using Test Method D 562, specimens should be maintained in 77°F (25°C) until two successive readings agree within 5 g. As is the case with all non-Newtonian fluids, viscosity variation is dependent to a degree upon intensity and duration of agitation. Utmost care should be taken that control and test specimens receive identical treatment during all stirring operations.

6.3 Immediately following the viscosity determinations, apply both specimens of paint to a test panel (3.3) by means of a clean 1-in. (25-mm) brush. Allow to dry at least 24 h, then compare the test specimen with the control. Note any changes in hiding power, gloss, speckiness, agglomeration, coagulation, or color as slight, moderate or pronounced.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as D 2243 - 64 T. Last previous edition D 2243 - 82 (1987)¹.

² Annual Book of ASTM Standards, Vol 06.01.

7. Report

7.1 Report on the condition of the paint in the can, in accordance with 6.1.

7.2 Report the viscosities of the test and control specimens, and the temperature of measurement, in accordance with 6.2.

7.3 Report comparatively on the visible dry film characteristics of the test and control specimens, in accordance with 6.3.

8. Precision

8.1 The precision of this test method in regard to viscosities is as specified in Test Methods D 562 or D 2196, whichever is employed.

8.2 No precision statement is made in regard to the other properties, because of the subjective nature of the observations.

9. Keywords

9.1 freeze-thaw resistance; package stability

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Standard Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates¹

This standard is issued under the fixed designation D 2244; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{ε1} NOTE—Superscript for b^2 in 6.4 was editorially corrected in December 1990.

INTRODUCTION

This test method originally resulted from the consolidation of a number of separately published methods for the instrumental evaluation of color differences. As revised in 1979, it included four color spaces in which color-scale values could be measured by instruments, many of which were obsolete, and the color differences calculated by ten equations for different color scales. The sections on apparatus, calibration standards and methods, and measurement procedures served little purpose in the light of modern color-measurement technology. The present revision omits these sections, and limits the color spaces and color-difference equations considered, to the three most widely used in the paint and related coatings industry.

1. Scope

1.1 This test method covers the calculation, from instrumentally measured color coordinates based on daylight illumination, of small color differences between nonfluorescent, nonmetameric, opaque specimens such as painted panels. (Where it is suspected that the specimens may be metameric, that is, possess different spectral curves though visually alike in color, Practices D 1729 and D 4086 should be used to verify instrumental results.) The color differences determined by these procedures are expressed in terms of approximately uniform visual color perception in CIE 1976 CIELAB opponent-color space (1),² Hunter L_H , a_H , b_H opponent-color space (2), and the Friele-MacAdam-Chickering (FMC-2) color space (3).

1.2 For product specification, the permissible color difference between test specimen and reference and the procedure for calculating the color difference shall be agreed upon by the purchaser and the seller. Specific color tolerances may be required for each material and condition of use since other appearance factors (for example, proximity, gloss, and texture) may affect the correlation between the magnitude of a measured color difference and its commercial acceptability.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish*

appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³
- D 3964 Practice for Selection of Coating Specimens for Appearance Measurements⁴
- D 4086 Practice for Visual Evaluation of Metamerism³
- E 179 Guide for Selection of Geometric Conditions for Measurement of Reflection and Transmission Properties of Materials³
- E 284 Definitions of Terms Relating to Appearance of Materials³
- E 308 Method for Computing the Colors of Objects by Using the CIE System³

3. Terminology

3.1 **Definitions**—For the following definitions as well as for other definitions of terms used in this test method, see Definitions E 284: tristimulus values, chromaticity coordinates, and opponent-color scales.

3.2 Description of Term Specific to This Standard:

3.2.1 color difference:

3.2.1.1 *color difference (perceived)*—the magnitude and character of the difference between two colors described by such terms as redder, bluer, lighter, darker, grayer, or cleaner.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 2244 - 64 T. Last previous edition D 2244 - 85.

² The boldface numbers in parentheses refer to the list of references at the end of this method.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

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3.2.1.2 *color difference (computed)*—the magnitude and direction of the difference between two psychophysical color stimuli defined by tristimulus values, or by chromaticity coordinates and luminance factor, as computed by means of a specified set of color-difference equations.

4. Summary of Test Method

4.1 The differences in color between a reference and a test specimen are determined from measurements made by use of a spectrophotometer or a colorimeter. Reflectance readings from such instruments are converted by computations to color-scale values, or these color-scale values may be read directly from instruments that automatically make the computations. Color-difference magnitudes are computed, from differences in these color-scale values, that represent the perceived color differences between the reference and the test specimen.

5. Significance and Use

5.1 The original CIE color scales based on tristimulus values X , Y , Z and chromaticity coordinates x , y are visually nonuniform. Each subsequent color scale based on CIE values has had weighting factors applied to provide some degree of uniformity so that color differences in various regions of color space will be more nearly comparable. On the other hand, color differences obtained for the same specimens evaluated in different color-scale systems are not likely to be identical. To avoid confusion, color differences among specimens should be compared only when they are obtained for the same color-scale system. There is no simple factor that can be used to convert accurately color differences in one system to differences in another system for all colors of specimens.

5.2 For uniformity of practice, the CIE recommended in 1976 the use of two new improved color scales, of which the CIELAB scale, with its associated color-difference equation, has found wide acceptance in the coatings and related industries. However, it has not completely displaced the use of the Hunter L_H , a_H , b_H and the FMC-2 scales, which are still utilized to allow comparison with earlier results and because of their familiarity to those responsible for interpreting them. Therefore, all three scales are included in this method as the most widely used in industrial laboratories.

5.3 Users of color differences have found that, in each system, summation of three vector color-difference components into a single scalar value is useful for determining whether a specimen color is within a specified tolerance from a standard. However, for control of color in production, it is necessary to know not only the magnitude of the departure from standard but also the direction of this departure. Information on the direction of a color difference is easily included by giving the three instrumentally determined components of the color difference.

5.4 Selection of color tolerances based on instrumental values should be carefully correlated with a visual appraisal of the acceptability of differences in hue, lightness, and saturation obtained by using Practice D 1729.

6. Description of Color-Difference Equations

6.1 CIE 1931 and 1964 Color Spaces—The daylight

colors of opaque specimens are represented by points in a space formed by three rectangular coordinates representing the lightness scale Y and chromaticity scales x and y , where:

$$x = \frac{X}{X + Y + Z}$$

$$y = \frac{Y}{X + Y + Z}$$

$$Y = Y$$

where X , Y , and Z are tristimulus values for either the 1931 CIE standard observer (2° observer) or the 1964 CIE supplementary standard observer (10° observer) and standard illuminant C , D_{65} , or another of daylight quality. These scales do not provide a perceptually uniform color space. Consequently, color differences are seldom if ever computed directly from differences in x , y , and Y .

6.2 CIE 1976 $L^* a^* b^*$ Uniform Color Space and Color-Difference Equation (1, 4)—This approximately uniform color space is a simplified version of the Adams-Nickerson color-scale system (5–7). It is produced by plotting in rectangular coordinates the quantities L^* , a^* , b^* , calculated as follows:

$$\begin{aligned} L^* &= 116(Y/Y_n)^{1/3} - 16 \\ a^* &= 500[(X/X_n)^{1/3} - (Y/Y_n)^{1/3}] \\ b^* &= 200[(Y/Y_n)^{1/3} - (Z/Z_n)^{1/3}] \end{aligned}$$

$$X/X_n, Y/Y_n, Z/Z_n > 0.01$$

The tristimulus values X_n , Y_n , Z_n define the color of the normally white object-color stimulus. Usually, the white object-color stimulus is given by the spectral radiant power of one of the CIE standard illuminants, for example, C , D_{65} or another of daylight quality, reflected into the observer's eye by the perfect reflecting diffuser. Under these conditions, X_n , Y_n , Z_n are the tristimulus values of the standard illuminant with Y_n equal to 100.

6.2.1 The total difference ΔE_{ab}^* between two colors each given in terms of L^* , a^* , b^* is calculated as follows:

$$\Delta E_{ab}^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

NOTE 1—The color space defined above is called the CIE 1976 $L^* a^* b^*$ space and the color-difference equation the CIE 1976 $L^* a^* b^*$ color-difference formula. The abbreviation CIELAB is recommended.

6.2.2 The CIE 1976 ($L^* a^* b^*$) space fails to approximate uniform color spacing when one or more of the ratios X/X_n , Y/Y_n , and Z/Z_n is less than 0.01. In calculating L^* , values of Y/Y_n less than 0.01 may be included if the normal formula is used for values of Y/Y_n greater than 0.008856, and the following modified formula is used for values of Y/Y_n equal to or less than 0.008856.

$$L^* = 903.3(Y/Y_n) \quad Y/Y_n \leq 0.008856$$

6.2.3 In calculating a^* and b^* , values of X/X_n , Y/Y_n , Z/Z_n less than 0.01, may be included if the normal equations are replaced by the following modified equations for all calculations of a^* and b^* :

$$\begin{aligned} a^* &= 500[f(X/X_n) - f(Y/Y_n)] \\ b^* &= 200[f(Y/Y_n) - f(Z/Z_n)] \end{aligned}$$

where:

$$\begin{aligned} f(X/X_n) &= (X/X_n)^{1/3} & X/X_n > 0.008856 \\ f(X/X_n) &= 7.787(X/X_n) + 16/116 & X/X_n \leq 0.008856 \\ f(Y/Y_n) &= (Y/Y_n)^{1/3} & Y/Y_n > 0.008856 \end{aligned}$$

$$f(Y/Y_n) = 7.787(Y/Y_n) + 16/116 \quad Y/Y_n \leq 0.008856$$

$$f(Z/Z_n) = (Z/Z_n)^{1/3} \quad Z/Z_n > 0.008856$$

$$f(Z/Z_n) = 7.787(Z/Z_n) + 16/116 \quad Z/Z_n \leq 0.008856$$

NOTE 2—To compare values of $\Delta E(AN40)$ calculated by means of the Adams-Nickerson (AN40) equation with ΔE_{ab}^* , multiply $\Delta E(AN40)$ values by the factor 1.1 to facilitate the comparison.

6.2.4 The magnitude, ΔE_{ab}^* , gives no indication of the character of the difference since it does not indicate the relative quantity and direction of hue, saturation, and lightness differences.

6.2.5 The direction of the color difference is described by the magnitude and algebraic signs of the components ΔL^* , Δa^* , and Δb^* :

$$\Delta L^* = L_1^* - L_0^*$$

$$\Delta a^* = a_1^* - a_0^*$$

$$\Delta b^* = b_1^* - b_0^*$$

where L_0^* , a_0^* , and b_0^* refer to the reference, and L_1^* , a_1^* , and b_1^* refer to the test specimen. The signs of the components ΔL^* , Δa^* , and Δb^* have the following approximate meanings (8):

$$+\Delta L^* = \text{lighter}$$

$$-\Delta L^* = \text{darker}$$

$$+\Delta a^* = \text{redder (less green)}$$

$$-\Delta a^* = \text{greener (less red)}$$

$$+\Delta b^* = \text{yellow (less blue)}$$

$$-\Delta b^* = \text{bluer (less yellow)}$$

6.2.6 For judging the direction of the color difference between two colors, it is useful to calculate their CIE 1976 hue angles h_{ab} and CIE 1976 chromas C_{ab}^* as follows:

$$h_{ab} = \tan^{-1}(b^*/a^*)$$

$$C_{ab}^* = [(a^*)^2 + (b^*)^2]^{1/2}$$

Differences in hue angle Δh_{ab} between the test specimen and reference can be correlated with differences in their visually perceived hue, except for very dark colors (9). Differences in chroma ΔC_{ab}^* can similarly be correlated with differences in visually perceived chroma.

6.2.7 For judging the relative contributions of differences in lightness, chroma, and hue to the total color difference between two colors, it is useful to calculate the CIE 1976 hue difference ΔH_{ab}^* between them as follows:

$$\Delta H_{ab}^* = [(\Delta E_{ab}^*)^2 - (\Delta L^*)^2 - (\Delta C_{ab}^*)^2]^{1/2}$$

where ΔE_{ab}^* is calculated as in 6.2.1 and C_{ab}^* is calculated as in 6.2.6; then the equation

$$\Delta E_{ab}^* = [(\Delta L^*)^2 + (\Delta C_{ab}^*)^2 + (\Delta H_{ab}^*)^2]^{1/2}$$

contains terms showing the relative contributions of lightness difference ΔL^* , chroma difference ΔC_{ab}^* , and hue difference ΔH_{ab}^* to the total color difference ΔE_{ab}^* .

6.3 Hunter L_H , a_H , b_H Color Space and Color-Difference Equation (2)—This approximately uniform color space is produced by plotting in rectangular coordinates the quantities L_H , a_H , b_H calculated as follows:

$$L_H = 10(Y)^{1/3}$$

$$a_H = 17.5(1.02X - Y)/(Y)^{1/3}$$

$$b_H = 7.0(Y - 0.847Z)/(Y)^{1/3}$$

where X , Y , and Z are tristimulus values for the CIE 1931 standard observer and standard illuminant C.

NOTE 3—The subscript L was used to denote Hunter in previous issues of this method.

6.3.1 The total difference ΔE_H between two colors each given in terms of L_H , a_H , b_H is calculated as follows:

$$\Delta E_H = [(\Delta L_H)^2 + (\Delta a_H)^2 + (\Delta b_H)^2]^{1/2}$$

6.3.2 The magnitude and direction of the color difference are described by considerations similar to those found in 6.2.4 and 6.2.5, respectively.

6.4 Friele-MacAdam-Chickering Color Space and Color-Difference Equation (3, 10)—This color space is more nearly perceptually uniform than the CIE 1931 space in terms of the MacAdam chromaticity-difference values (11). It is produced by a linear transformation of CIE 1931 tristimulus values X , Y , Z into tristimulus values P , Q , S as follows:

$$P = 0.724X + 0.382Y - 0.098Z$$

$$Q = -0.48X + 1.37Y + 0.1276Z$$

$$S = 0.686Z$$

Approximate lightness difference ΔL_{FMC-2} and chromatic differences ΔC_1 ("yellow-blue") and ΔC_3 ("red-green") are calculated as follows:

$$\Delta L_{FMC-2} = 0.279K_2(P\Delta P + Q\Delta Q)/aD$$

$$\Delta C_1 = K_1S(P\Delta P + Q\Delta Q)/bD^2 - K_1\Delta S/b$$

$$\Delta C_3 = K_1(Q\Delta P - P\Delta Q)/aD$$

where:

$$a^2 = 17.3 \times 10^{-6}(P^2 + Q^2)/[1 + 2.73P^2Q^2/(P^4 + Q^4)]$$

$$b^2 = 3.098 \times 10^{-4}(S^2 + 0.2015Y^2)$$

$$D = (P^2 + Q^2)^{1/2}$$

$$K_1 = 0.55669 + 0.049434Y - 0.82575 \cdot 10^{-3}Y^2$$

$$+ 0.79172 \times 10^{-5}Y^3 - 0.30087 \cdot 10^{-7}Y^4$$

$$K_2 = 0.17548 + 0.027556Y - 0.57262 \cdot 10^{-3}Y^2$$

$$+ 0.63893 \times 10^{-5}Y^3 - 0.26731 \cdot 10^{-7}Y^4$$

NOTE 4—The correlations between ΔL_{FMC-2} and perceived lightness, between ΔC_1 and perceived yellowness-blueness, and between ΔC_3 and perceived redness-greenness, are not well established and should not be used unless confirmed by visual observations.

NOTE 5—When $K_1 = K_2 = 1$, the FMC-1 equations are obtained.

6.4.2 The total difference ΔE_{FMC-2} between two colors is calculated as follows:

$$\Delta E_{FMC-2} = [(\Delta L_{FMC-2})^2 + (\Delta C_1)^2 + (\Delta C_3)^2]^{1/2}$$

7. Test Specimens

7.1 This method does not cover preparation techniques. Unless otherwise specified or agreed, prepare specimens in accordance with Practice D 3964.

8. Procedure

8.1 Select appropriate geometric conditions for color measurement in accordance with Guide E 179.

8.2 Operate the instrument in accordance with the manufacturer's instructions.

8.3 Standardize the instrument with a working standard. Restandardize at sufficient intervals to minimize the effects of instrumental drift.

8.4 When a spectrophotometer is used, obtain the reflectance values of the reference specimen and test specimens, in turn, at a sufficient number of wavelength intervals to permit accurate calculation of CIE tristimulus values. See Method E 308.

8.5 When a reflectance-reading filter colorimeter is used, obtain the reflectance values of the reference specimen and test specimens, in turn, with each of the filters.

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8.6 When a color-scale-reading colorimeter is used, obtain scale values of the reference and test specimens, in turn.

8.7 For any instrument used, remeasure the working standard to determine whether instrumental drift has occurred during measurement. Compensate for instrumental drift, if observed, or remeasure specimens.

8.8 Measure at least three portions of each specimen surface to obtain an indication of uniformity. Record the location where these measurements were made on the specimen.

9. Calculation

9.1 Calculate color-scale values L^* , a^* , b^* , or L_H , a_H , b_H , or tristimulus values P , Q , S , if not obtained automatically.

9.2 Calculate color differences ΔE_{ab}^* , ΔE_H , or ΔE_{FMC-2} and their components, if not obtained automatically, as described in 6.2, 6.3, or 6.4, respectively.

10. Report

10.1 Report the following information:

10.1.1 Total color difference ΔE_{ab}^* , ΔE_H , or ΔE_{FMC-2} , of each test specimen from its reference.

10.1.2 For CIELAB color differences, L^*_0 , a^*_0 , b^*_0 for the reference, ΔL^* , Δa^* , Δb^* , and if desired Δh_{ab}^* , ΔC_{ab}^* , and ΔH_{ab}^* for each specimen.

10.1.3 For Hunter color differences, $L_{H,0}$, $a_{H,0}$, $b_{H,0}$ and ΔL_H , Δa_H , Δb_H for each specimen.

TABLE 1 Precision of Calculated Color Differences Determined for Various Conditions of Measurement and Analysis

Measurement Conditions			ΔE Equation ^a	No. of Instruments	Mean ΔE	Standard Deviation	MAD ^a
Geometry	Illuminant	Observer					
45°/0°	C	1931	Hunter	26	0.94	0.04	0.12
45°/0°	C	1931	CIELAB	17	0.91	0.09	0.27
45°/0°	D ₆₅	1931	CIELAB	8	0.95	0.01	0.03
Sphere ^c	C	1931	Hunter	5	0.94	0.10	0.39
Sphere ^c	C	1931	CIELAB	34	0.91	0.06	0.17
Sphere ^c	D ₆₅	1931	CIELAB	7	0.92	0.03	0.10
Sphere ^c	D ₆₅	1964	CIELAB	21	0.88	0.02	0.06

^a Maximum acceptable difference.

^b The Hunter and CIELAB equations give essentially the same value of ΔE for the sample pair tested.

^c Specular component included for integrating-sphere measurements.

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Standard Test Method for Finishes on Primed Metallic Substrates for Humidity-Thermal Cycle Cracking¹

This standard is issued under the fixed designation D 2246; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes conditions for testing an organic coating on a primed metallic substrate for cracking from humidity-thermal cycling or from loss of plasticizer, or both.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

3. Summary of Test Method

3.1 The tendency of an organic coating to develop cracking from humidity-thermal cycling or from loss of plasticizer, or both, is measured by alternate exposure in the humidity cabinet and in the cold box. At the end of the exposure period, the panels are rated for their degree of cracking.

4. Significance and Use

4.1 This test method provides an accelerated means of determining the tendency of an organic coating to fail by cracking when exposed to humidity-thermal cycling.

5. Apparatus

5.1 *Humidity Cabinet*, of such design and construction as to maintain a relative humidity of 100 % with condensation on the test specimens at all times and a temperature of $100 \pm 1^\circ\text{F}$ ($38 \pm 0.5^\circ\text{C}$).

5.2 *Cold Box*, of such design and construction that a temperature of $-10 \pm 3^\circ\text{F}$ ($-23 \pm 1.5^\circ\text{C}$) can be maintained.

NOTE—Most commercial household freezers are capable of maintaining this temperature.

5.3 *Rack*, as shown in Fig. 1, for supporting test specimens at an angle of 0 to 30° from the vertical position in

such a manner as to prevent contact between them during the test procedure.

5.4 *Grid*, of thin wires 3 by $8\frac{1}{2}$ in. (75 by 215 mm) constituting one hundred and two $\frac{1}{2}$ -in. (13-mm) squares, held in a frame $\frac{1}{2}$ in. wide, with outside dimensions 4 by $9\frac{1}{2}$ in. (100 by 240 mm).

6. Test Specimens

6.1 The test specimens shall be either samples of metallic products, prepared and coated in accordance with the specified production schedule, or coated metallic panels. The recommended panel size is 4 by 12 in. (100 by 305 mm).

7. Preparation of Test Specimens

7.1 The test specimens shall consist of steel meeting the requirements of Methods D 609, cleaned and prepared for coating in accordance with Procedure A of Method D 609. If desired, the metal panels selected for coating with organic coatings may be of any selected composition and may be cleaned and prepared according to commercial practice or by any procedure that is mutually agreed upon.

7.2 After coating, the specimens shall not be cleaned, and they shall be handled only by the edges.

8. Procedure

8.1 Support panels or specimens so that they will be held in a position 0 to 30° from the vertical in such a manner as to prevent contact between the panels during the test.

8.2 Place the panels in a humidity cabinet at 100°F (38°C) and 100 % relative humidity for a period of $24 \pm \frac{1}{2}$ h. Then transfer them to the cold box at $-10 \pm 3^\circ\text{F}$ ($-23 \pm 1.5^\circ\text{C}$) allowing a maximum of 30 s for the transfer. Leave the panels in the cold box for a period of $20 \pm \frac{1}{2}$ h. Remove the panels and allow to remain at room temperature for a period of $4 \pm \frac{1}{2}$ h, during which time rate them. This constitutes one cycle. Run the test for 15 cycles or as mutually agreed upon. During any interruption of the normal cycling, as on weekends, always leave the panels in the cold box.

9. Evaluation of Test Specimens

9.1 Lay the specimen upon a horizontal surface, finish side up, with the framed grid (5.4) placed upon it so that the bottom $8\frac{1}{2}$ in. (225 mm) is covered by mesh. (The frame itself excludes the first $\frac{1}{2}$ in. (13 mm) from edges of the specimen which may have been handled, or which may otherwise not be representative of the general area.) Make an evaluation by counting the number of grid squares within which one or more cracks is visible. This number shall constitute the rating.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 2246 - 64 T. Last previous edition D 2246 - 65 (1981)¹.

² Annual Book of ASTM Standards, Vol 06.01.

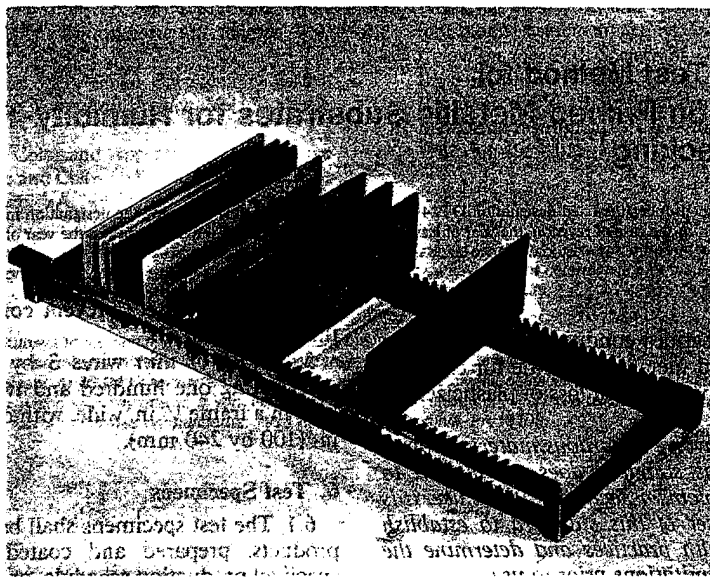


FIG. 1 Aluminum Rack for Supporting Test Specimens

9.2 If the specimen is necessarily smaller than 4 by 12 in. (100 by 305 mm), or is irregular in outline, handle in accordance with 3.4 and 9.1, and assign as the rating the percentage of those grid squares under which the specimen lies, that contain one or more cracks.

9.3 It is recommended that coatings be evaluated by comparison with a coating system known to have acceptable performance. For referee tests this coating system should be included in each test.

10. Report

10.1 Report the following information:

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

10.1.1 Temperature readings within the exposure zone of the humidity cabinet,

10.1.2 Temperature readings within the exposure zone of the cold box,

10.1.3 Method of supporting or suspending panel or specimen during test,

10.1.4 Exposure period, and

10.1.5 Ratings as described in 9.1 or 9.2.

11. Precision and Bias

11.1 The precision and bias of this test method have not been established.

1. Scope

1.1 This procedure is for testing the adhesion of a coating system to a substrate under conditions of 100 % relative humidity.

1.2 This test method is intended for use in specifying the performance of a coating system under conditions of 100 % relative humidity.

NOTE 1—Coatings include:

1.3 This test method is intended for use in specifying the performance of a coating system under conditions of 100 % relative humidity.

2. Refer

2.1 ASTM D 609 M Paint, D 610 T Paint, D 714 T Paint, D 823 Thiel Test, D 870 T Using, D 1193 min, D 1730 min, D 1735 Using, D 2616 Diff:

¹ This practice is related to ASTM D 01.27 Accelerated Current Exposure Test published as:

² Annual Book of ASTM Standards
³ Annual Book of ASTM Standards
⁴ Annual Book of ASTM Standards
⁵ Annual Book of ASTM Standards



Standard Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity¹

This standard is issued under the fixed designation D 2247; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice covers the basic principles and operating procedures for testing water resistance of coatings by exposing coated specimens in an atmosphere maintained at 100 % relative humidity so that condensation forms on the test specimens.

1.2 This practice is limited to the methods of obtaining, measuring, and controlling the conditions and procedures of tests conducted in 100 % relative humidity. It does not specify specimen preparation, specific test conditions, or evaluation of results.

NOTE 1—Alternative practices for testing the water resistance of coatings include Practices D 870, D 1735, and D 4585.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion²
- D 1193 Specification for Reagent Water³
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴
- D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus²
- D 2616 Test Method for Evaluation of Visual Color Difference With a Gray Scale⁵

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 Accelerated Testing.

Current edition approved Nov. 27, 1987. Published January 1988. Originally published as D 2247 - 66 T. Last previous edition D 2247 - 86a.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁴ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁵ Annual Book of ASTM Standards, Vol 14.02.

D 3359 Test Method for Measuring Adhesion by Tape Test²

D 3363 Test Method for Film Hardness by Pencil Test²

D 4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation²

3. Summary of Practice

3.1 Coated specimens are placed in an enclosed chamber containing a heated, saturated mixture of air and water vapor. The temperature of the chamber is usually maintained at 100°F (38°C). At 100 % relative humidity (RH), a very small temperature difference between the specimen and the surrounding vapor causes the formation of condensation on the specimens. The exposure condition is varied by selecting the duration of the test. Water permeates the coating at rates that are dependent upon the characteristics of the coating. Any effects such as color change, blistering, loss of adhesion, softening, or embrittlement are observed and reported.

4. Significance and Use

4.1 Water can cause the degradation of coatings, so knowledge of how a coating resists water is helpful in predicting its service life. Failure in tests at 100 % relative humidity may be caused by a number of factors including a deficiency in the coating itself, contamination of the substrate, or inadequate surface preparation. This practice is therefore useful for evaluating coatings alone or complete coating systems.

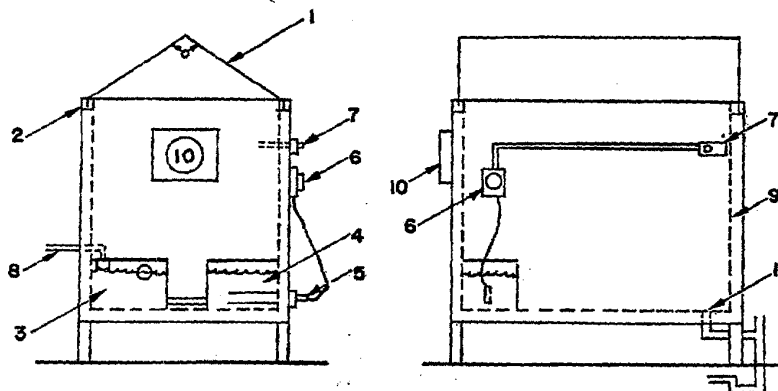
4.2 Tests at 100 % relative humidity are used for specification acceptance, quality control, and research and development of coatings and substrate treatments. These tests usually result in a pass or fail determination but the degree of failure may also be measured. A coating system is considered to pass if there is no evidence of water-related failure after a period of time.

4.3 Results obtained from the use of 100 % humidity tests in accordance with this practice should not be represented as being equivalent to a period of exposure to water in the natural environment, until the degree of quantitative correlation has been established for the coating or coating system.

4.4 The test chamber can be a small laboratory cabinet or a room large enough to hold an automobile or a truck. Some automobile manufacturers test completed vehicles in rooms maintained at 100 % relative humidity. Corrosion tests can be conducted, as the condensate dripping off the test articles is not recirculated.

5. Apparatus

5.1 *Test Chamber*, constructed of corrosion-resistant materials with supports for the test specimens.



- 0—Angle of lid, 90 to 125°
- 1—Hinged top, hydraulically operated, or counterbalanced
- 2—Water seal
- 3—Constant-level water tank unheated with overflow/outlet and equalizer connection
- 4—Heater water tank for supplying heat and humidity to cabinet
- 5—Immersion heater
- 6—Water temperature limit control
- 7—Thermostatic controller for room temperature. Primary limit control for immersion heater (5)
- 8—Water line
- 9—Insulation if necessary (see A1.3)
- 10—Temperature recorder (optional)
- 11—Drain

FIG. A1.1 Humidity Cabinet

5.2 *Heated Water Tank*, within the test chamber, a water supply, and a water level control.

5.3 *Thermostatic Control*, for the water heater with the sensor located adjacent to the specimen holders.

5.4 *Thermometer*, with sensor located adjacent to the specimen holders.

5.5 Diagrams and details of the apparatus are shown in the Annex.

6. Test Specimens

6.1 This practice does not cover the preparation of test specimens. The substrate composition and surface preparation, specimen preparation, and the number of specimens should be agreed upon prior to testing.

NOTE 2—Applicable methods for the preparation of test panels and substrates are given in Method D 609 and Practices D 1730. Test Methods D 823 cover application techniques for the production of uniform films.

7. Procedure

7.1 Fill the water tank with reagent water conforming to Type IV of Specification D 1193. Tap water may be used, but this will result in the accumulation of residues in the water tank over a period of time.

7.2 Adjust the temperature of the saturated air and water vapor mixture to $38 \pm 1^\circ\text{C}$ ($100 \pm 1^\circ\text{F}$).

NOTE 3—Due to heat loss to the specimens and the walls of the chamber, the temperature of the water in the tank will be above the temperature of the air and water vapor mixture.

7.3 Support the specimens approximately 15° from the vertical. Slotted wood supports are suitable for flat specimens. Provide for formation of condensation on the speci-

mens by spacing them at least 30 mm apart. Space the specimens so they do not touch each other, any metallic material, or any material capable of acting as a wick. Arrange specimens so that condensate from one specimen cannot drip on other specimens.

7.4 Droplets of condensation should be visible on the specimens at all times if the chamber is operating properly. Operate the test continuously with the test chamber closed unless otherwise specified. Short interruptions to inspect or remove specimens are permitted.

7.5 Conclude the test after a specified period of time or after effects from exposure to water are observed.

7.6 Wipe the test specimens dry. Rate specimens for changes in color, blistering, etc. Evaluate specimens no less than 5 min and no more than 10 min after removal from test, as the effects from water exposure can change within a short time. Remove only as many specimens as can be rated within the specified time.

NOTE 4—The 0 to 10 scale described in ASTM STP 500⁶ is preferred for rating. Relevant procedures for evaluating water effects are described in Test Methods D 610, D 714, D 2616, D 3359, and D 3363.

7.6.1 If possible, rate the specimens again after they have been removed from the test for a recovery period long enough that moisture absorbed within the specimens dries out and the specimens reach moisture equilibrium with room air. A recovery period from 12 to 24 h is generally sufficient. The post-recovery rating allows evaluation of the permanent effects of the exposure as distinct from the transient effects, and is especially important for evaluation of color and gloss.

⁶ Paint Testing Manual, ASTM STP 500, ASTM, 1972.

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8. Report

8.1 Report the following information:

8.1.1 Sample identification.

8.1.2 Results of the evaluation(s).

8.1.3 Reference to Practice D 2247.

8.1.4 Hours of test duration.

8.1.5 Test temperature.

8.1.6 Special conditions of test or any deviations in test procedure.

ANNEX

(Mandatory Information)

A1. Apparatus

A1.1 The apparatus must be constructed so that heated water vapor is generated at the bottom of the chamber. This saturates the air immediately above the water tank with water vapor. The saturated mixture of water vapor and air

temperature rises and then cools below the dew point, causing condensation on the specimens.

A1.2 The area of the heated water tank should be limited to no more than 25 % of the floor area of the chamber. The use of a large heated water tank would tend to make the temperature within the chamber uniform, and thus inhibit or prevent the formation of condensate on the specimens. The water temperature will be approximately from 10 to 20°F (5 to 10°C) above the vapor temperature when the water tank and chamber are properly proportioned.

A1.3 Insulation of the enclosure is not required and can possibly interfere with the formation of condensate by reducing the temperature differential within the chamber. It is difficult to produce condensation with small chambers because the temperature differential is slight.

A1.4 Large walk-in chambers may require more than one heated water tank to generate the convective currents needed to cause condensation at all points within the chamber. Circulating fans should be used with caution as their use may reduce the temperature differential and limit condensation.

A1.5 In a properly operating chamber, condensation is observed on the specimens or parts when the chamber is opened for inspections. It may be necessary to avoid placing specimens in the lower portion of the chamber as the temperature differentials at the lower levels may be too small to induce condensation.

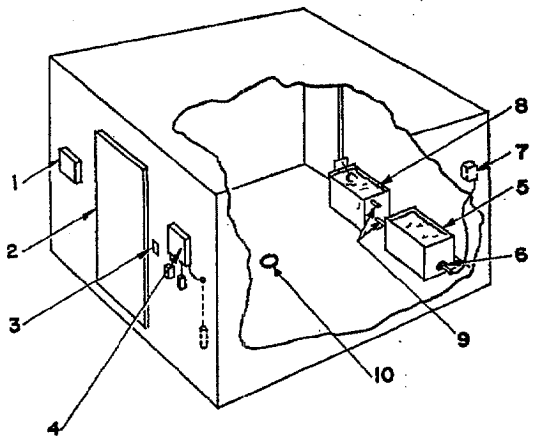
A1.6 In the event that enough condensation does not occur, or condensation does not form at all the desired points, it is possible to increase condensation by:

A1.6.1 Opening the chamber to allow the temperature to drop,

A1.6.2 Turning off the water heater periodically to cause temperature fluctuations, or

A1.6.3 Removing the specimens to allow them to cool down to room temperature.

A1.7 Many variations in the design of the apparatus are possible in the use of this practice. Two typical designs are shown in Figs. A1.1 and A1.2.



- 1—Temperature recorder
- 2—Fiberglass door with magnetic closers and rubber seal; inward sloping sill
- 3—Light switch
- 4—Thermostatic controller for room temperature. Primary limit control for immersion heater (6)
- 5—Heated water tank for supplying heat and humidity to room
- 6—Immersion heater
- 7—Water temperature limit control
- 8—Constant level water tank unheated with overflow outlet
- 9—Equalizer connection
- 10—Floor drain

NOTE—The chamber shall feature waterproof construction with insulation and vaporproof fixtures on the interior, lighting may be accomplished with a fluorescent fixture above insulating glass sealed in the ceiling. Polyvinyl chloride pipe and flanges shall be used for sleeves through walls.

FIG. A1.2 Walk-in Humidity Chamber

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Detergent Resistance of Organic Finishes¹

This standard is issued under the fixed designation D 2248; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the determination of the resistance to failure, in an accelerated manner, of organic finishes when immersed in a detergent solution.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 523 Test Method for Specular Gloss²
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer and Related Products²
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- D 1417 Methods of Testing Rubber Latexes—Synthetic³
- D 1474 Test Methods for Indentation Hardness of Organic Coatings²
- D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments²
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴
- D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Paint²
- D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion²

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved March 31, 1989. Published May 1989. Originally published as D 2248 - 64 T. Last previous edition D 2248 - 73 (1982).

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 09.01.

⁴ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates²

3. Significance and Use

3.1 Any effects such as color change, blistering, loss of adhesion, softening, or embrittlement are observed and noted.

4. Apparatus

4.1 **Container**—A corrosion-resistant container equipped with the means to control the solution temperature within the range specified and to control the liquid level at $\pm 3/16$ in. (5 mm). Agitation may be required to maintain temperature uniformity.

4.2 **Cover**—The container shall be provided with a cover to retard evaporation and to contain the test specimens completely.

5. Test Specimens

5.1 Unless otherwise specified, the test specimens shall be 4 by 12 in. by 20 gage (100 by 300 by 0.9 mm) in size. The test specimen composition, surface preparation, and number of test specimens shall be agreed upon by the purchaser and the seller.

NOTE 1—Applicable test panel description and surface preparation methods are as follows:

Methods D 609, Practices D 1730 and D 2092.

6. Coating of Test Specimens

6.1 The method of application, film thickness, curing, and conditioning of the test surface shall be agreed upon by the purchaser and the seller.

NOTE 2—Application and film thickness measurement methods are given as follows:

Test Methods D 823, D 1005, D 1186, and D 1400.

6.2 The backs, cut edges, and those areas containing identification marks or in contact with the supports, shall be protected with a suitable coating that is stable under the conditions of test.

7. Detergent Solution

7.1 Composition, concentration, and temperature of detergent solution shall be agreed upon by the purchaser and the seller.

7.2 A typical solid detergent composition is as follows:

Parts by Weight	
Tetrasodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$), anhydrous	53.0
Sodium sulfate (Na_2SO_4), anhydrous	19.0
Sodium metasilicate (Na_2SiO_3), anhydrous	7.0
Sodium carbonate (Na_2CO_3), anhydrous	1.0
Sodium salt of a linear alkylarylsulfonate (90 % flake grade)	20.0
Total	100.0

A suggested temperature of the detergent solution is $165 \pm 2^\circ\text{F}$ ($74 \pm 1^\circ\text{C}$).

8. Procedure

8.1 *Immersion*—Suspend the test specimens vertically in the container so that at least one half of the surface area is submerged in the detergent solution. Separate the test specimens so that they are not in contact with any metal and are no closer together than 1 in. (25 mm) at any point in the bath. Replace the detergent solution with fresh detergent solution every 168 h. If successive tests are to be correlated, use reference panels coated with a control paint.

8.2 *Examination of Specimens*—When the specimens are ready for examination, carefully remove, gently wash or dip in clean running water not warmer than the temperature of the detergent solution to remove the detergent from the surface, and then carefully dry by blowing with air or blotting with absorbent paper. During the progress of the test, examine for deterioration of the film immediately. If reimmersion is necessary, do not allow the specimens to remain out of the liquid in excess of $\frac{1}{2}$ h, unless otherwise specified. Examine the test coating for the failures specifically agreed upon by the purchaser and the seller.

NOTE 3—The following methods of evaluating finish degradation in this method are applicable:

Test Methods D 523, D 714, D 1474, and D 2197, and Methods D 610, D 1654, and D 2244.

8.3 *Length of Test*—The length of test and the intermediate examination periods shall be as designated by the specification covering the organic finish being tested or as mutually agreed upon by the purchaser and the seller.

9. Precision and Bias

9.1 Precision data are based on a round robin in which

three finishes applied to two substrates were tested in six laboratories with each laboratory making two runs of five replicates of each variation. Blister failure was reported by all cooperators using Test Method D 714. To simplify the evaluation of blistering data the blister ratings were converted to single numerical values by the formula $\text{Frequency Number} \times \text{Size} \div 10 = \text{Numerical Value}$. The Frequency Numbers assigned were as follows:

No blistering	10
Very few	9
Few	8
Few medium	6
Medium	5
Medium dense	4
Dense	2

By this system, few Size 8 blistering would have a numerical value of 6.4. The adequacy of precision depends upon the purpose of the test and should be determined by the user from the data supplied.

9.1.1 Using these numerical values the standard deviations for the three finishes were as follows (rounded to nearest-tenth):

Finish	Within Laboratory	Between-Laboratory
1	0.8	1.4
2	0.4	0.6
3	1.3	2.6

9.1.2 Repeatability:

Finish	Difference
1	2.8
2	1.5
3	4.5

9.1.3 Reproducibility:

Finish	Difference
1	4.9
2	2.2
3	9.0

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Standard Practice for Specifying Properties from Liquid Through Cured State for Coatings Factory Applied to Wood Products¹

This standard is issued under the fixed designation D 2336; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Keywords were added editorially in August 1991.

1. Scope

1.1 This practice summarizes the test methods that may be used to assist in quality control during application and in specifying pigmented coatings that shall be used in factory finishing of wood products.

1.2 This practice is not intended for use by the consumer of coated wood products in specifying such coated wood products.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 154 Guide for Testing Varnishes²
- D 523 Test Method for Specular Gloss²
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer²
- D 869 Test Method for Evaluating Degree of Settling of Paint²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1200 Test Method for Viscosity by Ford Viscosity Cup²
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems²
- D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- D 1474 Test Methods for Indentation Hardness of Organic Coatings²
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products²

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.52 on Factory-Coated Wood Building Products.

Current edition approved Oct. 30, 1987. Published December 1987. Originally published as D 2336 - 65 T. Last previous edition D 2336 - 81.

² Annual Book of ASTM Standards, Vol 06.01.

D 1644 Test Methods for Nonvolatile Content of Varnishes²

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer²

D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²

D 2691 Test Methods for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products²

D 2793 Test Method for Block Resistance of Organic Coatings on Wood Substrates²

D 2801 Test Method for Leveling Characteristics of Paints by Draw-Down Method⁴

D 2805 Test Method for Hiding Power of Paints by Reflectometry²

D 3003 Test Method for Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates²

D 3259 Practice for Infrared Determination of the Temperature of Applied Coatings on Wood Products During the Curing Cycle²

D 3278 Test Methods for Flash Point of Liquids by Set-Flash Closed-Cup Apparatus⁵

D 3359 Test Methods for Measuring Adhesion by Tape Test²

D 3960 Practice for Determining Volatile Organic Content (VOC) of Paints and Related Coatings²

2.2 U.S. Federal Test Method:

141b/3011 Condition in Container⁶

3. Significance and Use

3.1 Control of the parameters that are measured by the test methods outlined in this practice has been found to be the primary determinant of coating quality and reproducibility in the package, during coating application and on the coated product. Accurate measurement is essential if such control is to be achieved.

³ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

⁴ Discontinued; see 1989 Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 06.03.

⁶ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

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4. Characteristics in the Package

4.1 *Skinning, Settling, and Condition in the Container:*

4.2 Settling is determined in accordance with Test Method D 869, and by Federal Test Method No. 141b, Method 3011 which also covers condition in the container. Both of these methods are designed for trade sales type paints rather than industrial coatings, but could be adapted satisfactorily.

4.3 Skinning can occur in a partially filled container. Remove insoluble skins and perform a skinning test on a well-mixed portion in accordance with Guide D 154.

4.4 *Consistency*—Consistency of a paint varies considerably depending upon the type of instrument used to measure it. Consistency is determined in accordance with Test Method D 562 and Test Method D 1200. For most coatings there is not a straight line relationship between Stormer viscosity, a rotational method, and Ford cup viscosity, an efflux method. (The Zahn efflux cup and the Brookfield Rotational Viscometer (Test Methods D 2196) are also used widely by the paint industry.)

4.4.1 Most factory wood finishing is done with airless spray, curtain coating, or roller coating techniques. It is doubtful that any single method of viscosity measurement is truly indicative of the ability of coatings to be applied by production methods or, more especially, to be applied satisfactorily by curtain coating.

4.4.2 These methods are useful in that once a certain formula has been established, control of the viscosity by either method helps to ensure subsequent reproduction of that formula. In this way, they would be useful in specifying previously qualified coatings for factory finishing of wood products.

4.5 *Fineness of Dispersion*—The fineness of dispersion determined in accordance with Test Method D 1210, is based on visual observation of a few of the largest size particles remaining in a coating after dispersion. In itself, the test does not indicate the degree of dispersion of the bulk of the pigment in the coating except that when batches are dispersed in similar equipment the ratio of large particles remaining in the coating to the dispersion of the bulk of the pigment tends to remain constant. Therefore, if in an enamel with satisfactory gloss no particles remain that are above a size of 0.5 mil (7 Hegman dispersion), then successive batches of that coating having a similar Hegman rating can be said to have a good enamel grind. Hiding power and color control along with fineness of dispersion control should be adequate to ensure proper dispersion.

4.6 *Weight per Gallon*—Weight per gallon is determined in accordance with Test Method D 1475 to ensure batch-to-batch and composition product uniformity. This method is not related to the quality of the coatings per se.

4.7 *Nonvolatile Content*—Non-volatile content is determined in accordance with Test Methods D 1644 as another measure of coating uniformity and of the amount of film-forming material provided.

4.8 *Volatile Organic Content (VOC)*—VOC is determined in accordance with Practice D 3960 both to ensure coating uniformity and to comply with governmental regulation where required.

4.9 *Flash Point*—Flash point is determined in accordance with Test Methods D 3278 as a measure of the degree of

flammability or fire hazard of a coating material.

5. Coating Characteristics During and Immediately After Application

5.1 *Flow*—Flow is a property related to consistency, but it is influenced by other factors such as rate and order of solvent release. Visual observation of the leveling of a coating film during and after drying and the tendency of the film to sag during application and drying are usually adequate to determine flow. However, Test Method D 2801 may be used if desired.

5.2 *Gloss*—Specular gloss may be determined in accordance with Test Method D 523.

5.3 *Hiding Power*—Hiding power determined in accordance with Test Method D 2805 is a measure of the ability of a paint to hide the substrate. It is dependent upon uniform film thickness which is influenced by flow and leveling.

5.4 *Wet Film Thickness*—The thickness of a wet paint film is determined in accordance with Test Methods D 1212 as a rapid indication that the required amount of coating has been applied.

6. Coating Characteristics During and Immediately After Curing

6.1 *Curing Temperature*—The temperature of a coating film during the curing cycle is an important quality control parameter and should be monitored. Practice D 3259, discusses several infrared measuring instruments that can be used for such monitoring. Infrared measuring instruments can be based on the measurement of carefully filtered narrow bands of infrared energy emitted from test surfaces and can be unaffected by surface color or gloss. Thermocouples have been used but are subject to the defects that metals are good heat conductors and do not necessarily absorb heat from infrared radiation at the same rate as the paint film. Better results are obtained with portable pyrometers if they are preheated slightly above the operating temperature. Papers impregnated with wax powders of varying melting points have been suggested also. These tend to absorb infrared energy faster than most wet paint films and, therefore, give higher temperatures than actual.

6.2 *Film Thickness*—Measurement of film thickness in accordance with Test Methods D 1005, is designed for use on plane rigid surfaces such as metal or glass. It can be adapted to forest products by the simple use of an auxiliary panel of metal or glass placed alongside the wood substrate and coated in the identical way that the wood substrate is coated. Test Methods D 1400 or D 1186 also could be adapted to the forest products industry in a similar manner. This is a simpler but perhaps less accurate method. This adaptation leads to a measurement of application rate rather than to a true measurement of film thickness of the coating on a wood product because of the possibility of penetration by the coating into the wood product. Test Methods D 2691 gives a method for measuring the actual film thickness present on a wood products substrate.

6.3 *Hardness*—The measurement of hardness of coating films by means of an indenter, as described in Test Methods D 1474, calls for the measurement of film hardness on smooth, rigid substrates such as glass or metal. There may be some question as to its applicability on films applied to some

of the softer forest products substrates. However, if the films are thick enough (1.5 mils or greater), it should be applicable on the harder forest products substrate.

6.4 Color Difference—Color difference between a product and the standard can be measured instrumentally. Generally, the tolerance is agreed upon by the purchaser and the seller and may also be required if a product specification is involved. Although color instruments are not more sensitive than the eye, and Practice D 1729 may be used for visual evaluation of color differences, color instruments do provide numerical values that can be subsequently compared to later measurements. Test Method D 2244 covers the instrumental determination of small color differences observable in daylight illumination between nonfluorescent, nonmetameric, or opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation should be used to verify the results.

6.5 Adhesion—There are no available ASTM test methods

directly applicable to measuring adhesion on forest products substrates. It has been found that the available test methods have poor precision even on hard, smooth substrates. They would probably be even less satisfactory on wood substrates. Two methods that might give some indication of adhesion are Test Methods D 2197 and D 3359.

6.6 Blocking—Many coated wood products are immediately stacked after curing. Often they are subject to "blocking" or sticking together if improperly coated or cured. Test Method D 2793⁷ may be used to detect and quantify the degree of blocking. It may be used as a control test to detect this condition during the coating operation.

7. Keywords

7.1 cured properties; factory application; wood coatings; wood products

⁷ Subcommittee D01.52 is developing a revision to Test Method D 3003 to replace Test Method D 2793.

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Standard Test Method for Freeze-Thaw Stability of Multicolor Lacquers¹

This standard is issued under the fixed designation D 2337; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the extent to which multicolor lacquers resist coagulation and coalescence when subjected to freezing and subsequent thawing.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

- 2.1 *ASTM Standard:*
D 562 Test Method for Consistency of Paints Using the Stormer Viscometer²

3. Summary of Test Method

3.1 Specimens of multicolor lacquers are placed in a chamber maintained at a temperature between -10 and -15°F (-23 and -26°C) and allowed to remain 24 h. The specimens are removed from the chamber and allowed to remain at room temperature for 24 h to reach thermal equilibrium. After the specimens are mixed and the viscosity adjusted, they are then sprayed and allowed to dry before comparing with the control standard sprayed from the original specimen.

4. Significance and Use

4.1 Waterborne coatings, when subjected to cycles of freezing and thawing, can undergo detrimental changes in application and performance characteristics. This test method evaluates the ability of multicolor lacquers to resist these changes.

5. Apparatus and Materials

5.1 *Cabinet, Room, or Enclosed Space*, large enough to contain the specimens to be tested and allowing at least 1 in. (25 mm) of air space between the sides of adjacent cans. It should be capable of being maintained at a temperature between -10 and -15°F (-23 and -26°C).

5.2 *Stormer Viscometer*, with paddle-type rotor.

5.3 *Spray Gun*, pressure-feed internal-mix type, tip 0.086 in. (2 mm) in diameter with wide-spray nozzle.

5.4 *White Paper Stock*, minimum size 8 by 11 in. (200 by 280 mm).

6. Preparation of Samples

6.1 For all gloss and semigloss multicolor lacquers, prepare ten 1-qt (1-L) specimens and for flat multicolor lacquers four 1-qt specimens for testing. Mix the sample from which the specimens are filled well by boxing so that it is of a uniform consistency and appearance. After recording the consistency reading as determined with a Stormer viscometer (in accordance with Test Method D 562 fill all samples in baked varnish-lined containers and apply the lids promptly to prevent evaporation loss).

7. Preparation of Control Standard

7.1 Prepare a control standard by spraying from one of the 1-qt (1-L) specimens of each product to be tested. Use a coated white paper stock and spray on a sheet at least 8 by 11 in. (200 by 280 mm) in size. Adjust the fluid and atomizing pressures to give a spreading rate of 150 to 200 ft^2/gal (3.7 to 4.9 m^2/L) for complete covering and 200 to 300 ft^2/gal (4.9 to 7.35 m^2/L) for scatter coat application.

8. Exposure of Samples to Test Conditions

8.1 Place nine specimens of all gloss and semigloss multicolor lacquers and three specimens of all flat multicolor lacquers under test in the chamber that is maintained between -10 and -15°F (-23 and -26°C). Place the specimens on racks in such a manner that they do not touch the walls or bottom of the chamber and so that there is at least 1 in. (25 mm) of air space between adjacent specimens. On each cycle allow all specimens to remain in the chamber for 24 h before removal. Remove from the chamber all specimens of each multicolor lacquer and let them come to thermal equilibrium by allowing them to remain at room temperature (77°F (25°C)) for 24 h. Mix these specimens to a uniform consistency by boxing. Run a minimum of three specimens and all specimens shall pass the test for multicolor to be acceptable.

9. Examination of Samples and Spray After Test

9.1 Check the consistency of all specimens on the Stormer viscometer after each test cycle and adjust if necessary by adding water to the viscosity of the original before testing. Spray three specimens after each cycle on white paper stock using the same equipment and adjustments of the spray gun that were used to spray the control standard. Allow the sprayed panels to dry thoroughly (16 h) and compare with the control standard in each case for color, particle size, and pattern of the background as well as the accent colors.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Nov. 14, 1984. Published January 1985. Originally published as D 2337 - 65 T. Last previous edition D 2337 - 68 (1979) ϵ 1.

² *Annual Book of ASTM Standards*, Vol 06.01.

10. Report

10.1 Report significant change in color, particle size, or tendency to show coalescence or coagulation when compared with the control standard. To be considered satisfactory for freeze-thaw resistance, all gloss and semigloss multicolor lacquers shall show a satisfactory appearance after three cycles and all flat multicolor lacquers shall show a satisfactory appearance after one cycle.

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11. Precision

11.1 It is recognized that the seller has no control over the conditions under which the purchaser will apply multicolor lacquer finishes and, therefore, meaningful estimates of precision cannot be given. In round-robin tests fairly good agreement was obtained by the cooperators as long as the same equipment was used to spray the multicolor before and after the test.



Standard Test Method for Determining Particle Size of Multicolor Lacquers¹

This standard is issued under the fixed designation D 2338; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout, including the title, in April 1989.

1. Scope

1.1 This test method employs photographic reference standards to evaluate the particle size of multicolor lacquers.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Adjuncts:*
Grain Sizes (5 photos)²

3. Summary of Test Method

3.1 The samples to be tested are mixed by pouring from one container to another until they are uniform in consistency before spraying on coated white paper stock. After the lacquers are dry they are compared with photographic reference standards to determine the particle size.

4. Significance and Use

4.1 This test method provides a uniform nomenclature for the producer and user in the identification of the various particle sizes used in the manufacture of nitrocellulose-type multicolor lacquers.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Aug. 31, 1984. Published January 1985. Originally published as D 2338 - 65 T. Last previous edition D 2338 - 68 (1979)^{e1}.

² Glossy prints of the photographic reference standards showing various particle sizes are available at nominal charge from ASTM Headquarters. Request Adjunct No. 12-423380-00.

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5. Reference Standards

5.1 The photographic reference standards² were prepared by photographing actual sprayed panels of multicolor lacquers that were made in the five particle sizes normally used in commercial multicolor lacquers. The standard designations are as follows (Figs. 1 (a), (b), (c)): fine, small, medium, large, and extra large, and represent the correct nomenclature for properly applied multicolor lacquer.

6. Apparatus

6.1 *Spray Gun*, pressure-feed internal-mix type, tip 0.086 in. (2.2 mm) in diameter with wide-spray nozzle.

7. Procedure

NOTE—The particle size of multicolor lacquer is dependent upon the spray conditions during the application procedure. In order to get uniform results the exact spray conditions should be agreed upon between the purchaser and the seller.

7.1 Spray the lacquer on a coated white paper panel with the fluid and atomizing pressures adjusted to give a spreading rate of 150 to 200 ft²/gal (3.7 to 4.9 m²/L) for complete covering and 200 to 300 ft²/gal (4.9 to 7.35 m²/L) for scatter coat application. After the sprayed lacquer is thoroughly dry, determine the particle size on the panel by comparing it to the photographic reference standards.²

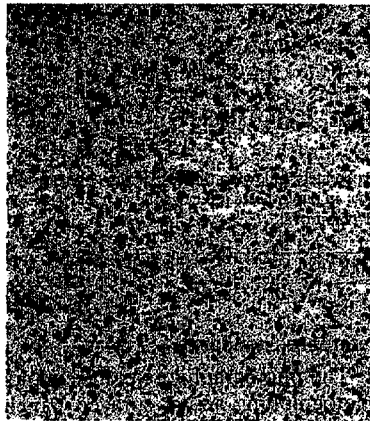
8. Precision

8.1 This test method entails the use of visual judgment and as such does not readily lend itself to a precision statement.

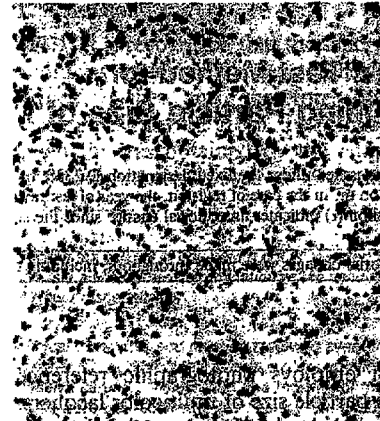
8.2 In general there has been good agreement between operators in judgment of particle size.

9. Keywords

9.1 particle size; multicolor lacquers; lacquer

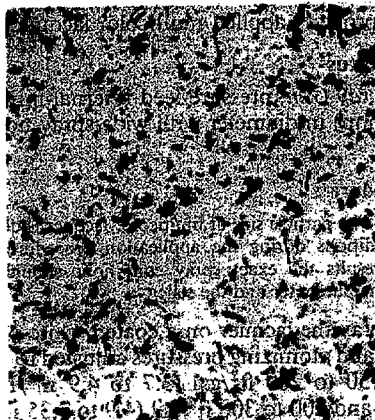


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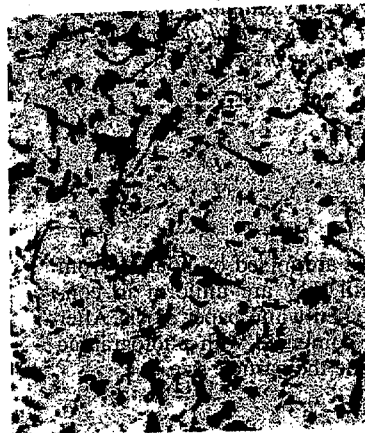


Small

FIG. 1(a) Particle Size (for other particle sizes, see Figs. 1(b) and 1(c))



Medium



Large

FIG. 1(b) Particle Size (for other particle sizes, see Figs. 1(a) and 1(c))



Extra large

FIG. 1(c) Particle Size (for other particle sizes, see Figs. 1(a) and 1(b))

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Standard Test Method for Arsenic in Paint¹

This standard is issued under the fixed designation D 2348; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of less than 0.5 % arsenic in whole paint.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The specimen of liquid paint is prepared for analysis by acid-digestion. The arsenic content of a distillate from the acid-digested sample is determined by titration with KBrO_3 solution.

4. Significance and Use

4.1 The permissible level of toxic elements in certain coatings is specified by governmental regulatory agencies. This test method provides a documented procedure for determining low concentrations of arsenic present in whole paint to determine compliance.

5. Apparatus

5.1 *Kjeldahl Flask*, 800-mL, with standard-taper 24/40 joint.

5.2 *Condenser Delivery Tube*, with standard-taper 24/40 joint.

5.3 *Mechanical Shaker*.

5.4 *Gas Burner*, suitable for heating Kjeldahl flask.

5.5 *Glass or Disposable Syringes*, 2 mL capacity.

5.6 *Erlenmeyer Flasks*, 250 and 500 mL capacity.

5.7 *Pipets*, 10 mL capacity.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chem-

ical Society, where such specifications are available.³ Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water conforming to Type II of Specification D 1193.

6.3 *Arsenous Oxide, Standard Solution*—Dissolve 0.3241 g of arsenous oxide (As_2O_3) in 25 mL of sodium hydroxide (NaOH) solution (100 g/L). Make slightly acid with sulfuric acid (H_2SO_4 , 1+8), and dilute to 1.0 L with water.

6.4 *Hydrazine Sulfate-Potassium Bromide Solution*—Dissolve 20 g of hydrazine sulfate ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{SO}_4$) and 23 g of potassium bromide (KBr) in 200 mL of concentrated hydrochloric acid (HCl , sp gr 1.19) (Precaution—see 7.1) and dilute to 1 L.

6.5 *Methyl Orange Indicator Solution*—Dissolve 0.1 g in 100 mL water.

6.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3) (Precaution—see 7.1).

6.7 *Potassium Bromate, Standard Solution*—Dissolve 0.18 g of potassium bromate (KBrO_3) in water and dilute to 1 L with water. Standardize as follows: Transfer 10.0 mL of standard arsenous oxide solution by pipet to a 250-mL flask. Add 100 mL of water and 25 mL of HCl (sp gr 1.19). Heat to 90°C and titrate with KBrO_3 as described in 8.5. Calculate the standardization factor, F , as follows:

$$F = (0.0032410 \times 0.7574) / V$$

where:

0.0032410 = As_2O_3 in 10.0 mL of solution, g,

0.7574 = factor converting As_2O_3 to As, and

V = KBrO_3 required for the titration, mL.

6.8 *Sodium Chloride* (NaCl).

6.9 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4) (Precaution—see 7.1).

7. Hazards

7.1 Handling concentrated acids can be hazardous. Refer to their individual Material Safety Data Sheets (MSDS).

8. Procedure

8.1 Mix the sample until it is homogeneous, preferably on a mechanical shaker.

8.2 Weigh to 1 mg from a glass syringe, 1.0 g of whole paint directly into the 800-mL Kjeldahl flask, being careful

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Material.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 2348 - 65 T. Last previous edition D 2348 - 85.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

not to get any on the sides of the flask. Add approximately 10 boiling aids.

8.3 Digest by adding the following: 40 mL of HNO_3 (allow to stand 20 min) and 25 mL of H_2SO_4 (allow to stand 5 min). Boil; when charring occurs, add HNO_3 cautiously until the solution becomes clear and begins to reflux within the flask. The flask should be at about a 45° angle while digesting. Cool, carefully add 150 mL of water, and boil to sulfur trioxide (SO_3) fumes.

8.4 Add 100 mL of water and a thermometer to an Erlenmeyer flask. After the solution in the Kjeldahl flask has cooled, add 25 mL of water, 25 g of NaCl, and 25 mL of the hydrazine sulfate-potassium bromide solution, and connect the distillation tube so that the delivery end is immersed in the receiving water. Quickly heat the Kjeldahl flask over a small, well-protected flame, and distill into the Erlenmeyer flask (Note 1). Adjust the flame so that the temperature of the distillate solution will rise to 80°C in 9 to 11 min, and then discontinue distillation by first removing the thermometer, lifting the distillation tube out of solution slightly and turning the heat off.

NOTE 1—Although it may be necessary to boil the solution, heating is primarily to bring about evolution of hydrogen chloride (HCl) gas, which carries over the arsenic trichloride (AsCl_3). Absorption of evolved HCl gas by water causes a rise in temperature which indicates progress of distillation. If distillation proceeds further, or if a larger quantity of H_2SO_4 than specified is used in digestion, sulfur dioxide (SO_2) will be distilled and titrated as arsenic.

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8.5 Titrate the distillate at once with KBrO_3 solution, using 3 drops of methyl orange indicator solution. Toward the end of the titration add the KBrO_3 solution very slowly with constant agitation to prevent a local excess. The end point is reached when a single drop of the KBrO_3 just destroys the final tinge of red. Correct the results for the volume of KBrO_3 used in a blank determination (digest 5 g of pure sucrose, using the same reagents and procedure).

NOTE 2—The blank titration should not exceed 0.7 mL of KBrO_3 solution. The method is accurate down to variations in the blank, which should not exceed 0.1 mL when reagents from the same lot are used.

9. Calculation

9.1 Calculate the percent of arsenic in the sample as follows:

$$\text{Arsenic, \%} = [(V - B) \times F] / S \times 100$$

where:

V = KBrO_3 solution required for the sample, mL,

B = KBrO_3 solution required for the blank, mL,

S = sample used, g, and

F = factor as calculated in 6.7.

10. Precision and Bias

10.1 Precision and bias of this test method have not been determined.

11. Keywords

11.1 arsenic; paint

1. Scope

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Standard Test Method for Qualitative Determination of Nature of Solvent Composition in Solvent-Reducible Paints¹

This standard is issued under the fixed designation D 2349; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the nature of solvent in solvent-reducible house paints containing only hydrocarbon solvents.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 215 Methods of Chemical Analysis of White Linseed Oil Paints²
- D 233 Methods of Sampling and Testing Turpentine³
- D 1193 Specification for Reagent Water⁴

3. Significance and Use

3.1 This test method provides a procedure for determining qualitatively the composition of solvent in paints containing only hydrocarbon solvents.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

4.3 *Antimony Pentachloride* (SbCl_5).

4.4 *Chloroform*.

4.5 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

4.6 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

5. Preparation of Sample

5.1 Mix and prepare the sample in accordance with Methods D 215.

6. Nature of Solvent

6.1 Transfer approximately 150 g of the paint to a 500-mL flask fitted with a 2-hole cork stopper carrying a spray trap connected with a suitable condenser. Through the other hole in the stopper pass an influx tube for steam. (This tube should dip below the surface of the paint.) Heat the flask in an oil or air bath at 100°C and pass through it a current of steam; with the steam still passing through, raise the temperature of the bath to 130°C. Catch the distillate in a small separatory funnel; continue distillation until 300 mL of water have been obtained. Portions of this water may be drawn from the cock of the separatory funnel from time to time, but care must be taken not to draw out any of the volatile solvent.

6.2 Let the distillate stand until it separates into two layers, then draw off the water and filter the volatile solvent through a dry filter paper into a dry flask. If the solvent is apparently turpentine, examine the distillate in accordance with Color, Odor, Specific Gravity, Refractive Index and Distillation sections of Methods D 233. If the solvent is a mixture of turpentine and petroleum spirits, an approximate determination of the amount of turpentine may be made in accordance with the Polymerization sections of Methods D 233. (It should be noted that turpentine is slightly soluble in water, about 0.3 to 0.4 mL/100 mL of water.)

6.3 To test for benzene, add a few drops of the distillate to a small quantity of a mixture of HNO_3 (sp gr 1.42) and H_2SO_4 (sp gr 1.84) and heat cautiously. The characteristic odor of nitrobenzene will be noted if benzene is present.

NOTE 1—Toluene may give a similar odor.

6.4 If the solvent is apparently all petroleum spirits, no further examination is necessary unless further information is desired.

6.5 When the amount of turpentine in the solvent is so small that its presence is questionable, it may be detected by placing 2 drops of the distillate and 2 to 3 mL of chloroform in a dry test tube and adding 1 drop of SbCl_5 . A slow or slight change in color indicates the absence of turpentine. A rapid change in color to a dark red or purple indicates the possibility of turpentine. The iodine number for turpentine

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 25, 1990. Published July 1990. Originally published as D 2349 - 65 T. Last previous edition D 2349 - 72 (1984)⁶.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc. Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

by the Wijs method under these conditions is approximately 340. An iodine number of 20 or over is additional proof of the presence of turpentine and enables calculation of the approximate amount.

7. Keywords

7.1 hydrocarbon solvent; solvent composition

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Last ASTM Designation: D 2353 - 83

**Standard Test Method for
Flow Ratings of Organic Coatings Using the Shell Flow
Comparator**

This test method covers the determination of the flow rating, in terms of flow-out characteristics or degree of film smoothness, of an organic film applied to a flat substrate.
Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials, this test method was discontinued in 1992.

Standard Test Method for Accelerated Testing of Moisture Blister Resistance of Exterior House Paints on Wood

This test method covers only an accelerated evaluation of the resistance to moisture blistering of exterior house paints for use on wood substrates.

Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials, this test method was discontinued in 1989.

Refer to Practice D 4585¹.

¹ *Annual Book of ASTM Standards*, Vol 06.01.



Standard Test Method for Volatile Content of Coatings¹

This standard is issued under the fixed designation D 2369; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 404 1.1 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method describes a procedure for the determination of the weight percent volatile content of solvent-reducible and water-reducible coatings. Test specimens are heated at $110 \pm 5^\circ\text{C}$ for 60 min.

NOTE 1—The coatings used in these round-robin studies represented air-dried, air-dried oxidizing, and heat-cured baking systems.

1.2 Sixty minutes at $110 \pm 5^\circ\text{C}$ is a general purpose test method based on the precision obtained with both solvent-reducible and water-reducible coatings (see Section 9). These coatings (single package, heat cured) are commonly applied in factories to automobiles, metal containers, flat (coil) metal and large appliances, and many other metal parts.

NOTE 2—Testing at $110 \pm 5^\circ\text{C}$ for 20 min was utilized for the establishment of the original test method in 1965. Precision data are not available and may not have been properly generated at the time. The nine paints tested then were all solvent-reducible. These conditions, 20 min at $110 \pm 5^\circ\text{C}$, are no longer satisfactory for the determination of the volatile content of many coatings being tested at the present time. Water-reducible and solvent-reducible coatings were tested in the development of Test Method D 2369 using $110 \pm 5^\circ\text{C}$ for 60 min and 20 min for which precision data have been generated. See Appendix X1 for precision statements on the 20-min oven residence time.

1.3 This test method does not cover multi-package coatings wherein one or more parts may, at ambient conditions, contain liquid coreactants that are volatile until a chemical reaction has occurred with another component of the multipackage coating.

NOTE 3—Committee D-1 is running round-robin studies on the volatiles of multicomponent paint systems. The only change in procedure is to premix the weighed components in the correct proportions and allow the solvent reduced specimens in the aluminum pans to stand at room temperature for 1 h prior to placing them into the oven. Preliminary results with these changes appear to show that this test method is viable for multicomponent systems.

1.4 This test method may not be applicable to all types of coatings such as some printing inks (see Test Method D4713), and other procedures may be substituted with mutual agreement of the producer and user.

NOTE 4—If unusual decomposition or degradation of the specimen occurs during heating, the actual time and temperature used to cure the coating in practice may be substituted for the time and temperature

specified in this test method, subject to mutual agreement of the producer and user.

1.5 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement see Note 6.*

2. Referenced Documents

2.1 ASTM Standards:

D 343 Specification for 2-Ethoxyethyl Acetate (95 % Grade)²

D 362 Specification for Industrial Grade Toluene³

D 1193 Specification for Reagent Water⁴

D 4713 Test Methods for Nonvolatile Content of Printing Inks, Resin Solutions, and Vehicles⁵

E 145 Specification for Gravity Convection and Forced-Ventilation Ovens⁶

E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁷

3. Summary of Test Method

3.1 A designated quantity of coating specimen is weighed into an aluminum foil dish containing 3 mL of an appropriate solvent, dispersed, and heated in an oven at $110 \pm 5^\circ\text{C}$ for 60 min. The percent volatile is calculated from the loss in weight.

4. Significance and Use

4.1 This test method is the procedure of choice for determining volatiles in coatings for the purpose of calculating the volatile organic content in coatings under specified test conditions. The inverse value, nonvolatile, is used to determine the weight percent solids content. This information is useful to the paint producer and user and to environmental interests for determining the volatiles emitted by coatings.

5. Apparatus

5.1 *Aluminum Foil Dish*, 58 mm in diameter by 18 mm

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Sept. 12, 1990. Published November 1990. Originally published as D 2369 - 65 T. Last previous edition D 2369 - 87 ϵ 2.

² Discontinued; see 1982 Annual Book of ASTM Standards, Part 29.

³ Annual Book of ASTM Standards, Vol 06.03.

⁴ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Annual Book of ASTM Standards, Vol 15.05.

high with a smooth (planar) bottom surface. Precondition the dishes for 30 min in an oven at $110 \pm 5^\circ\text{C}$ and store in a desiccator prior to use.

5.2 *Forced Draft Oven*, Type IIA or Type IIB as specified in Specification E 145.

5.3 *Syringe*, 5-mL, capable of properly dispensing the coating under test at sufficient rate that the specimen can be dissolved in the solvent (see 7.2).

5.4 *Test Tube*, with new cork stopper.

5.5 *Weighing or Dropping Bottle*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁸ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

6.3 *Toluene*, technical grade, Specification D 362.

6.4 *2-Ethoxyethyl Acetate*, technical grade, Specification D 343.

7. Procedure

7.1 Mix the sample, preferably on a mechanical shaker or roller, until homogeneous. If air bubbles become entrapped, stir by hand until the air has been removed.

7.2 Using an appropriate weighing container (5.3, 5.4, or 5.5, with the syringe preferred for highest precision), weigh to 0.1 mg, by difference, a specimen of 0.30 ± 0.10 g for coatings believed to have a volatile content less than 40 weight % or a specimen of 0.50 ± 0.10 g for coatings believed to have a volatile content greater than 40 weight %, into a tared aluminum foil dish (5.5) into which has been added 3 ± 1 mL of suitable solvent (6.2, 6.3, or 6.4). Add the specimen dropwise, shaking (swirling) the dish to disperse the specimen completely in the solvent. If the material forms a lump that cannot be dispersed, discard the specimen and prepare a new one. Similarly prepare a duplicate.

NOTE 5—If the specimen cannot be dispersed in the solvents listed (6.2, 6.3, or 6.4) a compatible solvent may be substituted provided it is no less volatile than 2-ethoxyethyl acetate (6.4).

⁸ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

7.3 Heat the aluminum foil dishes containing the dispersed specimens in the forced draft oven (5.2) for 60 min at $110 \pm 5^\circ\text{C}$.

NOTE 6: *Precaution*—In addition to other precautions, provide adequate ventilation, consistent with accepted laboratory practice, to prevent solvent vapors from accumulating to a dangerous level.

7.4 Remove the dishes from the oven, place immediately in a desiccator, cool to ambient temperature, and weigh to 0.1 mg.

8. Calculation

8.1 Calculate the percent volatile matter, V , in the liquid coating as follows:

$$V = 100 - [(W_2 - W_1)/S] \times 100 \quad (1)$$

where:

W_1 = weight of dish,

W_2 = weight of dish plus specimen after heating, and

S = specimen weight.

8.2 The percent of nonvolatile matter, N , in the coating may be calculated by difference as follows:

$$N = 100 - V \quad (2)$$

9. Precision and Bias

9.1 The precision estimated for tests at 60 min at $110 \pm 5^\circ\text{C}$ are based on an interlaboratory study⁹ in which 1 operator in each of 15 laboratories analyzed in duplicate on 2 different days 7 samples of water-based paints and 8 samples of solvent-based paints containing between 35 and 72 % volatile material. The paints were commercially supplied. The results were analyzed statistically in accordance with Practice E 180. The within-laboratory coefficient of variation was found to be 0.5 % relative at 213 degrees of freedom and the between-laboratories coefficient of variation was 1.7 % relative at 198 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

9.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 1.5 % relative.

9.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 4.7 % relative.

9.2 *Bias*—Bias has not been determined.

10. Keywords

10.1 nonvolatile determination; VOC baking temperature; VOC in paints; volatile determination

⁹ Supporting data are available from ASTM Headquarters. Request RE D01-1026.

APPENDIX

(Nonmandatory Information)

X1. PRECISION STATEMENT FOR 20-MIN OVEN RESIDENCE

X1.1 Oven residence time of 20 min for the paint test specimen at $110 \pm 5^\circ\text{C}$ was the original procedure for this test method. For information purposes, the precision statements for 20-min residence time are as follows:

- (1) X1.1.1 The precision estimates are based on an interlaboratory study in which 1 operator in each of 15 laboratories analyzed in duplicate on 2 different days 7 samples of water-based paints and 8 samples of solvent-based paints containing from 35 to 72 % volatile material. The paints were commercially supplied. The results were analyzed statistically in accordance with Practice E 180. The within-laboratory coefficient of variation was found to be 1.1 %

relative at 193 df and the between-laboratory coefficient of variation was 2.5 % relative at 178 df. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

(a) *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days, should be considered suspect if they differ by more than 2.9 % relative.

(b) *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories, should be considered suspect if they differ by more than 7.1 % relative.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Tensile Properties of Organic Coatings¹

This standard is issued under the fixed designation D 2370; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense to replace Method 6224.1 of Federal Test Method Standards No. 141A. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Paragraph 12.1.2 was editorially changed in November 1987.

^{e2} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This method covers the determination of the elongation, tensile strength, and stiffness (modulus of elasticity) of organic coatings when tested as free films.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 882 Test Methods for Tensile Properties of Thin Plastic Sheeting³
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 3980 Practice for Interlaboratory Testing of Paint and Related Materials²

3. Summary of Method

3.1 Free unsupported films of the materials to be tested are prepared. The tensile properties of the free films are determined by means of a tensile testing apparatus.

4. Significance and Use

4.1 Tensile properties determined by this method are of value in studying the behavior of coatings subjected to environmental stresses, such as those produced by aging and weathering. (See Refs. (1) through (10).)⁴

4.2 Tensile properties may vary with specimen thickness, method of preparation, gage length, rate of load application,

tensile tester response, and type of grips used. Consequently, where precise comparative results are desired, these factors must be carefully controlled.

5. Description of Terms

5.1 *tensile strength (nominal)*—the load per original unit area at which a specimen fails or yields in a tension (pull) test.

5.2 *elongation*—the increase in specimen length from the point of initial load application to the point of film rupture in a tension test.

5.3 *stress-strain curve*—the curve resulting from a plot of tensile load against the distance of jaw separation (elongation of specimen).

5.4 *stiffness (modulus of elasticity)*—the load per unit area required to elongate the film 1 % from the first point in the stress-strain curve where the slope becomes constant.

5.5 *gage length*—the initial length of the test specimen between the jaws of the tensile tester.

6. Apparatus

6.1 Equipment for applying films of uniform thickness as described in Test Methods D 823.

6.2 *Micrometer Film Thickness Gage* as described in Method D 1005.

6.3 *Tensile Tester* of the constant rate of jaw separation type, equipped with load cells having capacities of 0.2 to 4.4 lb (100 to 2000 g), and equipped with an indicating device such as an electronic constant speed chart recorder, a digital device that displays numerical values, or a printer that records the numerical values.⁵

6.4 *Precision Specimen Cutter* having a double blade with a foot to hold the sample in place.⁶

6.5 *Alternative Substrates* on which test material can be deposited.

6.5.1 *Dental Tin Foil*, preferably 1 mil (25 μ m) thick.⁷

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved June 25, 1982. Published September 1982. Originally published as D 2370 - 65 T. Last previous edition D 2370 - 68 (1973).

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 08.01.

⁴ Boldface numbers in parentheses refer to the list of references at the end of this standard.

⁵ Suitable tensile testers are supplied by the Instron Engineering Corp., Canton, Mass., the Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, PA 19154, and the Tinius Olsen Testing Machine Co., P.O. Box 429, Willow Grove, PA 19090-0429.

⁶ The JDC Precision Cutter manufactured by Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, PA 19154, is satisfactory for this purpose.

⁷ A satisfactory source is Yates and Bird, Chicago, IL 60610.

6.5.2 *Sheet* preferably 2 m

NOTE 1—Other thick polyethylene fluoropolymer co

7. Hazards

7.1 *Mercury* vapors are ex mercury can exceed the TL (in a hood) a when handling Droplets of m pipet connect

8. Test Spec

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8.2 Prepar dures:

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8.2.1.4 A ally agreed t period, rem substrate an specimen ct

NOTE 3—A nick-free edge necessary to c mm) of waste

⁸ Teflon FEF Pont de Nemou
⁹ Dry lubric Chemical Co., I

6.5.2 *Sheet of FEP* (fluorinated ethylene-propylene),⁸ preferably 2 mils (50 μm) thick, coated with a dry lubricant.⁹

NOTE 1—Other substrates that may be suitable are 10-mil (250- μm) thick polyethylene (7), photographic paper (8), polished steel (9), and fluoropolymer coated metal panels.

7. Hazards

7.1 *Mercury*—Mercury is a toxic metallic liquid. Its vapors are extremely hazardous. Small amounts of spilled mercury can vaporize sufficiently at room temperature to exceed the TLV of the vapor. Use with adequate ventilation (in a hood) and clean up spills immediately. Wear gloves when handling mercury. Containers should be kept closed. Droplets of mercury can be picked up by using a small glass pipet connected to a suction flask with a rubber hose.

8. Test Specimens

8.1 The test specimens shall be free films having a width that is between $\frac{1}{2}$ and 1 in. (13 and 25 mm). No specimen shall vary by more than $\pm 2\%$ in width along its entire length. The length shall be at least 2 in. (50 mm) longer than the gage length selected for the test.

8.2 Prepare the free films by one of the following procedures:

8.2.1 Dental Tin Foil Substrate:

8.2.1.1 Apply each material to be tested to a strip of dental tin foil. First, mount the foil on a smooth glass plate on which there may be placed a small pool of high-boiling liquid, which may be useful in holding the foil in close contact with the glass. Spread the foil out to a smooth flat surface by means of a rubber squeegee. Apply a uniform coating of the material on the foil by automatic spray or drawdown procedures given in Test Method D 823.

NOTE 2—Use a liquid that is known to have little or no effect on the test coating. In many cases a light mineral oil would be suitable.

8.2.1.2 Dry the applied films at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity or bake under conditions mutually agreeable to producer and user. Dry film thicknesses must not vary by more than $\pm 5\%$ of the average film thickness. Most films with a thickness of less than 2.0 mils (50 μm) are very difficult to handle.

8.2.1.3 When required for further treatment such as weathering, remount the coated foil on a convenient smooth, flat substrate such as a glass or steel panel. In this case, a high boiling liquid (Note 2) may be used to hold the foil in close contact with the substrate over extended periods of time.

8.2.1.4 Age or expose the coated foil to conditions mutually agreed upon. At the end of the specified or agreed upon period, remove the foil-backed film from the glass or metal substrate and cut the specimens to size using the precision specimen cutter.

NOTE 3—A precision specimen cutter must be used to ensure nick-free edges on the specimens. Even with this instrument, it is necessary to cut each specimen independently, allowing at least $\frac{1}{4}$ in. (6 mm) of waste between specimens.

⁸ Teflon FEP 2-mil film thickness (Card No. 03111, Item #29499) from E. I. du Pont de Nemours & Co., Inc., Wilmington, DE 19898, is satisfactory.

⁹ Dry lubricant (MS-122 Fluorocarbon Release Agent) from Miller-Stephenson Chemical Co., Danbury, CT 06810, is satisfactory.

8.2.1.5 Place the foil-backed specimen film side up on a pool of mercury to remove the foil by amalgamation.

NOTE 4—Care should be exercised at all times to prevent the high-boiling liquids used in mounting the foil from coming in contact with the test film as these liquids may soften the film. For instance, the foil should be cleaned before amalgamation since any liquid remaining on the back of the foil and the test film will float on the bath of mercury.

8.2.1.6 After amalgamation is completed, pick up an end of the floating film with tweezers, and carefully brush the unsupported film free of mercury and amalgam with a soft camel hair brush.

8.2.2 FEP Sheet Substrate:

8.2.2.1 Apply each material to be tested to a sheet of FEP. First, cover a smooth, flat, polished glass plate with a sheet of FEP. Coat the sheet uniformly with a dry fluorocarbon lubricant and allow to dry for 24 h at standard conditions. Then apply a uniform coating of the test material on the sheet by automatic spray or drawdown procedures given in Method D 823.

8.2.2.2 Dry the applied films at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity or bake under conditions mutually agreeable to producer and user. Dry film thicknesses must not vary by more than $\pm 5\%$ of the average film thickness. Most films with a thickness of less than 2.0 mils (50 μm) are very difficult to handle.

8.2.2.3 When required for further treatment such as weathering, remount the coated sheet on a convenient smooth, flat substrate such as a glass or steel panel.

8.2.2.4 Age or expose the coated sheet to conditions mutually agreed upon. At the end of the aging or exposure period, remove the sheet-backed film from the glass or metal substrate, and cut the specimens to size using the precision specimen cutter (see Note 3). Carefully strip the coating film from the sheet substrate.

9. Calibration

9.1 Balance, zero, and calibrate the load weighing and recording system of the tensile tester in accordance with methods specified by the manufacturer.

10. Conditioning

10.1 Unless otherwise agreed upon between the producer and the user, condition the test specimens for at least 24 h at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and 50% relative humidity and test in the same environment.

11. Procedure

11.1 Select a mutually agreed upon gage length in the range of 1 in. (25 mm) to 5 in. (125 mm).

11.2 Prepare 10 test specimens for each material to be evaluated. These specimens should not exhibit any nicks or flaws. Measure the thickness of each specimen to ± 0.1 mil (2.5 μm) with a micrometer in accordance with Test Methods D 1005, taking five measurements within the gage length area.

11.3 Set the jaw separation of the tensile tester at the gage length selected. Place the test specimen in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. Tighten the grips evenly and firmly to the degree necessary to minimize

slipping of the specimen during test.

NOTE 5—Mounting is facilitated by the use of air activated jaws. Line-type jaws will minimize slippage and breakage. The application of pressure-sensitive cloth to the ends of the film can improve jaw grip.

11.4 Select a mutually agreed upon rate of elongation (strain rate) that is in the range of 5 to 100 %/min. Set the crosshead speed of the tensile tester to provide this rate for the gage length chosen.

NOTE 6—A rate of elongation should be selected that is optimum for testing the types of materials to be evaluated. For relatively brittle films, elongation rates of 5 to 20 % are suggested. For relatively extensible films, elongation rates of 50 to 100 % are suggested. (Refer to Test Methods D 882 for relation of elongation rate to elongation at break).

11.5 Elongate the test specimen until rupture of the film occurs and evaluate the stress-strain curve as follows:

11.5.1 Determine the specimen elongation by measuring the increase in jaw separation from the point of original load application to the point of rupture.

11.5.2 Measure the tensile pull in pounds (kg) required to rupture the film.

11.5.3 If stiffness is desired, determine the tensile pull in pounds (kg) to elongate the film 1 % from the first point in the stress-strain curve where the slope becomes constant.

11.6 Using the procedures in sections 11.1 through 11.5, run ten test specimens for each material under test.

12. Calculations

12.1 For each specimen compute the following:

12.1.1 The elongation E , in percent from the following equation:

$$E = 100 \left(\frac{\Delta L}{L} \right)$$

where:

ΔL = increase in specimen length to break, and

L = initial specimen length (gage length).

12.1.2 The tensile strength, T.S., in pounds per square inch, from the equation:

$$\text{T.S.} = (P_R)/(TW)$$

where:

P_R = tensile pull to rupture, lb (kg),

T = thickness of test specimen, in. (mm), and

W = width of test specimen, in. (mm).

12.1.3 The stiffness (modulus of elasticity), S , from the following equation:

$$S = (P_E)/(TW)$$

where:

P_E = pull in pounds (kg) to elongate the film 1 % from the first point in the stress-strain curve where the slope remains constant,

T = thickness of test specimen, in. (mm), and

W = width of test specimen, in. (mm).

12.2 Examine the uniformity of the tensile strength and elongation results obtained for the ten specimens measured for a material. Choose one of the following procedures for discarding spurious values and calculate the mean of the remaining results:

12.2.1 Use the results from those five specimens showing the highest tensile strength in evaluating all three properties, discarding those from the remaining five. This is done on the basis that the expected errors (nicks or flaws in the specimen, breaks within the jaw, slippage in the jaw, etc.) would all tend to produce results on the low side.

12.2.2 Discard the values for those specimens where the tensile strength and elongation values are significantly lower than those for most of the specimens. Use the test for outliers given in Practice D 3980.

13. Report

13.1 Report the mean values obtained with the test specimens for:

13.1.1 Tensile strength,

13.1.2 Elongation, and

13.1.3 Stiffness (modulus of elasticity), if desired.

13.2 Report the conditions of the test.

13.2.1 Procedure for preparation of free films,

13.2.2 Rate of elongation in percent per minute,

13.2.3 Specimen size (length, width, and thickness),

13.2.4 Temperature and relative humidity during test and treatment, and

13.2.5 Aging and treatment given the specimens.

14. Precision

14.1 An interlaboratory test of this method was conducted in which three coatings (representing a brittle film, a film with limited elasticity, and a very elastic film) were tested by five laboratories. The test was conducted with an Instron tensile tester operated at two elongation rates (10 and 100 %) with a specimen gage length of 2 in. (50 mm) and specimen thicknesses of 2.5 to 3.5 mils (65 to 90 μm). The between laboratory coefficients of variation were found to be 30 % for percent elongation and 18 % for tensile strength. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Reproducibility*—Two results for percent elongation obtained by operators in different laboratories should be considered suspect if they differ by more than 118 % of their mean. Two results for tensile strength obtained by operators in different laboratories should be suspect if they differ by more than 70 % of their mean.

15. Keywords

15.1 elongation; organic coatings; stiffness; tensile properties; tensile strength

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Standard Test Method for Pigment Content of Solvent-Reducible Paints¹

This standard is issued under the fixed designation D 2371; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Section 10 was added editorially in May 1990.

1. Scope

1.1 This test method covers the procedure for the quantitative separation of the vehicle from the pigment in solvent-reducible coatings.

1.2 This test method has been proven to be applicable to the following types of paints: white linseed oil outside house paint, white soya and phthalic alkyd enamel, white linseed *o*-phthalic alkyd enamel, red lead primer, zinc chromate primer, flat white inside enamel, white epoxy enamel, white vinyl toluene modified alkyd, and white amino modified baking enamel. It is considered to be applicable to most solvent-reducible paints.

1.3 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 6.

2. Referenced Document

2.1 ASTM Standard:

D 2698 Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging²

3. Significance and Use

3.1 This test method is suitable for setting specifications for the pigment content of solvent-reducible paints as well as for monitoring manufacturing quality control.

3.2 This test method provides the isolated pigment fraction from solvent-reducible paints that may be used for pigment analysis.

4. Apparatus

4.1 *Centrifuge*, explosion-proof, capable of developing 1000 to 2000 g.

NOTE 1—The centrifuge should be equipped with a suitable head to take the proper size trunnion cups necessary for use of the 90-mL tubes or 4-oz (120-mL) bottles. A two, four, or six-place head can be used with the bottles and an eight-place head can be used with the tubes.

4.2 *Centrifuge Tube*, 90-mL, heavy-walled. In place of the

90-mL centrifuge tube a 4-oz (120-mL) screw cap bottle with vinyl-lined screw cap may be used.³

4.3 *Laboratory Oven*, vented and capable of maintaining a temperature of $105 \pm 2^\circ\text{C}$.

4.4 *Syringe*, 5-mL.

4.5 *Water Bath*.

5. Solvents

5.1 *Ethyl Ether or Petroleum Ether*: (Warning—See 6.1).

5.2 *Extraction Mixture*—Mix 10 volumes of ethyl ether, 6 volumes of benzene or toluene, 4 volumes of methyl alcohol, and 1 volume of acetone (Warning—See 6.2, 6.3, and 6.4).

6. Hazards

6.1 *Ethyl Ether and Petroleum Ether*—Flammable. Vapor is harmful. May be fatal if inhaled or swallowed. Use only with adequate ventilation. Avoid prolonged contact with skin. Avoid contact with flame, hot surface, or sparks. Do not get in eyes, on skin, or on clothing. Refer to suppliers' Material Safety Data Sheet.

6.2 *Acetone*—Flammable. Vapor is harmful. May be fatal if inhaled or swallowed. Use only with adequate ventilation. Avoid prolonged contact with skin. Avoid contact with flame, hot surface, or sparks. Do not get in eyes, on skin, or on clothing. Refer to suppliers' Material Safety Data Sheet.

6.3 *Methyl Alcohol*—Flammable. May be fatal or cause blindness if swallowed. Cannot be made nonpoisonous. Harmful if inhaled. Keep away from heat, sparks, or open flame. Avoid breathing vapor. Use only with adequate ventilation. Refer to suppliers' Material Safety Data Sheet.

6.4 *Benzene* has been declared carcinogenic.

6.4.1 *Benzene and Toluene*—Flammable. Vapors and liquid are harmful and may be fatal if swallowed. Keep away from heat, sparks, or open flame. Keep in a well-closed container. Avoid breathing vapor. Avoid contact with eyes, skin, or clothing. Refer to suppliers' Material Safety Data Sheet.

7. Procedure

7.1 Mix the sample until it is homogeneous, preferably on a mechanical shaker.

7.2 Weigh to 1 mg from a syringe, 5 to 10 g of sample into a tared 90-mL centrifuge tube with a screw-type glass stirring rod (the rod is tared with the centrifuge tube), or a glass bottle as described in 4.2. The weight of the specimen is the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

Current edition approved May 31, 1985. Published July 1985. Originally published as D 2371 - 65 T. Last previous edition D 2371 - 73 (1979)^{e1}.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Bottles and caps that have been successfully used may be obtained from the Sargent Welch Scientific, 7300 N. Linder Ave., Skokie, IL 60076 and are designated as follows: Cat. No. S9185C, 4-oz bottles.

weight of syringe and paint minus the weight of the syringe after transferring the specimen to the tube or bottle.

7.3 Add 60 mL of extraction mixture (5.2) and stir well with the glass, screw-type stirring rod, preferably using a power stirrer. Wash the rod clean with a stream of extraction mixture into the tube, and reserve the rod. If the glass bottle is used, mixing may be accomplished by hand, shaking vigorously, or by use of a mechanical shaker.

7.4 Centrifuge at 1000 to 2000 *g* (Note 2) until the pigment is clearly separated. A faster speed may cause the pigment to pack too hard for future mixing. (See Test Method D 2698.)

NOTE 2—Calculate the gravity developed by the centrifuge as follows:

$$g = 1.118 \times 10^{-6} \times r \times n^2 \quad (1)$$

where:

r = rotating radius, mm, and

n = rotating speed, r/min.

7.5 Discard the upper layer by decantation.

7.6 Repeat the extraction twice as described in 7.3, 7.4, and 7.5, making certain that the settled pigment is sufficiently stirred or shaken so that no lumps can be seen sticking to the side of the centrifuge tube or bottle and all pigment can easily be washed from the stirring rod into the tube. It may be necessary to break up the pigment cake before stirring. The bottle and contents may be mixed by tapping at an angle on a cloth pad on a bench top to break up the pigment cake and then shaking vigorously to complete the pigment dispersion in the extraction mixture.

7.7 Make a fourth extraction using ethyl ether or petro-

leum ether following the same precautions noted in 7.6. Decant the upper layer. Drive off the ether by heating (gently at first to avoid spattering) on a steam bath in a hood away from open flames or sparks. The pigment should be broken up by tapping the tube or bottle on a cloth pad on a bench top. Dry the tube or the bottle at $105 \pm 2^\circ\text{C}$ for 2 h.

7.8 Cool and weigh the tube (including stirring rod) or bottle, plus pigment.

8. Calculation

8.1 Calculate the percent of pigment, *P*, as follows:

$$P = [(W_1 - W_2)/S] \times 100 \quad (2)$$

where:

*W*₁ = weight of the tube (including stirring rod) plus pigment, g,

*W*₂ = weight of the tube (including stirring rod), g, and

S = weight of paint sample used, g.

NOTE 3—All pigment obtained using this test method has minute amounts of vehicle adsorbed on the pigment. However, this will not significantly affect the precision of this test method.

9. Precision

9.1 Data are not available to determine the precision of this test method. There are no plans at present to obtain such data. This test method has been in use for several years and is considered acceptable.

10. Keywords

10.1 high speed centrifuging; pigment content; pigment separation; solvent-reducible paint

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Standard Practice for Separation of Vehicle From Solvent-Reducible Paints¹

This standard is issued under the fixed designation D 2372; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Section 6 was added and the title was changed editorially in May 1990.

1. Scope

1.1 This practice covers the procedure for the separation of the vehicle from the pigment in solvent-reducible paint.

1.2 In the development of the practice the following material were tested: white soya and white fish oil isophthalic alkyd semi-gloss enamels, white linseed oil paint, white soya and white linseed *o*-phthalic alkyd enamels. It is considered to be applicable to similar materials.

1.3 *This standard does not purport to address the safety problem associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints²

D 2698 Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging²

3. Significance and Use

3.1 Separation of the vehicle from the pigment in solvent-

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

Current edition approved May 31, 1985. Published July 1985. Originally published as D 2372 - 65 T. Last previous edition D 2372 - 73 (1979)^{ε1}.

² Annual Book of ASTM Standards, Vol 06.01.

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reducible paints is required in order to characterize paint vehicles by chemical, spectroscopic, or chromatographic techniques. Characterization of vehicles is important since they play a major role in the performance of coatings.

4. Apparatus

4.1 *Centrifuge*, high-speed, capable of developing in excess of 10 000 g.

NOTE 1—Calculate the gravity developed by the centrifuge as follows:

$$g = 1.118 \times 10^{-6} \times r \times n^2$$

where:

r = rotating radius, mm, and

n = rotating speed, r/min.

5. Procedure

NOTE 2—If the percent of pigment is desired, see Test Method D 2371 and Test Method D 2698.

5.1 Mix the sample until it is homogeneous, preferably on a mechanical shaker.

5.2 Centrifuge about 20 mL of the whole paint on a high-speed centrifuge in excess of 10 000 g (Note 1) for 30 min or until separation is completed. If difficulty is encountered, add 10 mL of a suitable solvent (Note 3). Mix with the vehicle layer in the centrifuge tube and centrifuge as before. Pour off the supernatant liquid into a bottle and stopper under nitrogen for further use.

NOTE 3—If quantitative work must be done on the vehicle, the weight of the paint specimen and of the added solvent must be known.

6. Keywords

6.1 centrifuge; paint vehicle separation; solvent-reducible paint



Standard Test Method for Slump of Face Glazing and Bedding Compounds on Metal Sash¹

This standard is issued under the fixed designation D 2376; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers face glazing or bedding compounds, or both, used on exterior steel, aluminum, and other metal sash.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 A triangular bead of glazing or bedding compound is tooled into a steel channel and then placed in a $122 \pm 4^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$) oven for 7 h and then visually inspected for slump.

3. Significance and Use

3.1 This test method provides an accelerated means for predicting slump of such glazing compounds.

4. Apparatus

4.1 *Number 28 U.S. Gage Steel Channel* (smooth, unpainted), 1 in. (25.4 mm) wide, $\frac{1}{2}$ in. (12.7 mm) deep, and 8 in. (203 mm) in length. The flanges of the channel shall be approximately at right angles with the web of the channel. The fillet at the bottom of the channel shall have an internal radius of no more than $\frac{1}{16}$ in. (1.6 mm).

4.2 *Gravity Convective Oven*, having a temperature controlled at $122 \pm 4^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$).

4.3 *Putty Knife*.

5. Reagent

5.1 *Solvent*, such as methyl ethyl ketone or ethylene dichloride.

6. Sampling

6.1 Thoroughly mix the entire contents of a full, previ-

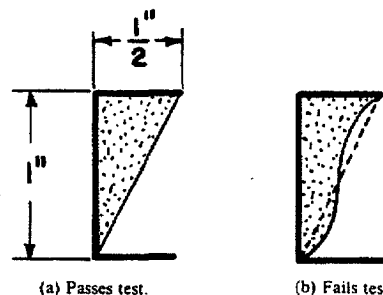


FIG. 1 Slump Test Diagram

ously unopened container on a clean, nonabsorptive surface, and take from this the sample to be tested.

7. Conditioning

7.1 Condition both the channel and the mixed compound (in a closed container) for at least 5 h at $73.4 \pm 3.6^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$).

8. Procedure

8.1 Thoroughly clean the channel with solvent.

8.2 Apply a portion of the thoroughly mixed compound to the channel in a triangular shape with a putty knife as illustrated in Fig. 1(a).

8.3 When the channel is so filled, set it in an oven with the flanges horizontal, as shown in Fig. 1(a), with the mass of the material toward the top, and maintain the temperature at $122 \pm 4^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$) for 7 h.

9. Report

9.1 The report shall indicate if there was sagging or slumping of the compound at the top towards the bottom, as illustrated in Fig. 1(b).

9.2 Any sagging or slumping from the original configuration, Fig. 1(a), shall be construed as failure.

10. Precision and Bias

10.1 No statement is made about the precision or bias for measuring slump since the result merely states whether there is conformance to the criteria for success in the procedure.

¹ This test method is under the jurisdiction of ASTM Committee C-24 on Building Seals and Sealants and is the direct responsibility of Subcommittee C24.12 on Oil and Resin Base Glazing and Caulking Compounds.

Current edition approved Nov. 30, 1984. Published January 1985. Originally published as D 2376 - 65 T. Last previous edition D 2376 - 74 (1979).

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Standard Practice for Determining the Effect of Overbaking on Organic Coatings¹

This standard is issued under the fixed designation D 2454; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the determination of the time-temperature effect of overbaking on the physical and chemical properties of organic coatings.

1.2 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings²
- D 523 Test Method for Specular Gloss²
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- D 1540 Practice for Effect of Chemical Agents on Organic Finishes Used in the Transportation Industry²
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature²
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴
- D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting⁴
- D 2092 Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting²

D 2197 Test Method for Adhesion of Organic Coatings by Scrape Adhesion²

D 2201 Test Method for Preparation of Hot-Dipped Nonpassivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates²

D 2794 Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)²

D 3359 Test Methods for Measuring Adhesion by Tape Test²

D 3363 Test Method for Film Hardness by Pencil Test²

E 805 Practice for Identification of Instrumental Methods of Color or Color-Difference, Measurement of Materials³

3. Terminology

3.1 Definition:

3.1.1 *overbaking*—an exposure of the coating to a moderately higher temperature or to a longer period of baking, or both, than recommended by the manufacturer of the coating for normal curing. This condition is in contrast to "heat resistance" which is a parameter relating to the service life of a coating.

4. Summary of Practice

4.1 Four panels are prepared and baked at the schedule normally recommended for the coating. Two of the panels are then removed and the remaining two are subjected to an additional overbake in which the time and temperature are mutually agreed upon between the purchaser and the seller. The sets of panels, after a suitable conditioning interval, are then evaluated for the properties that are compatible with the substrate. Among these are gloss, color, flexibility, adhesion, impact resistance, and resistance to reagents. Note that glass substrates should not be tested for impact, and zinc-coated substrates can influence both flexibility and impact.

5. Significance and Use

5.1 Most coatings are designed for a specific baking time and temperature. For a variety of reasons (line stoppages, rerouting back through ovens, oven overheating, etc.) the prescribed time or temperature, or both, of the bake is often exceeded. This practice has been found to be useful in evaluating the effects of overbakes on coatings.

6. Materials

6.1 *Standard Baking-Type Coating* mutually agreed upon between the purchaser and the seller.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Feb. 22, 1991. Published April 1991. Originally published as D 2454 - 66 T. Last previous edition D 2454 - 84a.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

7. Hazards

7.1 The flash points of most solvents used in many organic coatings and related products are low enough that adequate ventilation is needed to avoid exceeding 25 % of the lower explosive limits of the solvents when test panels are being prepared and baked. As these materials are considered toxic, take care to avoid inhalation of solvent vapor and unnecessary contact of solvent with the skin.

8. Procedure

8.1 Application of Organic Coating:

8.1.1 Apply coatings to steel panels prepared in accordance with Methods D 609.

8.1.2 Apply coatings to zinc-coated surfaces prepared in accordance with Practices D 2092, when zinc has been applied by the hot-dip method or by electroplating.

8.1.3 Apply coatings to nonpassivated galvanized steel prepared in accordance with Test Method D 2201, when the zinc is applied by a continuous galvanizing method using an aluminum-bearing zinc.

8.1.4 Apply coatings to aluminum surfaces on all-aluminum materials prepared in accordance with Practices D 1730.

8.1.5 Apply coatings to hot-dip aluminum coated surfaces prepared in accordance with Practices D 1731.

8.1.6 In cases where a primer is used in practice under the topcoat, apply the entire system to the panels. The film thickness of each coat shall be mutually agreed upon between the purchaser and seller, but, in the absence of such agreement, the total thickness shall be 2 ± 0.2 mils (50 ± 5 μm).

8.1.7 In instances where clear coatings are to be tested, they may be applied to aluminum or white carrara glass, one side of which has been polished to a smooth, high-gloss finish. Other selected substrata previously agreed upon between the purchaser and the seller may also be used. In the absence of a specific agreement between the purchaser and the seller in regard to film thickness, apply the coatings by automatic spray. (Refer to Test Methods D 823, Method A) at a dry film thickness of 2 ± 0.2 mils (50 ± 5 μm). Multiple coats may be used to obtain this film thickness where necessary due to the nature of the coating material (Note 1). Manual spray application or the drawdown method with wire-wound draw bars may be used when automatic equipment is not available.

NOTE 1—Dry film thickness should be measured in accordance with Test Methods D 1005, D 1186, or D 1400, whichever is applicable. The adhesion, flexibility, and color can vary considerably with thickness.

8.2 Baking:

8.2.1 The baking schedule for each coating including primer, topcoat, and primer/topcoat systems shall be mutually agreed upon between the purchaser and the seller and shall include the following:

8.2.1.1 *Normal Baking Schedule (Time and Temperature)* recommended for the development of optimum film properties, and

8.2.1.2 *Overbaking Cycle*—This cycle shall be within practical limits in order to simulate conditions that might be encountered in actual production where baking oven or conveyor lines, or both might malfunction temporarily due to mechanical or electrical failure.

8.3 Prepare four panels of each coating or coating system and bake them at the schedule normally recommended to obtain optimum properties. Conduct the baking of these panels in a mechanical recirculating air oven set to $\pm 2^\circ\text{F}$ ($\pm 1^\circ\text{C}$) of the specified baking temperature. At the end of the specified time remove two panels from the oven and subject the remaining two to the overbake cycle by:

8.3.1 A continuation of the normal bake but for a previously agreed upon time, for example, 50 or 100 % increase in time, or

8.3.2 Resetting the oven temperature to the agreed overbake temperature and then continuing the bake for a specified period of time after the overbake temperature has been reached, or removing the panels until the overbake temperature is reached and then baking them for the agreed upon time.

NOTE 2—The conditions described in 8.3.2 are not usually reproducible between laboratories or different ovens because of variations in the heating characteristics of ovens.

8.3.3 After the sets of panels have been baked according to the mutually agreed upon baking cycles, place them in a suitable rack and condition for 24 h at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and 50 ± 5 % relative humidity (see Test Methods D 1640) prior to testing.

9. Test Methods

9.1 Determine the properties of both sets of panels in accordance with the following ASTM methods:

9.1.1 *Gloss*—Test Method D 523.

9.1.2 *Color*—Practice D 1729 (Visual) or Practice E 805 (instrumental), or both

9.1.3 *Flexibility*—Test Method D 522 (conical mandrel and cylindrical mandrel).

9.1.3.1 Heat-aged flexibility values should be determined in accordance with a baking cycle that is in agreement between the purchaser and the seller.

9.1.4 *Adhesion*—Test Methods D 2197 (mechanical) or D 3359 (tape), or both.

9.1.5 *Impact Resistance*—Test Method D 2794.⁵

9.1.6 *Hardness*—Test Method D 3363 (pencil test).

9.1.7 *Exposure to Reagents*—If this test is desired, the choice of materials to which the coating is to be exposed shall be governed by the ultimate use of the coating and shall be agreed upon between the purchaser and the seller. The general procedure to be followed in this test is given in Test Method D 1308 or Practice D 1540.

10. Report

10.1 Report the following information:

10.1.1 Substrate employed, including type and thickness,

10.1.2 Type of coating,

10.1.3 Film thickness, including primer, if any,

10.1.4 Normal bake schedule, or schedules,

10.1.5 Overbake cycle,

10.1.6 Test methods employed, and

10.1.7 Values determined, including the measured changes between the normal and overbake cycles.

⁵ It is suggested that impact resistance be determined by use of the Gardner Variable Impact Tester obtainable from the Gardner Laboratory, Inc., Bethesda, MD.

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Standard Test Methods for Evaluating Coatings For High Temperature Service¹

This standard is issued under the fixed designation D 2485; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the evaluation of the heat-resistant properties of coatings designed to protect steel surfaces exposed to elevated temperatures during their service life. Two test methods are described as follows:

Method A—Interior Service Coatings

Method B—Exterior Service Coatings

1.2 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 5.

2. Referenced Documents

2.1 ASTM Standards:

A 36/A 36M Specification for Structural Steel²

A 283/A 283M Specification for Low and Intermediate Tensile Strength Carbon Steel Plates²

A 285/A 285M Specification for Pressure Vessel Plates, Carbon Steel, Low- and Intermediate-Tensile Strength²

B 117 Test Method of Salt Spray (Fog) Testing³

D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings⁴

D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁴

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁴

D 1014 Test Method for Conducting Exterior Exposure Tests of Paints on Steel⁴

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁴

D 2200 Pictorial Surface Preparation Standards for Painting Steel Surfaces⁴

G 7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials⁵

3. Summary of Test Methods

3.1 Panels suitably coated with the material under test are

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved May 15, 1991. Published July 1991. Originally published as D 2485 - 66 T. Last previous edition D 2485 - 84.

² Annual Book of ASTM Standards, Vol 01.04.

³ Annual Book of ASTM Standards, Vols 03.02 and 06.01.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 14.02.

evaluated under one or both of two test methods depending on the intended usage.

3.1.1 *Test Method A, Interior Service Coatings*—Coated panels are heated for 24 h in a muffle furnace at a temperature agreed upon between the purchaser and the seller. One panel is plunged into water and the other cooled and then subjected to a bend test.

3.1.2 *Test Method B, Exterior Service Coatings*—Coated panels are subjected to temperatures that increase in steps from 400 to 800°F (205 to 425°C). One panel is subjected to salt spray for 24 h, and one is exposed outdoors for 12 months.

3.2 When tests are completed, the panels are examined for evidence of film degradation including rust formation, blistering, loss of adhesion, dulling, and chalking.

4. Significance and Use

4.1 Some coating systems are developed for use over steel that is exposed to high temperatures during service life. This method provides an accelerated means of determining the performance of these coating systems. Testing of coatings designed for interior service and of coatings designed for exterior (weather-exposed) service is included.

5. Apparatus

5.1 Automatic spray equipment as described in Test Methods D 823 (Method A) or any other suitable method that will give the required uniformity of film and film thickness.

5.2 Muffle furnace capable of maintaining the temperatures described in 7.2 and 7.3.

5.2.1 *Precaution:* Due to the high temperatures involved in operating the muffle furnace, extreme caution should be used.

5.3 Salt spray apparatus as described in Method B 117.

6. Panel Preparation

6.1 *Type*—Panel dimensions shall be established by prior agreement between the purchaser and the seller. Use panels of 1/4 in. (6.4 mm) hot-rolled steel conforming to Specification A 36/A 36M or Specification A 283/A 283M, or when appropriate, Specification A 285/A 285M for the water quench test in Test Method A and for all tests under Test Method B. For the bend test in Test Method A, the panels must be thin and soft enough to be bent as described in Test Methods D 552.

6.2 *Cleaning*—Clean the panels for both test methods in accordance with Methods D 609, Methods B or D. Then, using a fine silica sand (graded to pass through a No. 40 (425- μ m) sieve), blast the panels for Method B to "white" metal (removing all mill scale and rust) so that the panels

comply with grade A SP-5 of Standard D 2200. Other blast media and blast methods are acceptable if agreed upon between the buyer and the seller. Blast-cleaned panels cannot be subjected to the bend test because they are too thick to bend.

6.3 Coatings Applications—Apply the coatings by automatic spray, Method A of Test Methods D 823, or by any other suitable method that will assure the required uniformity of film thickness. The number of coats, technique, and cycle for drying between coats of multicoat systems, limits of dry film thickness, and drying time before exposure shall be established by prior agreement between the purchaser and the seller. Determine dry film thickness in accordance with Test Methods D 1186. Allow coatings that air dry at ambient temperature to dry a 168 h at a temperature of $75 \pm 5^\circ\text{F}$ ($24 \pm 2.5^\circ\text{C}$) prior to exposing to test conditions. Bake coatings that require heat conversion in accordance with the recommendation of the supplier.

7. Procedure

7.1 Sampling—When panels have been coated at another location, select at random from different packages two specimens for each method of each type of coating under investigation.

7.2 Test Method A:

7.2.1 Place one panel each of the two types described in 6.1 and 6.2 coated with each paint under test in a muffle furnace maintained at the test temperature mutually agreed upon between the purchaser and the seller, and allow to remain for 24 h. At the end of this time plunge one of the panels immediately into water maintained at $70 \pm 5^\circ\text{F}$ ($21 \pm 2.5^\circ\text{C}$). After removing from the water, examine the coating film for evidence of film failure including dulling, blistering, cracking, and loss of adhesion. Allow the second panel to cool at a room temperature of $75 \pm 5^\circ\text{F}$ ($24 \pm 2.5^\circ\text{C}$) for 1 h, then rapidly bend double over a $\frac{1}{2}$ -in. (12.7-mm) diameter steel mandrel with coated side uppermost in accordance with Test Methods D 522, Method B. Examine this panel for evidence of film degradation such as cracking and loss of adhesion.

7.3 Test Method B:

7.3.1 Expose duplicate coated panels finished with the materials under test to the elevated temperature test schedule mutually agreed upon between the purchaser and the seller. In the absence of such a specified schedule use the following:

7.3.2 Place the panels in a muffle furnace maintained at 400°F (205°C) for 8 h, then increase the temperature to 500°F (260°C) for 16 h. Increase the temperature in 100°F (55°C) increments, alternating the time periods indicated, to the final temperature maximum previously agreed to between the purchaser and the seller. Thus the schedule would be:

400°F (205°C) for 8 h
500°F (260°C) for 16 h
600°F (315°C) for 8 h
700°F (370°C) for 16 h
800°F (425°C) for 8 h

NOTE—Many high temperature paints depend upon service conditions to provide the extreme heat required to cure or sinter their films. If these paints are never exposed to the extreme heat, they may be found to be unsuitable. Consequently, it is good practice to make observations also at temperatures less than the expected service temperatures.

7.3.3 Remove the test panels and make a visual inspection following exposure at each temperature level for evidence of failure, including peeling, cracking, blistering, abnormal discoloration, or loss of adhesion, using ASTM standards where possible. Disregard such failures up to $\frac{1}{4}$ in. (6.4 mm) in from the edges of the test panels.

7.3.4 At the end of the complete exposure, remove the panels from the oven or muffle furnace and allow to air cool at ambient temperature for a minimum period of 1 h. As described in 7.3.3, inspect for evidence of failure due to the high temperature exposure.

7.3.5 If both panels of each pair pass the heat test satisfactorily, use them, respectively, for salt spray (fog) testing and exterior exposure testing.

7.3.5.1 Place one panel of each pair in a salt spray cabinet operated in accordance with the requirements of Method B 117, for a period of 24 h. At the conclusion of the exposure period, examine each panel for rusting or any evidence of corrosive attack.

7.3.5.2 Expose the second panel of each pair on an insulated-type rack, as described in Practice G 7, or as agreed upon between the purchaser and seller, for a period of 12 months. Make a visual inspection after 6 months and after termination of the exposure. The site to conduct the test and the angle of the exposure rack should be agreed upon between the purchaser and the seller and should relate to the end-use application of the coating.

8. Report

8.1 Report the following information:

8.1.1 Description of substrates, substrate preparation, and application conditions used.

8.1.2 Air drying versus baking schedule used.

8.1.3 Details of outdoor exposure when applicable, including:

8.1.3.1 Exposure site, type (insulated rack, black box, open rack, etc.) and angle of exposure,

8.1.3.2 Date of exposure start,

8.1.3.3 Radiation data in terms of kilojoule per square metre or langley's during exposure period, and

8.1.4 Any defects or changes as described under Section 7.

9. Precision

9.1 Four operators in four laboratories tested six materials covering a range of heat-resistant properties and were able to rank products in order of their heat-resisting qualities. Since rankings were of a qualitative nature, no mathematical statement of reproducibility was established.

10. Keywords

10.1 heating tests, paints; resistance, heat; temperature tests, elevated

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Standard Test Method for Scrub Resistance of Interior Latex Flat Wall Paints¹

This standard is issued under the fixed designation D 2486; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers an accelerated procedure for determining the resistance of latex flat wall paints to erosion caused by scrubbing.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D4213 Test Method for Wet Abrasion Resistance of Interior Paints²

3. Summary of Test Method

3.1 The test paint is applied to a black plastic panel. After aging, the coated panel is placed over a 1/2-in. by 10-mil (12.7 by 0.25-mm) shim and held in place on a glass plate in a washability machine by means of a gasketed frame. It is then scrubbed with a nylon bristle brush and an abrasive scrub medium until failure occurs over the shim.

4. Significance and Use

4.1 Interior wall paints often become soiled especially near doorways, windows, and in work and play areas. This test method covers determination of the relative resistance of different wall paints to erosion when repeatedly scrubbed to remove the stains during the life of the paint.

4.2 Results developed by use of this test method do not necessarily represent the scrub resistance of aged paint films.

4.3 This test method measures scrub resistance by the traditional scrub-to-failure concept. Test Method D 4213 is a more precise method that measures essentially the same property by a weight-loss technique and reports it as a volumetric film erosion rate. Test Method D 2486 is maintained as an ASTM standard along with the newer Test Method D 4213 because it has served the paint industry usefully for many years, its techniques are familiar, and its results are meaningful. To discard it as a standard would be to minimize a great deal of significant past experience.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved March 6, 1989. Published May 1989. Originally published as D 2486 - 66 T. Last previous edition D 2486 - 79.

² Annual Book of ASTM Standards, Vol 06.01.

5. Apparatus

5.1 Washability Machine.³

5.1.1 Accessory Apparatus: (Fig. 1).

5.1.1.1 Nylon Bristle Brush, Rubber Mat, and Holder (total weight 454 g).

5.1.1.2 Glass Plate, measured to fit.

5.1.1.3 Shim, 1/2-in. by 10-mils (12.7- by 0.25-mm).

5.1.1.4 Gasketed Frame and Clamps.

5.2 Film Caster,⁴ having 7-mil (0.18-mm) clearance.

6. Reagents and Materials

6.1 Black Plastic Panels.⁵

6.2 Masking Tape.

6.3 Flannel Cloth.

6.4 Detergent.⁶

6.5 Scrub Medium⁷ (Note 1), consisting of the following:

	grams
Water, distilled or deionized	49.7 ⁴
Hydroxyethyl cellulose ⁸	1.0
Ammonium hydroxide, 28 %	0.1
Detergent ⁶	2.0
Trisodium phosphate, anhydrous	2.0
Silica ⁹	45.0
Acetic acid (glacial)	0.2 ^B
Preservative ¹⁰	0.1
	100.0

⁴ Adjust to achieve viscosity of 110 to 120 Krebs Units before use.

^B Vary to achieve a pH of 9.5 to 10.0.

NOTE 1—When a referee test is made, prepare fresh medium or use standardized scrub medium⁷ from a previously unopened container that is no more than 1 year old.

6.5.1 Slowly add the hydroxyethyl cellulose to the water while stirring mechanically. Stir until uniform, then continue stirring for an additional 5 min. Then slowly add 3 drops of

³ The Gardner washability machine Model M-105A and accessory apparatus, available from the Gardner Laboratories, Inc., 5521 Landy Lane, Bethesda, MD 20014, has been found satisfactory for this purpose. Other straight-line wash testers may be adapted to meet the requirements of this test method.

⁴ The Dow film caster, available from the Gardner Laboratories, Inc., has been found satisfactory for this purpose.

⁵ Leneta P-121-10N dull black plastic panels 6 1/2 by 17 in. by 10 mils (165 by 432 by 0.25 mm) in size (production tolerance: ± 1 mil), obtainable from the Leneta Co., P.O. Box 576, Ho-Ho-Kus, NJ, are suitable for the purpose.

⁶ Octyl phenoxy polyethoxy ethanol has been found satisfactory. Triton X-100 obtainable from Rohm & Haas Co., Independence Sq., Philadelphia, PA, is representative of this product.

⁷ Laboratory standardized scrub medium is available from the Leneta Co.

⁸ Cellosize QP-4400, obtainable from Union Carbide Corp., Chemicals Div., 270 Park Ave., New York, NY, has been found satisfactory for this purpose.

⁹ Silica No. 22, obtainable from Whittaker, Clark, & Daniels, Inc., 100 Church St., New York, NY 10007, has been found satisfactory for this purpose.

¹⁰ 1,3,5-triethyl hexahydro-sym-triazine (Vancide TH) obtainable from R. T. Vanderbilt Co., 230 Park Ave., New York, NY 10017, has been found satisfactory.

28 % ammonium hydroxide while mixing, and continue mixing until solution turns clear. In the order given, add the other ingredients separately, employing mechanical stirring. Be sure that each item is uniformly dispersed before adding the next one. The addition of the silica should take about 5 min to ensure uniform dispersion. Finally, add the preservative and adjust the pH with glacial acetic acid.

7. Preparation of Apparatus

7.1 Washability Machine—The washability machine should be leveled before use and operated at 37 ± 1 counts per minute. Before each test check the tightness of the motor belt or chain drive, alignment of the pulleys and cables in both horizontal and vertical planes, and tautness of the cable.

7.2 Brush—The bristles, if new, must be leveled before use to permit uniform wear on the paint surface. Leveling is accomplished by running the brush over 100 or 120-mesh aluminum oxide close grain sandpaper or emery cloth attached lengthwise on the glass plate in the washing machine. The total number of cycles required will vary from 1000 to 5000. Additional weight, up to 454 g, may be used to expedite the procedure. Change the sandpaper or emery cloth if it becomes clogged. Tape down the edges if any tearing is observed. Replace brush when bristles have worn to extend less than $\frac{5}{8}$ in. (16 mm) from block.

7.3 Brush Holder—Screws on the brush holder shall be removed and not used. Instead, insert a $\frac{1}{8}$ -in. (3.2-mm) thick rubber mat in the holder above the brush block. When in operation, this mat allows the brush to ride evenly on the paint surface without tilting (Fig. 1). The brush must fit loosely in the holder.

8. Procedure

8.1 Clean the top of the glass plate (or preferably suction plate) and both sides of the black plastic panel to be sure they are free of specks. Place the black panel on the plate and tape one end to the plate. Smooth the panel along the plate by rubbing with flannel, creating static electricity which improves adhesion to the plate.

8.2 Stir the test paint thoroughly and strain to remove all skins and particles. Draw down the paint on the panel using the 7.0-mil (0.18-mm) side of the film caster, starting from the taped end of the panel. The time for application should be fairly slow—3 to 4 s from end to end—to prevent formation of pinholes or holidays in the film. Air dry in a horizontal position for 7 days in an open room kept at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and 50 ± 5 % relative humidity.

8.3 Clean the plate and set it in the pan of the washability machine. Be sure that the shim is smooth and free of burrs and placed across the center of the plate. Wipe the test panel carefully and place it on top of the shimmed plate with the painted side up. Be sure that there are no defects in the film above the shim and that the test area is level. Prewet the gasket of the frame and wipe it dry before use. Place the frame over one half of the drawdown, being sure that the entire gasket is on the painted surface. (The other half can be used for a second test if desired.) Clamp the gasket in place. The clamps should be tight enough to ensure close contact, but not tight enough to cause warping of the panel.

8.4 Soak the brush in a 1 % solution of detergent⁶

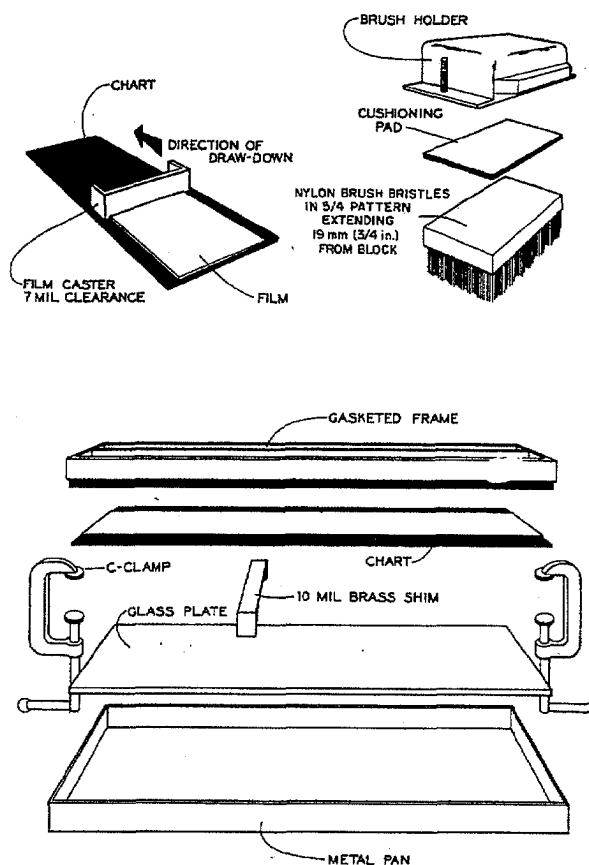


FIG. 1 Accessory Apparatus for Scrub Test

overnight. Remove the brush and shake it vigorously to remove any excess. Mount the brush in the holder being sure that the mat is in place above the brush block and that the brush is always in the same position. (Mark both brush and holder beforehand.) Stir the scrub medium and spread 10 g evenly on the brush bristles. Place the brush at one end of the path and attach the guide cables. Wet the panel with 5 mL of reagent water in the path of the brush.

8.5 Set the cycle counter at zero. Start the test. After each 400 cycles before failure remove the brush (do not rinse); add 10 g of stirred scrub medium; replace the brush, being sure that brush and holder are in the same position with relation to each other and in the machine. Place 5 mL of water on the path before continuing.

8.6 Record the number of cycles to remove the paint film fully in one continuous line across the $\frac{1}{2}$ -in. (12.7-mm) width of the shim. Stop the machine and wipe off the area to determine the end point.

8.7 Make three drawdowns of each specimen. Test two and average them if they are within 25 % repeatability. If they are not, test a third and average the three, unless one of the results is obviously discrepant, in which case it should be discarded. According to the 90 % confidence limits specified herein, the running of a third test will be required, on the average, only one out of ten times for normal results.

9. Report

- 9.1 Report the mean number of cycles to failure (see Section 10).
- 9.2 Outline any deviations from standard procedure.

10. Precision

- 10.1 The following criteria should be used for judging the acceptability of results of scrub resistance tests at a 90 % confidence level.

10.1.1 *Repeatability*—Duplicate results by a single operator should be considered suspect if they differ by more than 25 % of their mean value.

10.1.2 *Reproducibility*—Two results (each the mean of duplicate measurements) obtained by operators in different laboratories should be considered suspect if they differ by more than 50 % of their mean.

11. Keywords

- 11.1 Scrub resistance; abrasion resistance

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Standard Guide for Testing Wood Furniture Lacquers¹

This standard is issued under the fixed designation D 2571; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This Guide covers the evaluation of gloss or flatted, unpigmented lacquers designed for use on wood substrates. This Guide is to be used in conjunction with Test Methods D 333. Also included are several methods of special relevance to the application of lacquer on wood. The selection of the tests to be used for any given product or system must be governed by experience and by the requirements agreed upon by the producer and user.

1.2 The tests on films apply to those films applied in sufficient quantity to form a continuous film. It is recommended that reports include the thickness of the film under test.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry-Cleaning Solvent)²
- D 333 Test Methods for Clear and Pigmented Lacquers³
- D 1211 Test Method for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood³
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes³
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)⁴
- D 2091 Test Method for Print Resistance of Lacquers³
- D 2199 Method for Measurement of Plasticizer Migration from Vinyl Fabrics to Lacquers³
- D 3359 Test Methods for Measuring Adhesion by Tape Test³
- D 3459 Test Method for Humid-Dry Cycling for Coatings on Wood and Wood Products³
- G 23 Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials⁵

¹ This Guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Performed Products.

Current edition approved March 25, 1988. Published September 1988. Originally published as D 2571 - 67. Last previous edition D 2571 - 81(1987).

² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.01, 06.02 and 06.03.

⁵ Annual Book of ASTM Standards, Vols 14.02 and 06.03.

3. Significance and Use

3.1 This Guide is intended to compile as well as provide screening tests in evaluating wood furniture coatings both high gloss and flatted type, as used by the furniture industry.

3.2 Each wood finishing system may vary from a simple two-step operation to a complex operation consisting of as many as 15 steps.

3.3 The substrate is a complex and varied substrate that does absorb moisture from the surrounding environment. Variation in the moisture content of the wood before and after coating and during the testing affects the test results. Grease, dirt and foreign matter also affect the test results.

3.4 Results from these various tests are not necessarily useful in evaluating performance of all different types of furniture finishing systems.

4. Test Panels and Panel Preparation

4.1 Test panels should be regular production finish panels.

4.2 If regular production finish panels are not available, the producer and the user should agree on the substrate to be used and on the complete finishing system.

5. Nonvolatile Matter

5.1 Test for nonvolatile matter in accordance with Test Methods D 333.

6. Self-Lifting Properties

6.1 Apply a second coat after the first top-coat has been applied and air dried for 1, 6, and 24 h. Report any tendency of self-lifting.

7. Color

7.1 Test color of the liquid lacquer in accordance with Test Method D 1544.

8. Rubbing Properties

8.1 In the absence of a specified procedure, air dry the lacquer 18 h, sand with No. 320 W/D sandpaper wetted with mineral spirits, and then rub with 3-F pumice and paraffin oil (white mineral oil, U.S.P. heavy, or other suitable rubbing compound).

8.2 Examine the rubbed surface immediately and after 24 h, or the time agreed upon by the producer and the user, in a

standard atmosphere, for example, $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and 50 % relative humidity, and report any difference in appearance.

9. Print Test

9.1 Test for resistance to imprinting in accordance with Test Method D 2091.

10. Cold Check Test

10.1 Test for resistance to checking and cracking in accordance with Test Method D 1211 on a substrate agreed upon by the producer and the user. Report any difference in appearance.

11. Resistance to Oils, Greases, Cosmetics, and Other Household Chemicals

11.1 Test for resistance to the agents in 11.2 to 11.5. For other household chemicals test in accordance with Test Method D 1308.

11.2 *Cosmetic Stain*—Apply to the finished surface a heavy smear of lipstick of indelible type and place the panel in a 125°F (50°C) oven overnight. Remove the lipstick with mineral spirits meeting the requirements of Specification D 235, and evaluate.

11.3 *Alcohol Resistance*—Place 0.5 mL of 50 % ethanol (by weight) in water on the lacquer film and trap with a 2-in. (50-mm) watch glass. Pure, undiluted ethyl alcohol or denatured alcohol conforming to Formula 2-B of the U.S. Bureau of Internal Revenue are considered equivalent for this test. One hundred proof vodka may be used in place of 50 % ethanol. After at least 6 h, remove the watch glass and allow the alcohol to evaporate. Report whitening or spotting that cannot be removed with light polishing using a dry cotton pad.

11.4 *Boiling Water Resistance*—Pour 25 mL of boiling distilled or deionized water on the leveled panel and allow to cool to room temperature. Dry and examine. Report graying, spotting, softening, or other film deterioration.

11.5 *Coffee Stain Resistance*—Prepare coffee by each of the following methods:

11.5.1 Dissolve 1 teaspoon (5 cm^3) of instant coffee, 1 teaspoon of sugar, and 1 teaspoon of powdered cream substitute in 8 fl oz (240 mL) of at least 180°F distilled or deionized water.

11.5.2 Dissolve 1 teaspoon (5 cm^3) of instant coffee and sufficient synthetic sweetener (dry or liquid) to supply 5 mg of saccharin in 8 fl oz (240 mL) of at least 180°F distilled or deionized water.

11.5.3 Place 1 mL of each type of hot coffee on the panel and cover with 2-in. (50-mm) watch glasses. After 2 h, remove the watch glasses, and allow to evaporate. Wash the panel with a rag moistened with water. Report how readily the residue is removed.

12. Light Exposure

12.1 Test for the effect of light exposure in accordance with all methods except 1, 2, and 3 of Practice G 23, but operated without water spray. Report method used and results.

13. Tape Marring

13.1 Press a strip of cellophane tape, on the test panel, rolling it down with a 70 to 80 durometer hardness rubber roller. Allow the tape to remain on the panel for 30 min. Slowly remove the tape. After 30 min recovery, report any visible marring.

14. Tape Adhesion

14.1 Test for tape adhesion in accordance with Test Methods D 3359.

15. Resistance to Plasticizer Migration

15.1 Test and rate plasticizer migration in accordance with Test Method D 2199.

16. Accelerating Aging by Humid-Dry Cycling

16.1 Evaluate and report the accelerated aging in accordance with Test Method D 3459.

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Standard Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms¹

This standard is issued under the fixed designation D 2574; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the resistance of emulsion paints against attack in the container by microorganisms.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 Spoilage of paint in the container can result in putrefaction, lowered pH, gas formation, and decrease in viscosity. This test method provides a standard procedure for the evaluation of the resistance of emulsion paints to microbial deterioration. The results should enable: (1) the paint manufacturer to select an effective preservative and (2) the supplier of preservatives to evaluate the performance in emulsion paints of competitive and developmental preservatives.

2.2 This test method should preferably be used by persons who have had basic microbiological training.

3. Apparatus

- 3.1 Refrigerator, maintained at 10 to 13°C.
- 3.2 Balance, accurate to 0.1 g.
- 3.3 Swabs, sterile cotton.
- 3.4 Screwcap Borosilicate Test Tubes, 125 by 15-mm.
- 3.5 Borosilicate Flasks, 1-L.
- 3.6 Screwcap Bottles, 150-mL.
- 3.7 Autoclave, capable of producing 15 psi (103 kPa) of steam pressure at 120°C and maintaining it for a minimum of 15 min. An autoclave is not necessary if prepared agar slants are used.

4. Materials

- 4.1 Culture of *Pseudomonas aeruginosa*.²
- 4.2 1 U.S. qt (945 mL) of Paint Under Test.
- 4.3 1 U.S. qt (945 mL) of paint identical to 4.2, but containing no microbiocide.

4.4 Dehydrated Tryptone-Glucose Extract Agar.³

4.5 Beef Extract.^{4,5}

4.6 Tryptone.^{4,5}

4.7 Glucose.^{4,5}

5. Preparation of Materials

NOTE—Observe conventional microbiological techniques in making these tests. Handle all materials so as to avoid contamination from the air, fingers, or work surfaces.

5.1 Preparation of Tryptone-Glucose Extract Agar Slants:

5.1.1 Add 12 g of tryptone-glucose extract agar to 500 mL of cold distilled water in a 1-L flask. Heat to boiling with frequent agitation to dissolve the medium completely.

5.1.2 Distribute 10 mL of the dissolved medium into each of 50 test tubes.

5.1.3 Autoclave tubes (with caps loose) for 15 min at 15 psi (103 kPa) and a temperature of 120°C.

5.1.4 Upon removal from the autoclave, tighten caps and place the tubes at an approximate 30° angle position to cool, making a slope about 2 in. (50 mm) long.

5.1.5 Store agar slants in a refrigerator at 10 to 13°C until needed.

5.2 Preparation of Tryptone-Glucose Extract Broth:

5.2.1 Add 1.5 g of beef extract, 2.5 g of tryptone, and 0.5 g of glucose to 500 mL of cold distilled water in a 1000-mL flask. Heat to boiling to dissolve the medium completely.

5.2.2 Distribute 10 mL of the dissolved medium into each of 50 test tubes.

5.2.3 Autoclave tubes (with caps loose) for 15 min at 15 psi (103 kPa) and a temperature of 120°C.

5.2.4 Upon removal from the autoclave, allow the tubes to cool to room temperature, tighten the caps, and store until needed.

5.3 Inoculation of Tryptone-Glucose Extract Broth With *Pseudomonas Aeruginosa*:

5.3.1 To an agar culture of *Pseudomonas aeruginosa*, add 3 mL of the tryptone-glucose extract broth prepared in 5.2. Using a sterile cotton swab, remove bacterial growth from the surface of the agar and mix with the broth.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.28 on Biodeterioration.

Current edition approved May 30, 1986. Published July 1986. Originally published as D 2574 - 67 T. Last previous edition D 2574 - 73 (1980).

² Suitable culture may be obtained from the American Type Culture Collection, Washington, DC.

³ Tubed tryptone-glucose extract agar slants available from Difco Laboratories, Inc., Detroit, MI 48201, Baltimore Biological Laboratories, Baltimore, MD 21218, or equivalent are suitable for use by those who do not have the necessary equipment for preparation of their own media.

⁴ Available from Difco Laboratories, Inc., Detroit, MI 48201, Baltimore Biological Laboratories, Baltimore, MD 21218, or equivalent.

⁵ Tryptone-glucose extract broth may be prepared by dissolving a commercially produced tablet in water. Suitable tablets may be obtained from Consolidated Laboratories, Chicago Heights, IL.

5.3.2 Remove the cotton swab soaked with broth and place in a tube of sterile tryptone-glucose extract broth. Transfer the inoculated broth to the sterile broth by pressing the swab against the inside wall of the tube.

5.3.3 Remove the cotton swab and incubate the broth culture at 27 to 28°C for 24 h.

5.3.4 Soak a second sterile cotton swab in the broth added to the culture of *Pseudomonas aeruginosa* described in 5.3.1. Remove the cotton swab soaked with broth and transfer the inoculated broth to the surface of a tryptone-glucose extract agar slant prepared in 5.1 by pressing the swab gently on top of the agar surface. Incubate slant at 27 to 28°C for 24 h or until luxuriant colony growth is observed on the agar surface. Label as culture of *Pseudomonas aeruginosa* ATTC No. 10145 and store in a refrigerator at 10 to 13°C until 5.3.1 is repeated for a future test.

5.3.5 Soak a sterile cotton swab in the inoculated broth culture following the incubation period described in 5.3.3.

5.3.6 Remove the swab and prepare a second broth culture by repeating 5.3.2 and 5.3.3.

5.3.7 Following the incubation period, use the broth culture prepared in 5.3.6 to proceed as described in Section 6.

6. Procedure

6.1 Preparation of Spoiled Paint:

6.1.1 Transfer the well-shaken broth culture prepared in 5.3.6 to the 1-qt (945-mL) sample of paint (see 4.3). Shake the inoculated paint well before proceeding.

6.1.2 Incubate the paint at 27 to 28°C for 1 week.

6.1.3 To determine whether or not the paint contains bacteria at the end of the incubation period, use a sterile cotton swab to transfer a specimen of paint to the surface of a tryptone-glucose agar slant. Incubate agar slant at 27 to 28°C for 72 h. If colonies of *Pseudomonas aeruginosa* appear on the agar surface, paint is satisfactory for use as spoiled paint in 6.4.1. If colonial growth of *Pseudomonas aeruginosa* does not appear on the surface, repeat 6.1.1 and 6.1.2.

6.2 Preliminary Examination of Paint Under Test:

6.2.1 Examine the can for evidence of swelling. If the can is swollen, exercise caution in removing the lid.

6.2.2 Remove the lid and smell the contents of the can. Deterioration of paint by microorganisms is often characterized by distinct odors. Such odors may be either putrefactive or fermentative.

6.2.3 Observe the contents of the can for the presence of stringy structures characteristic of the presence of certain microorganisms.

6.2.4 Observe the contents for noticeable losses in viscosity. This physical change frequently occurs as the result of microbiological deterioration.

6.3 Determination of Presence or Absence of Living Microorganisms in Paint Under Test:

6.3.1 Soak a sterile cotton swab in the paint under test. Remove excess paint by pressing gently against inside of container.

6.3.2 Transfer the test paint to the surface of a tryptone-glucose agar slant by pressing the swab gently on the agar surface.

6.3.3 In order to obtain duplicate agar slants, repeat 6.3.1 and 6.3.2.

6.3.4 Incubate the inoculated agar slants at 27 to 28°C for a minimum of 1 week.

6.3.5 If colonial growth of bacteria is observed on the agar surface at the end of the incubation period, the test is complete and may be reported in accordance with Section 7.

6.3.6 If colonial growth of bacteria is not observed on the agar surface at the end of the incubation period, continue the test as described in 6.4.

6.4 Challenging Test Paint by Inoculation:

6.4.1 To 100 g of the paint under test, add 1 g of paint spoiled in 7.1.

6.4.2 Incubate the paint for 6 weeks at 27 to 28°C.

6.4.3 At the end of 24, 48, 72 h and 1 week of incubation and once each succeeding week throughout the incubation period, soak a sterile cotton swab in the paint. Remove excess paint by pressing against the side of the container and transfer the paint to the surface of a tryptone-glucose agar slant.

6.4.4 Incubate the agar slant at 27 to 28°C for 1 week and observe the presence or absence of colonial growth of bacteria.

6.4.5 At the end of 6 weeks add an additional 1 g of paint spoiled in 6.1. Incubate for 2 weeks at 27 to 28°C and repeat 6.4.3 and 6.4.4.

7. Report

7.1 Report the following information:

7.1.1 Results of observations noted under 6.2.

7.1.2 Presence or absence of living microorganisms in the paint as received, and after it has been inoculated (or reinoculated) with a sample of spoiled paint.

7.1.3 If living microorganisms are found in the paint as received, or after inoculation (or reinoculation), the paint shall be reported as "not resistant in the container to attack by microorganisms."

7.1.4 If living organisms are not found, the paint shall be reported as "resistant in the container to attack by microorganisms."

8. Precision and Bias

8.1 No statement is made about either the precision or bias of this test method for measuring the resistance of emulsion paints in the container to attack by microorganisms, since the result merely states observations of the paint under test and presence or absence of living microorganisms in the paint as received or after inoculation.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Light Stability of Clear Coatings¹

This standard is issued under the fixed designation D 2620; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the discoloration of clear coatings by sunlight through glass.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D1729 Practice for Visual Evaluation of Color Differences of Opaque Materials²

D1925 Test Method for Yellowness Index of Plastics³

G24 Practice for Conducting Natural Light Exposures Under Glass²

3. Summary of Method

3.1 The effect of sunlight on discoloration of clear coatings is determined by exposing under glass, coated panels masked at one end, to natural sunlight for a specified time. At the end of the exposure, the mask is removed and the panels evaluated for degree of discoloration.

4. Significance and Use

4.1 Color change, particularly yellowing, is not generally acceptable to users of coatings when it is readily noticeable. In designing clear coating systems for interior use on various substrates, it is of interest to know how well the clear coats will resist color change caused by ambient light. This test method permits the evaluation of the effect of natural indoor light on the color stability of clear coatings.

5. Apparatus

5.1 *Exposure Cabinet* as described in Practice G 24.

6. Test Specimens

6.1 For each clear coating under test, use one test panel approximately 3 by 5 in. (75 by 125 mm) of white carrara glass or other specified substrate material.

NOTE 1—Other materials that may be considered for this purpose include: (1) White ceramic tile. (2) A white pigmented, light-stable

coating which would not be lifted by the clear topcoat. (A baking enamel with the composition of 30 % melamine formaldehyde resin and 70 % of the phthalic anhydride/coconut alkyd resin with 3 lb/gal (1.36 kg/L) of titanium dioxide per gallon has been found satisfactory.) (3) The substrate intended for use with the clear coating. The light stability of the substrate may be determined by including an extra test panel without a clear topcoat.

6.2 Prepare specimens of the test materials by applying the coatings on the test panels to a dry film thickness of 2 ± 0.2 mil (50 ± 5 μ m).

6.3 Prepare a reference specimen, representing a material of known performance in this test, with each set of test specimens.

6.4 Record the panel preparation details: method of coating application, baking conditions, film thickness, etc. These details will be established by mutual agreement between purchaser and seller in accordance with the type of product and its intended use.

7. Conditioning

7.1 Unless otherwise specified, condition the coated panels at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and 50 ± 5 % relative humidity for 48 h if baked, or 7 days if air dried.

8. Procedure

8.1 Prior to exposure, mask one-half of each test panel with aluminum foil to exclude sunlight from the other half of the coating.

NOTE 2—The tape used to attach the mask to the panel should not extend over the edge of the mask but should extend beyond the ends of the mask and stick to the rear of the panel, thereby holding the mask in position. This will prevent contact between the tape and the finish and eliminate the possibility of staining from the tape.

8.2 Mount the test specimens in the glass-covered exposure cabinet continuously 24 h a day for the specified period of exposure. It is recommended this period should be approximately 5 weeks, since longer periods of exposure do not significantly change the results.

NOTE 3—In cooperative tests, exposure of nitrocellulose lacquers, cellulose acetate butyrate lacquer, and low-bake alkyd enamel for 22 weeks did not show significantly greater discoloration than after exposure for 5 weeks.

9. Interpretation of Results

9.1 Visual ratings of the degree of discoloration may be made. However, it is preferred that an instrumental method be used as described in Test Method D 1925, (see Test Method D 1729).

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 2620 - 67. Last previous edition D 2620 - 68 (1981)⁴.

² *Annual Book of ASTM Standards*, Vol 14.02.

³ *Annual Book of ASTM Standards*, Vol 08.02.

10. Color Changes Not Caused by Light

10.1 In many cases atmospheric conditions (temperature, humidity, reactive gases) may produce significant color changes even without the presence of light. An indication as to whether or not side effects are operating may be obtained by retaining unexposed a duplicate set of specimens in the laboratory. A difference in color between the unexposed specimens and the covered portion of the exposed specimens indicates that the material has been affected by some agent other than light.

10.2 Further clarification regarding atmospheric effects may be obtained by simultaneously exposing a duplicate set of test specimens in another cabinet of the same type used in the light exposures but with the glass covered with an opaque material so that the light is excluded.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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11. Report

11.1 Report the following information:

- 11.1.1 Visual ratings or instrumental values for degree of discoloration of coatings or both,
- 11.1.2 Date and location of exposure,
- 11.1.3 Length of exposure,
- 11.1.4 Substrate used,
- 11.1.5 Details of panel preparation, and
- 11.1.6 Conditioning of panels prior to testing.

12. Precision

12.1 Because of the many variations in visual rating of discoloration, meaningful estimates of precision cannot be given. In cooperative tests, however, fairly good agreement was obtained by the various cooperators in the visual rating of the test panels.



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Standard Test Method for Infrared Identification of Vehicle Solids From Solvent- Reducible Paints¹

This standard is issued under the fixed designation D 2621; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the qualitative characterization or identification of separated paint vehicle solids by infrared spectroscopy within the limitations of infrared spectroscopy.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1467 Guide for Testing Fatty Acids Used in Protective Coatings²
- D 1962 Test Method for Saponification Value of Drying Oils, Fatty Acids, and Polymerized Fatty Acids²
- D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints³
- E 131 Definitions of Terms and Symbols Relating to Molecular Spectroscopy⁴
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers⁴

3. Definitions

3.1 For definitions of terms of symbols, refer to Definitions E 131.

4. Summary of Test Method

4.1 Infrared spectra are prepared from dried films of isolated paint vehicles. Vehicle types are identified by comparing the spectra to a collection of reference infrared spectra.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved June 26, 1987. Published August 1987. Originally published as D 2621 - 67 T. Last previous edition D 2621 - 69 (1981)⁴.

² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 14.01.

5. Significance and Use

5.1 The ability to qualitatively identify paint vehicles is useful for characterizing unknown or competitive coatings, for complaint investigations, and for in-process control.

6. Apparatus

6.1 *Spectrophotometer*—A recording double-beam infrared spectrophotometer with a wavelength range from at least 2.5 to 15 μm and a spectral resolution of at least 0.04 μm over that range. See Practice E 275.

6.2 *Demountable Cell Mount* with NaCl window.

6.3 *Vacuum Drying Oven* thermostatically controlled to operate at $60 \pm 2^\circ\text{C}$. A water aspirator vacuum source is satisfactory.

6.4 *Oven, Gravity or Forced Draft*, capable of maintaining temperature from 105 to 110°C.

7. Procedure

7.1 Place the vehicle, separated from the paint in accordance with Method D 2372, on a NaCl window and spread to form a uniform film. The thickness of the film should be such that when the infrared spectrum is recorded the transmittance of the strongest band falls between 5 and 15 % (Note). Dry the film in an oven at 105 to 110°C for 15 min and cool in a desiccator. Inspect the film visually for defects such as bubbles, wrinkles, contamination, etc. If defects are present, cast another film. If easily oxidizable substances are present such as tung, oiticica, or linseed oils, the film should be dried at $60 \pm 2^\circ\text{C}$ in a vacuum oven for 1 h. If solvents of low volatility such as cyclohexanone or isophorone are present, the film may need to be dried for several hours in a 60°C vacuum oven.

NOTE—Numerous procedures and variations may be used to obtain a film on which to prepare a suitable spectrum. These include liquid mounting between two NaCl plates, transmission through free films, and reflectance from highly polished surfaces.

7.2 Immediately record the infrared spectrum from 2.5 to 15 μm so that a spectral resolution of 0.04 μm is maintained throughout that range (methods for achieving this resolution will vary according to the directions of the manufacturer of the instrument used).

7.3 Compare the spectrum obtained with reference spectra prepared from nonvolatile vehicles of known composition (see Annex A1) or consult other published spectra available in the literature (Annex A3). Interpret the spectrum on the basis of available information, recognizing certain limitations of infrared spectroscopy, and qualifying the interpretation accordingly (Annex A2).

TABLE 1 Correlation of Absorption Bands in Alkyd Spectra

Wavelength, μm	Wavenumbers, cm^{-1}	Group Vibration
2.9	3448	O—H stretch
3.4 to 3.5	2941 to 2857	alkane C—H stretch
5.8	1724	ester, C=O stretch
6.2, 6.3, 6.6, 6.7	1613, 1587, 1515, 1493	skeletal in-plane aromatic C=C
6.9, 7.3	1449, 1369	aliphatic C—H bending
7.5 to 9.4	1333 to 1063	ester, C—O—C stretch (o-phthalate ester)
8.6	1163	ester, C—O—C stretch (fatty acid ester)
9.6, 13.5, 14.3	1042, 741, 699	out-of-plane aromatic C—H bending denoting o-disubstituted benzene ring.

ANNEXES

(Mandatory Information)

A1. INFRARED SPECTRA OF NONVOLATILE VEHICLES OF KNOWN COMPOSITION

A1.1 A set of reference infrared spectra on grating and prism is reproduced on the following pages.

A2. CONSIDERATIONS IN THE INTERPRETATION OF INFRARED SPECTRA OF NONVOLATILE VEHICLES SEPARATED FROM SOLVENT-TYPE PAINTS

INTRODUCTION

The infrared spectra of vehicles recovered from whole paint are presented in Annex A1. The aim of this compilation is to aid those using Test Method D 2621 in the practical interpretation of the spectra they obtain.

The spectra are compiled with one representative spectrum of each vehicle presented in both a prism and a grating format. In the discussion of the spectra, the general assignment refers to the first spectrum. The subsequent spectra discussion will include only those bands which aid in the identification of the particular modifications being illustrated. In addition, some practical information is provided where it is believed to be helpful to the analyst. In general, previously noted band assignments are not repeated.

The data compiled here were obtained from spectra prepared on very carefully calibrated instruments. In comparing them to spectra prepared in any given laboratory, it is expected that the wavelength values of absorption bands may differ slightly depending upon the calibration of the instrument used.

GROUP I-ALKYDS

A2.1 Spectrum 1: Ortho-Phthalic Alkyd, Medium Oil Length

A2.1.1 2.9- μm Region (3448 cm^{-1})—The 2.9- μm band in alkyds is due to the O—H stretching vibration. This is usually attributed to the unesterified hydroxyl OH on the polyhydric alcohol used in manufacturing the alkyd. This absorption is known to increase on drying of unsaturated oil modified alkyds due to oxidation of the double bonds. This absorption band can be used to determine the hydroxyl number of alkyds.

A2.1.2 3.3 to 3.6- μm Region ($3030\text{ to }2778\text{ cm}^{-1}$)—The bands in this area are all due to aromatic and aliphatic C—H stretching vibrations.

A2.1.3 5 to 6- μm Region ($2000\text{ to }1667\text{ cm}^{-1}$)—The 5.8- μm band in alkyds is due to the combined C=O stretch of the phthalate and fatty acid esters. Unreacted phthalic anhydride, if present, may be detected by the appearance of a sharp absorption band at approximately 5.6 μm (1786 cm^{-1}). Free carboxyl groups (due to unreacted fatty acid or

incompletely reacted phthalic acid) may often be detected by the appearance of a shoulder on the high wavelength (low frequency) side of the ester carbonyl band.

A2.1.4 6.2 to 6.4- μm Region ($1613\text{ to }1563\text{ cm}^{-1}$)—The doublet appearing in this region of the spectrum is due to vibrations associated with the double bonds in an aromatic ring. The band shape and position of this doublet is characteristic of non-oil modified, o-phthalic alkyds.

A2.1.5 6.8 to 6.9- μm Region ($1470\text{ to }1449\text{ cm}^{-1}$)—This absorption is produced by C—H bending vibrations of methylene (scissoring deformation) and methyl (asymmetrical deformation) groups in the alkyd. The intensity of this absorption band will vary with oil length.

A2.1.6 7.2 to 7.3- μm Region ($1389\text{ to }1370\text{ cm}^{-1}$)—This absorption band is due to the C—CH₃ symmetrical deformation vibration, and is produced by the methyl groups on the fatty acid chains.

A2.1.7 7.5 to 10.0- μm Region ($1333\text{ to }1000\text{ cm}^{-1}$)—The absorption bands in this region are due to the C—O—C

stretching vibrations of the phthalate ester. These absorptions are most strongly influenced by the acid portion of the ester rather than the alcoholic portion.

A2.1.8 13.5 and 14.2- μm Regions (741 and 704 cm^{-1})—These two bands are due to out-of-plane bending vibrations of ring hydrogens of aromatic compounds having four adjacent hydrogens (orthodisubstitution).

A2.1.9 Comments:

A2.1.9.1 Note that in oil-modified alkyds, the intensity of the absorption at 8.6 μm (1163 cm^{-1}) is indicative of the amount of oil modification or oil length of the alkyd. In unmodified alkyds, this band may be little more than a shoulder on the 8.9- μm (1124- cm^{-1}) C—O—C absorption. The correlation to oil length is only a very general one in that within a given group of alkyds one may say a sample is a "short," "medium," or "long" oil type.

A2.1.9.2 Alkyd spectra generally reveal little or no information concerning the type of combined oil or polyol present.

A2.1.9.3 Identification of polyol and unsaponifiables may usually be accomplished by infrared examination of saponification fractions. Identification of the oil acids used usually requires gas chromatographic analysis of the methylated fatty acids recovered by saponification. (For saponification procedures see Test Methods D 1467 and D 1962.)

A2.2 Spectrum 2: Ortho-Phthalic Alkyd, Long Oil Length

A2.2.1 8.6 μm (1163 cm^{-1}); fatty acid ester C—O—C

A2.2.2 Comments—Note the difference in the 8.6- μm (1163- cm^{-1}) peak compared to Spectrum 1, due to increased oil length.

A2.3 Spectrum 3: Ortho-Phthalic Alkyd, Tung Oil Modified

A2.3.1 10.12 μm (988 cm^{-1}); —C=C—C=C—C=C— Conjugated triene unsaturation

A2.3.2 Comments—Note the difference in band shapes in the 10 to 10.4- μm region (1000 to 962 cm^{-1}) compared to Spectra 1 and 2. Absorption due to conjugated unsaturation (in such oil types as tung, oiticica, dehydrated castor, and conjugated safflower) occurs here. Oil types used for alkyds 1 and 2 contain only isolated double bonds.

A2.4 Spectrum 4: Ortho-Isophthalic Alkyd

A2.4.1 7.8 μm (1282 cm^{-1}) isophthalate ester C—O—C

A2.4.2 8.2 μm (1220 cm^{-1}) isophthalate ester C—O—C

A2.4.3 8.9 μm (1124 cm^{-1}) isophthalate ester C—O—C

A2.4.4 13.7 μm (730 cm^{-1}) meta-disubstituted benzene ring

A2.4.5 Comments—The spectrum of this alkyd is typical of an isophthalic alkyd. The major band that identifies this as an isophthalate is the 13.7- μm (730- cm^{-1}) band. The presence of orthophthalic alkyd can be suspected by comparison to a straight isophthalic alkyd spectrum (see following) and noting the influence of the ortho-phthalate at 7.9 μm (1266 cm^{-1}), 9.0 μm (1111 cm^{-1}), 9.4 μm (1064 cm^{-1}), and at 14.2 μm (704 cm^{-1}).

A2.5 Spectrum 5: Ortho-Phthalic Alkyd, Benzoic Acid Modified

A2.5.1 14.0 to 14.1 μm (714 to 709 cm^{-1}); aromatic ring vibration where ring contains five adjacent hydrogens. Posi-

tion is characteristic of benzoate esters.

A2.5.2 Comments—The band at approximately 14.0 μm (714 cm^{-1}) is the identifying peak for this modification. Because of the *o*-disubstitution peak at 14.3 μm (699 cm^{-1}) present in *o*-phthalates, it is difficult to observe this band when the benzoic acid modification drops below 3 %.

A2.6 Spectrum 6: Ortho-Phthalic Alkyd, Para-Tertiary Butyl Benzoic Acid Modified

A2.6.1 8.4 μm (1190 cm^{-1}) C—O—C *p*-tert. butyl benzoate

A2.6.2 9.6 μm (1042 cm^{-1}) C—O—C *p*-tert. butyl benzoate

A2.6.3 9.8 μm (1020 cm^{-1}) C—O—C *p*-tert. butyl benzoate

A2.6.4 11.7 μm (855 cm^{-1}) aromatic ring substitution patterns

A2.6.5 12.9 μm (775 cm^{-1}) aromatic ring substitution patterns

A2.6.6 Comments—The characteristic bands for the identification of the paratertiary butyl benzoic acid modification are the 11.7- μm (855- cm^{-1}) and the 12.9- μm (775- cm^{-1}) bands. The other absorption bands, although sharp and distinctive, can tend to be lost in the background of the spectrum when the modification drops below 2 to 3 %.

A2.7 Spectrum 7: Ortho-Phthalic Alkyd, Tall Oil, Rosin Modified

A2.7.1 12.3 μm (813 cm^{-1}) abietic acid ring vibration

A2.7.2 Comments—The curve shows only a very slight depression at 12.3 μm (813 cm^{-1}). In general, the band is never very intense and, if suspected, the presence of rosin is readily confirmed by a Lieberman-Storch spot test. Note also the obscured nature of the 6.3 to 6.5- μm (1587 to 1538- cm^{-1}) region. This is most likely due to the salt or "soap" formation with the acids present in the system and the pigment used.

A2.8 Spectrum 8: Ortho-Phthalic Alkyd, *p*-Phenyl Phenol Modified

A2.8.1 11.4 μm (877 cm^{-1}) associated with substituted aromatic rings

A2.8.2 12.1 μm (826 cm^{-1}) associated with substituted aromatic rings

A2.8.3 13.1 μm (763 cm^{-1}) associated with substituted aromatic rings

A2.8.4 14.4 μm (694 cm^{-1}) associated with substituted aromatic rings

A2.8.5 Comments—The main identifying band is the 13.1- μm (763- cm^{-1}) band. The other bands are less distinctive, especially the 14.4- μm (694- cm^{-1}) area. It is always best to consider the positions of the 3 or 4 absorptions in the far end of the curve as a group in assigning the modifying structure.

A2.9 Spectrum 9: Ortho-Phthalic Alkyd, Styrene Modified

A2.9.1 6.7 μm (1493 cm^{-1}) aromatic ring vibration

A2.9.2 13.2 μm (758 cm^{-1}) monosubstituted aromatic (5 adjacent ring hydrogens)

A2.9.3 14.3 μm (699 cm^{-1}) monosubstituted aromatic (5 adjacent ring hydrogens)

A2.9.4 Comments—The very general forebroadening in the 13 to 13.3- μm (769 to 758- cm^{-1}) area of the ortho substitution band is characteristic of styrene modification. The 14.3- μm (699- cm^{-1}) absorption that obscures the normally present small 14.3- μm (699- cm^{-1}) band is the primary styrene absorption. Note also the sharp 6.7- μm (1493- cm^{-1}) peak which is associated with the presence of an aromatic.

A2.10 Spectrum 10: Ortho-Phthalic Alkyd, Vinyl Toluene Modified

A2.10.1 6.6 μm (1515 cm^{-1}) aromatic ring vibrations

A2.10.2 6.7 μm (1492 cm^{-1}) aromatic ring vibrations

A2.10.3 11.4 μm (877 cm^{-1}) meta-disubstituted aromatic

A2.10.4 12.3 μm (813 cm^{-1}) para-disubstituted aromatic

A2.10.5 12.8 μm (781 cm^{-1}) meta-disubstituted aromatic

A2.10.6 14.2 μm (704 cm^{-1}) meta-disubstituted aromatic

A2.10.7 *Comments*—The general pattern of peaks at the end of the spectrum is characteristic for vinyl-toluene modification. They arise from the meta-para mixed isomers.

A2.11 Spectrum 11: Ortho-Phthalic Alkyd, Acrylonitrile Modified

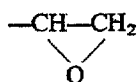
A2.11.1 4.5 μm (2222 cm^{-1}) C $\equiv$ N nitrile stretching vibration

A2.11.2 *Comment*—The 4.5- μm (2222- cm^{-1}) band is the outstanding feature characteristic of an acrylonitrile modification.

2.12 Spectrum 12: Ortho-Phthalic Alkyd, Bis-Phenol Epoxy Modified

A2.12.1 8.4 μm (1190 cm^{-1}) aromatic C—O—C

A2.12.2 10.9 μm (917 cm^{-1}) terminal epoxy grouping



A2.12.3 12.1 μm (826 cm^{-1}) para-disubstituted aromatic.

A2.12.4 *Comments*—The band at 10.9 μm (917 cm^{-1}) is due to the weakly absorbing terminal epoxy group. The 12.1- μm (826- cm^{-1}) absorption is due to the bis-phenol backbone of the epoxy polymer. The band at 8.4 μm (1190 cm^{-1}) is also always present in conjunction with the 12.1- μm (826- cm^{-1}) band.

A2.13 Spectrum 13: Ortho-Phthalic Alkyd, Urea-Formaldehyde Modified

A2.13.1 3.1 μm (3226 cm^{-1}) N—H stretching vibration.

A2.13.2 6.1 μm (1639 cm^{-1}) amide linkage band

A2.13.3 6.6 μm (1515 cm^{-1}) amide linkage band

A2.13.4 9.3 μm (1075 cm^{-1}) C—O—C ether

A2.13.5 13.0 μm (769 cm^{-1}) unknown (but present in all urea-formaldehyde resins).

A2.13.6 *Comments*—The presence of urea-formaldehyde in an o-phthalic alkyd can always be observed in the spectrum by its influence at the wavelengths listed above. The general curve shape is somewhat depressed throughout. The 3.1- μm (3226- cm^{-1}) absorption appears as a shoulder on the O—H stretch at 3.0 μm (3333 cm^{-1}).

A2.14 Spectrum 14: Ortho-Phthalic Alkyd, Melamine-Formaldehyde Modified

A2.14.1 6.5 μm (1538 cm^{-1}) C $\equiv$ N

A2.14.2 12.3 μm (813 cm^{-1}) triazine ring vibration

A2.14.3 *Comments*—A melamine modification is always distinguishable from the urea-formaldehyde modification in that it lacks the 6.1- μm (1639- cm^{-1}) absorption and contains the 12.3- μm (813- cm^{-1}) triazine ring vibration.

A2.15 Spectrum 15: Ortho-Phthalic Alkyd, Benzoguanamine-Formaldehyde Modification

A2.15.1 6.3 μm (1587 cm^{-1}) aromatic ring vibration

A2.15.2 6.5 μm (1538 cm^{-1}) C $\equiv$ N

A2.15.3 12.1 μm (826 cm^{-1}) characteristic band for benzoguanamine derived modification

A2.15.4 12.8 μm (781 cm^{-1}) characteristic band for benzoguanamine derived modification

A2.15.5 14.2 μm (704 cm^{-1}) characteristic band for benzoguanamine derived modification

A2.15.6 *Comments*—The C $\equiv$ N band occurs at a slightly lower wavelength than in the melamine resins. The band at 12.1 μm (826 cm^{-1}) rather than at 12.3 μm (813 cm^{-1}) also helps to distinguish between the two types of triazine based resins.

A2.16 Spectrum 16: Ortho-Phthalic Alkyd, Hexamethoxymethylmelamine Modified

A2.16.1 9.3 μm (1075 cm^{-1}) C—O—C ether

A2.16.2 *Comments*—The presence of hexamethoxymethylmelamine can be observed in the spectrum of an ortho-phthalic-alkyd by its influence at 6.5 μm (1538 cm^{-1}); 6.7 μm (1493 cm^{-1}); 9.3 μm (1075 cm^{-1}); 9.9 μm (1010 cm^{-1}); 10.9 μm (917 cm^{-1}); 11.5 μm (870 cm^{-1}); and 12.3 μm (813 cm^{-1}).

A2.17 Spectrum 17: Ortho-Phthalic Alkyd, Rosin-Maleic Adduct Modified

A2.17.1 5.4 μm (1852 cm^{-1}) C=O stretching vibrations associated with anhydrides

A2.17.2 5.6 μm (1786 cm^{-1}) C=O stretching vibrations associated with anhydrides

A2.17.3 *Comments*—The most characteristic absorptions are the ones listed above which cause multiple bands in the carbonyl area. The presence of the rosin-maleic adduct can also be seen at 10.6 μm (943 cm^{-1}); 10.9 μm (917 cm^{-1}); 11.7 μm (855 cm^{-1}); and 12.2 μm (820 cm^{-1}). These bands are generally found in all rosin-maleic adducts even if slightly changed in intensities.

A2.18 Spectrum 18: Ortho-Phthalic Alkyd, Vinyl Chloride-Acetate Modified

A2.18.1 7.0 μm (1428 cm^{-1}) —CH₂—

A2.18.2 8.0 μm (1250 cm^{-1}) —CH in —CHCl—

A2.18.3 14.5 μm (690 cm^{-1}) C—Cl

A2.18.4 *Comments*—The most characteristic features of this spectrum are the 8 to 8.1- μm (1250 to 1234- cm^{-1}) absorption and the very broad band peaking at approximately 14.5 μm (690 cm^{-1}). A general depression of the entire curve is noted between 7.0 μm (1428 cm^{-1}) and 10.0 μm (1000 cm^{-1}).

A2.19 Spectrum 19: Ortho-Phthalic Alkyd, Phenyl Siloxane Modified

- A2.19.1 7.0 μm (1429 cm^{-1}) aromatic silicon bond
 A2.19.2 8.8 μm (1136 cm^{-1}) Si—O—Si
 A2.19.3 10.0 μm (1000 cm^{-1}) aromatic silicon bond
 A2.19.4 14.4 μm (694 cm^{-1}) mono-substituted aromatic
 A2.19.5 *Comments*—The general influence of the presence of silicone is seen in the depressed area from 8.8 μm (1136 cm^{-1}) to 10.0 μm (1000 cm^{-1}).

A2.20 Spectrum 20: Ortho-Phthalic Alkyd, Methyl Siloxane Modified

- A2.20.1 7.9 μm (1266 cm^{-1}) —Si—CH₃
 A2.20.2 12.5 μm (800 cm^{-1}) —(CH₃)₂—Si

A2.21 Spectrum 21: Ortho-Phthalic Alkyd, Nitrocellulose Modified

- A2.21.1 6.1 μm (1639 cm^{-1}) R—O—NO₂ stretching vibration
 A2.21.2 7.8 μm (1282 cm^{-1}) R—O—NO₂ stretching vibration
 A2.21.3 9.5 μm (1053 cm^{-1}) general C—O—C ether from the cellulose ring
 A2.21.4 11.9 μm (840 cm^{-1}) low-frequency vibrations associated with R—O—NO₂
 A2.21.5 *Comments*—The most prominent absorptions are the 6.1- μm (1639 cm^{-1}) band and the very broad 11.9- μm (840 cm^{-1}) peak. An additional reliable feature of

nitrocellulose is the ether linkage which causes a general depression in the mid-section of the spectrum.

A2.22 Spectrum 22: Ortho-Phthalic Alkyd, Urethane Modified

- A2.22.1 5.8 μm (1724 cm^{-1}) R—NH—CO—OR amide I band
 A2.22.2 6.3 μm (1587 cm^{-1}) aromatic vibration (in aromatic di-isocyanate systems)
 A2.22.3 6.5 μm (1538 cm^{-1}) amide II band, C—N
 A2.22.4 12.3 μm (813 cm^{-1}) 1,2,4 tri-substitution (in systems with toluene di-isocyanate)

A2.23 Spectrum 23: Isophthalic Alkyd, Medium Oil Length

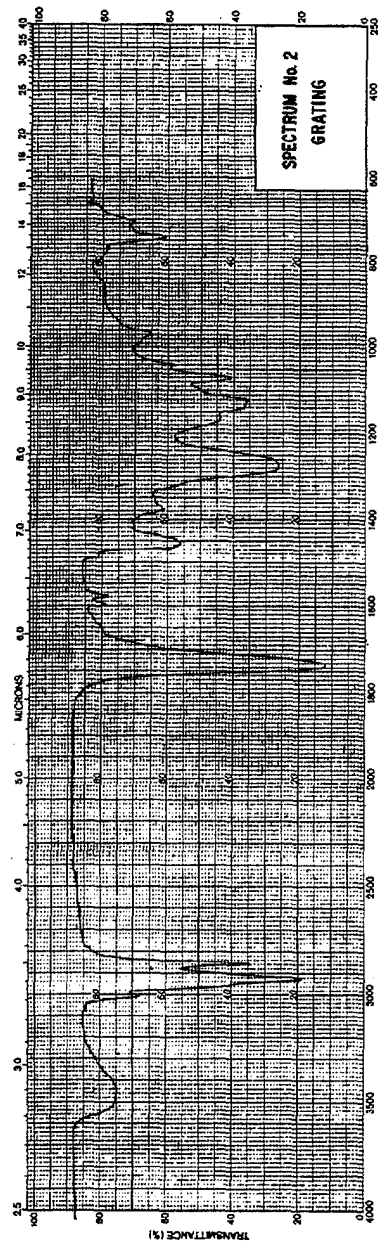
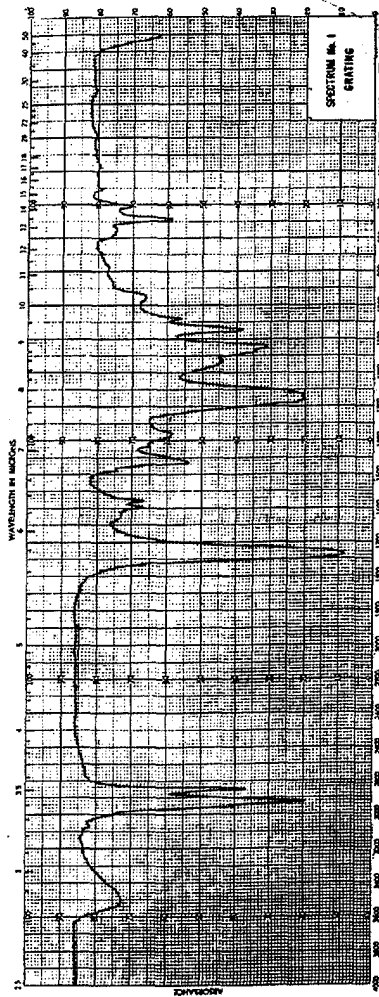
- A2.23.1 6.2 μm (1613 cm^{-1}) ring unsaturation
 A2.23.2 13.8 μm (725 cm^{-1}) meta-disubstitution band
 A2.23.3 *Comments*—Note the change from a doublet in the 6.2- μm (1613 cm^{-1}) region to the singlet. The 13.8- μm (725 cm^{-1}) band is characteristic for isophthalic alkyds.

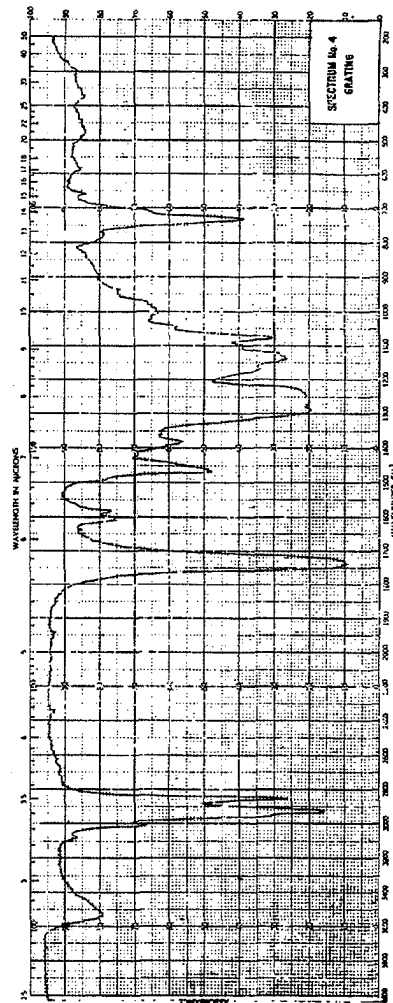
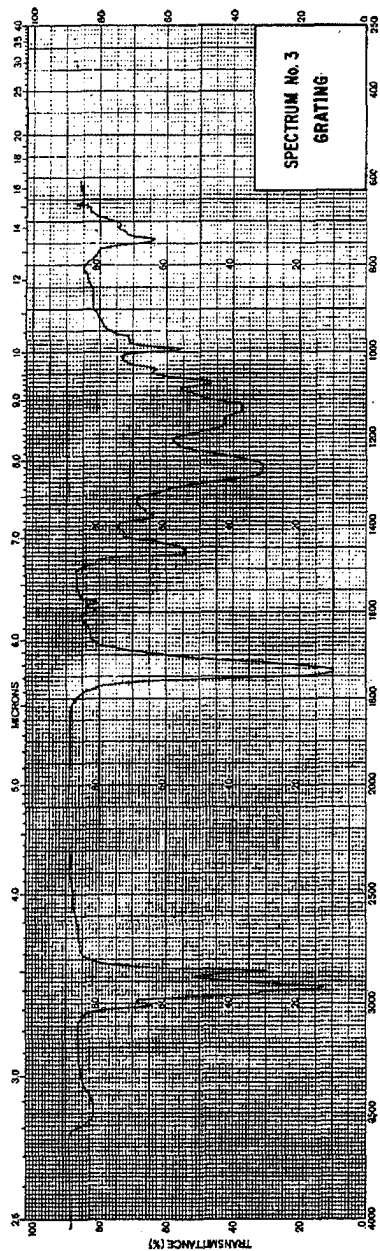
A2.24 Spectrum 24: Tere-Phthalic Alkyd, Medium Oil Length

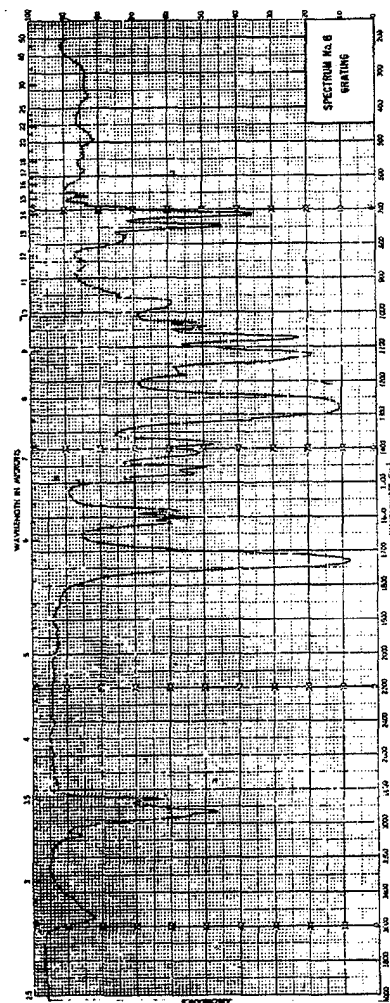
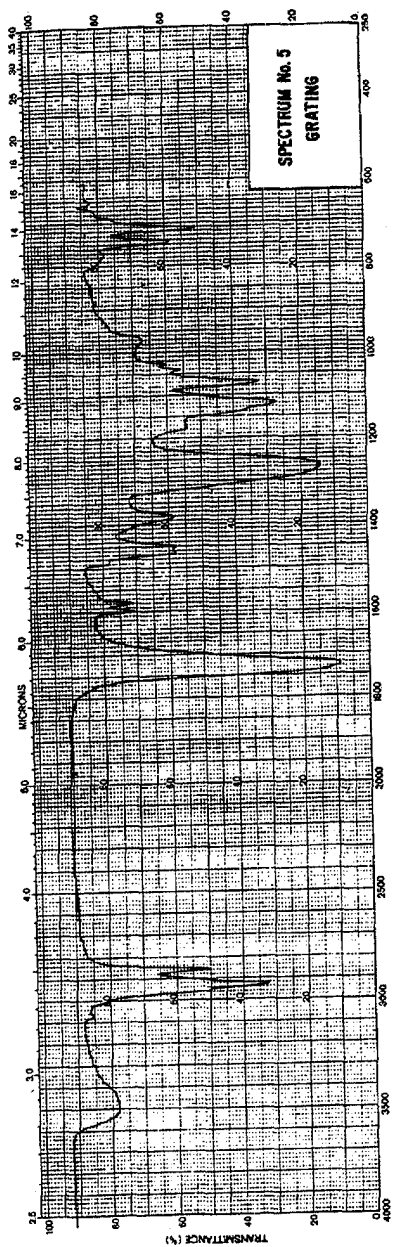
- A2.24.1 8.6 μm (1163 cm^{-1}) C—O—C
 A2.24.2 9.8 μm (1020 cm^{-1}) C—O—C
 A2.24.3 *Comments*—The tere-phthalate spectrum is identified mainly by the 8.6- μm (1163 cm^{-1}) and 9.8- μm (1020 cm^{-1}) peaks combination in conjunction with a 13.8- μm (725 cm^{-1}) peak position.

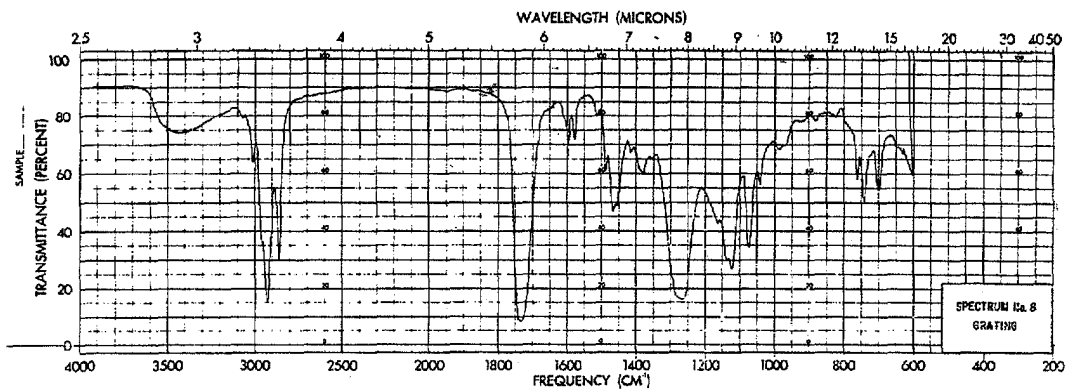
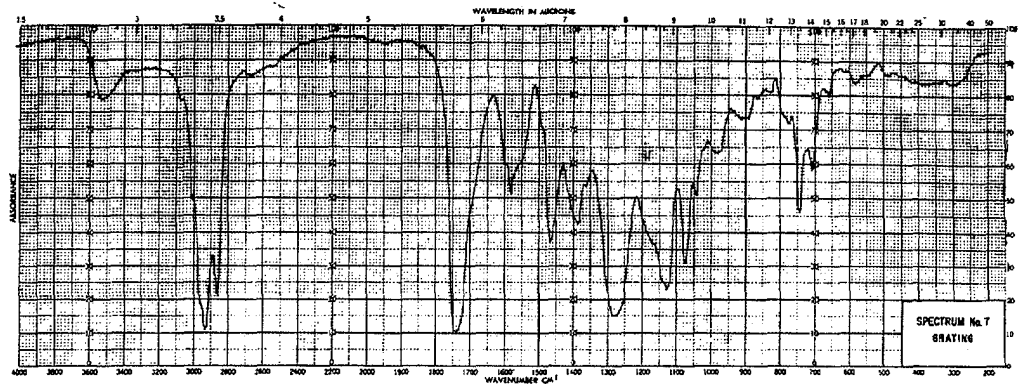
A2.25 Spectrum 25: Ortho-Phthalic Alkyd, Chlorendic Acid Modified

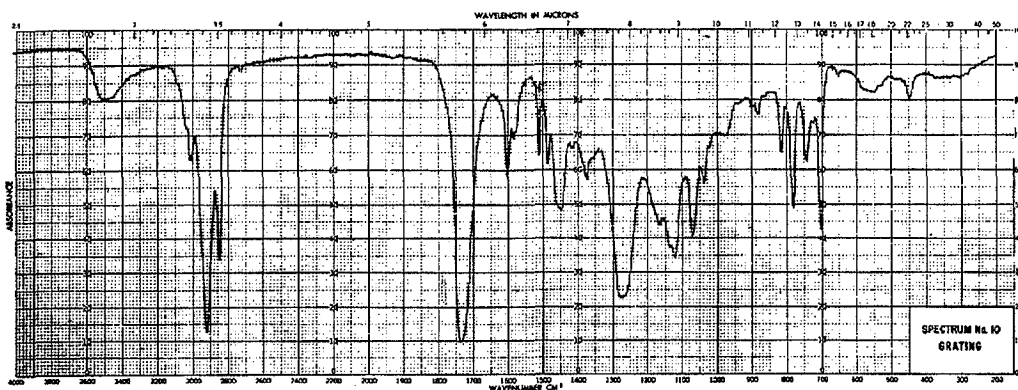
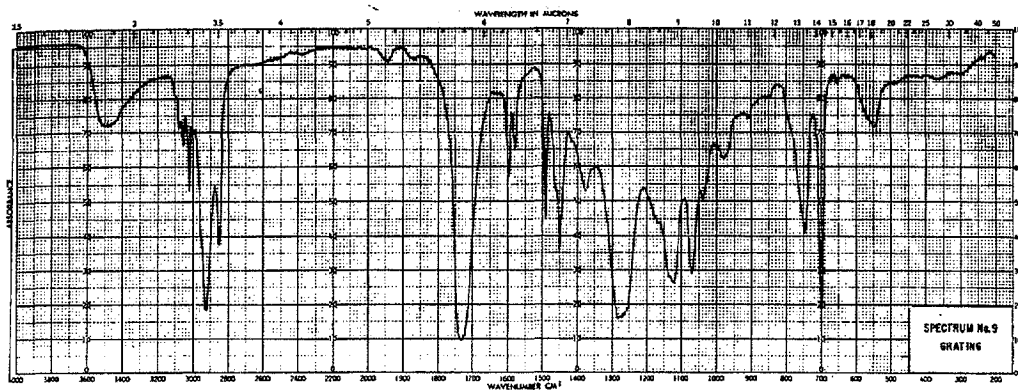
- A2.25.1 *Comments*—The overall appearance of the absorption band pattern in this spectrum is characteristic of this modification.

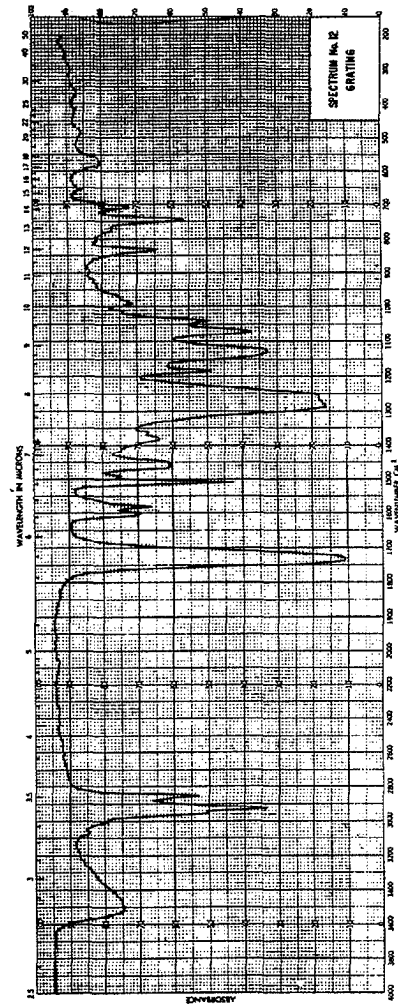
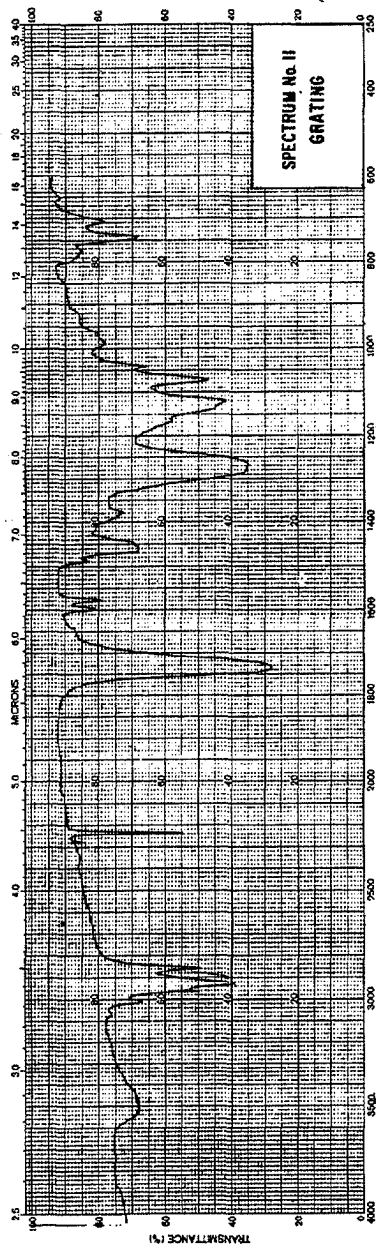


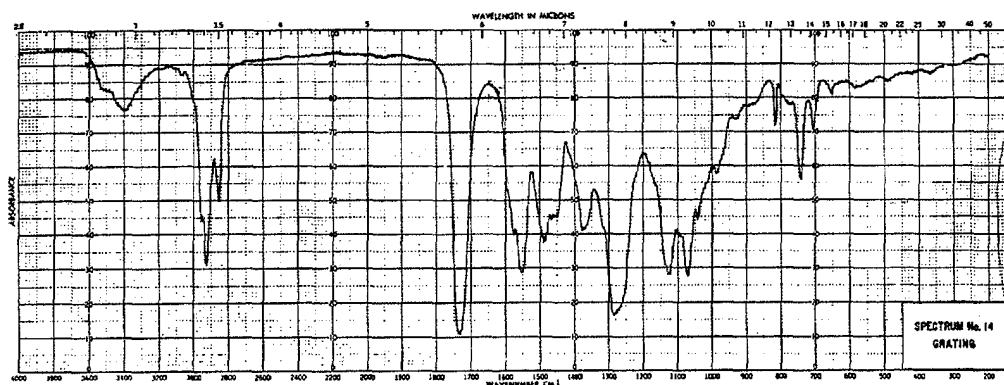
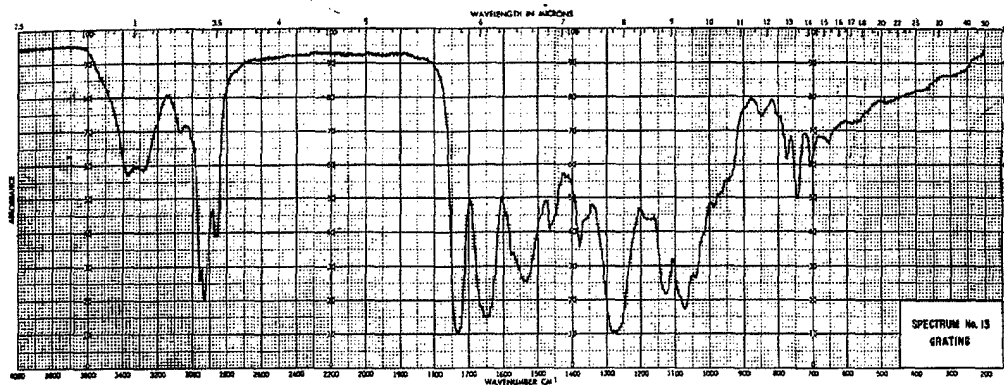


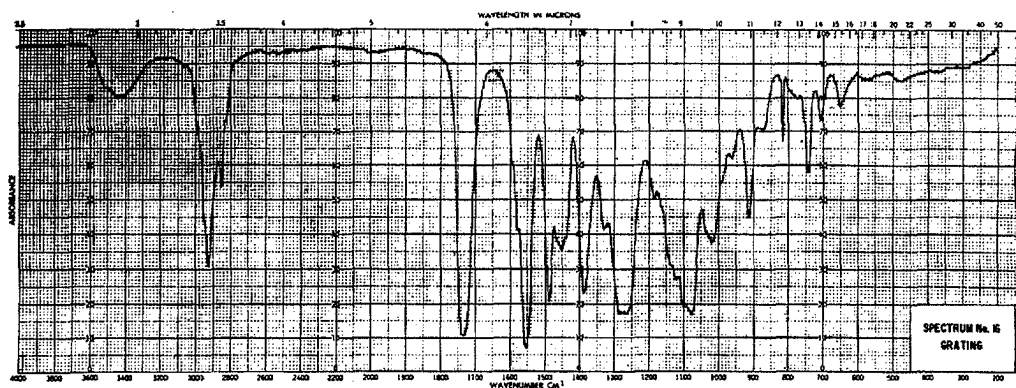
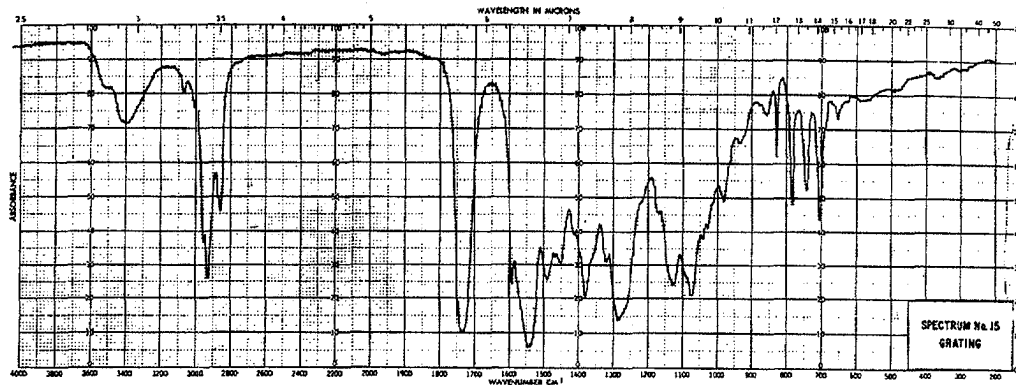


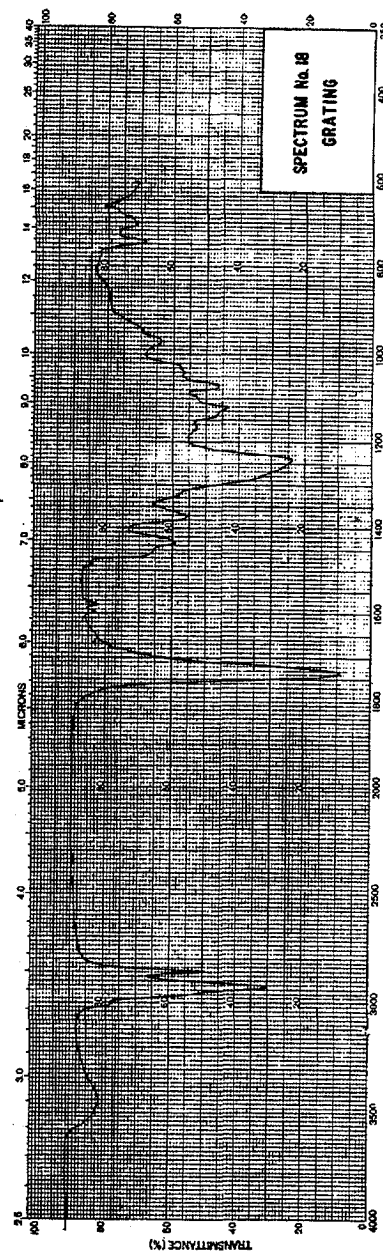
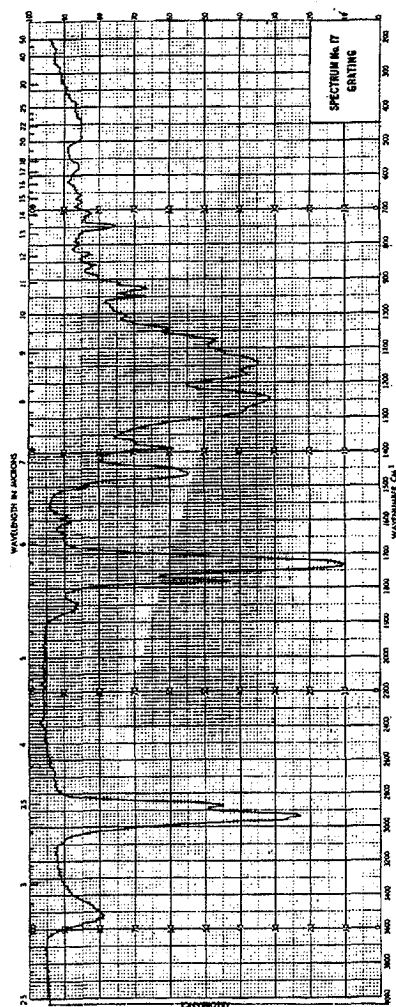


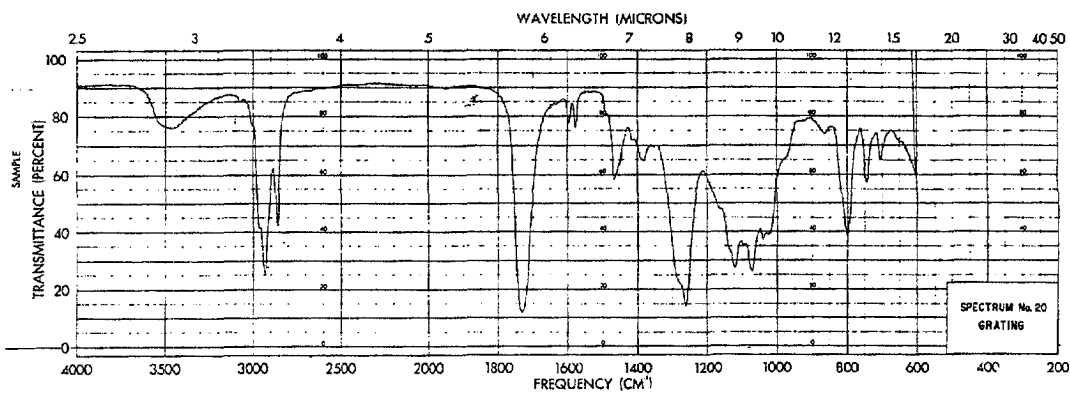
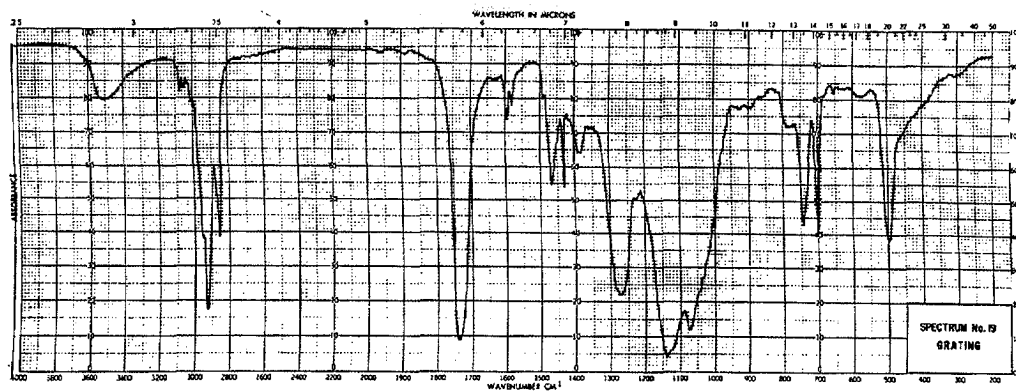


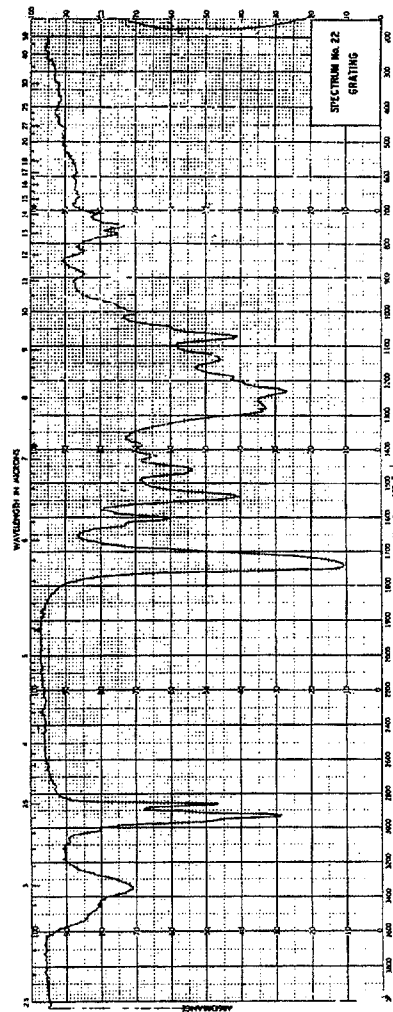
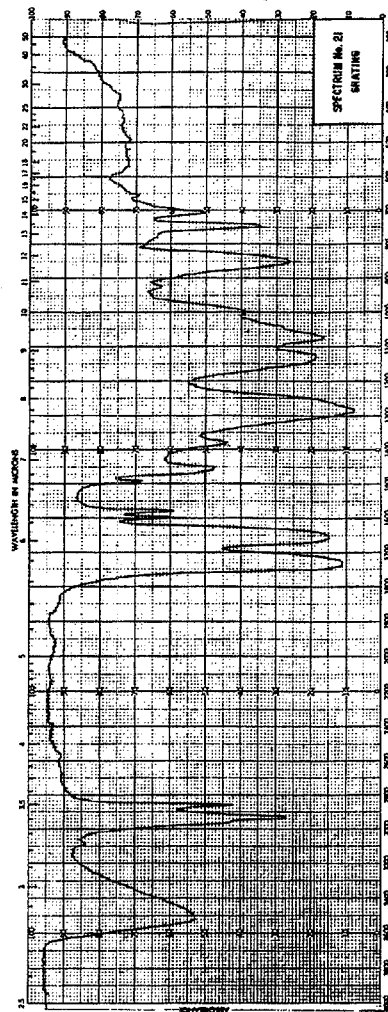


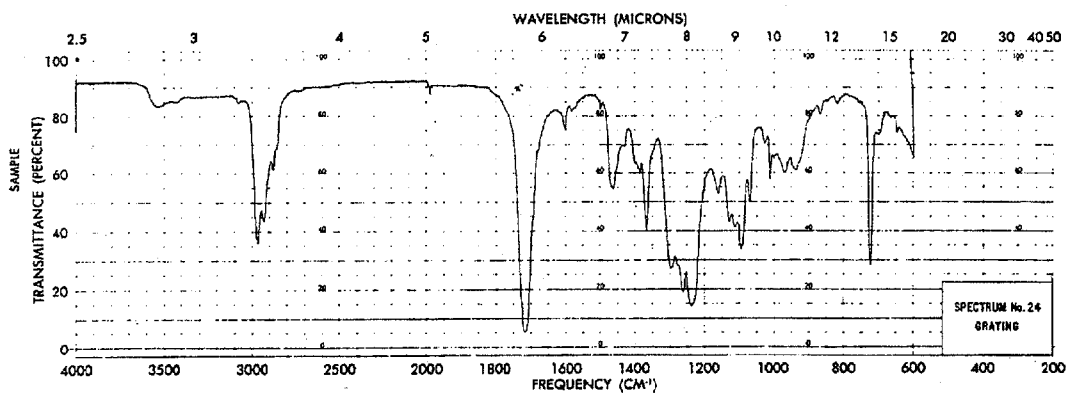
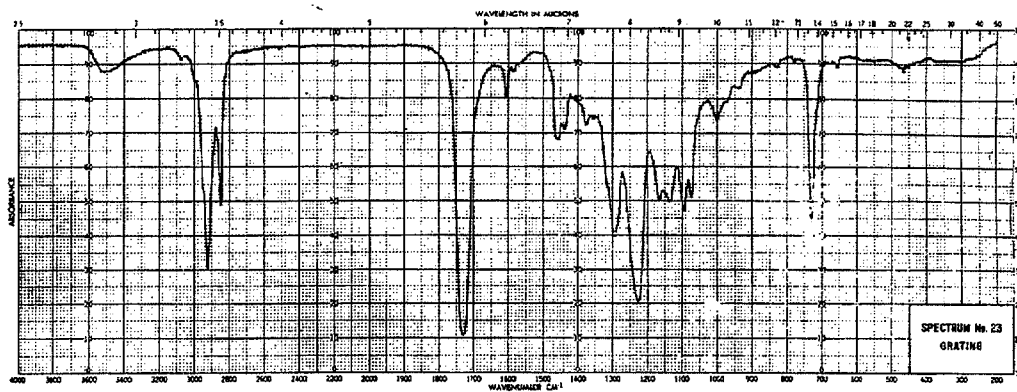


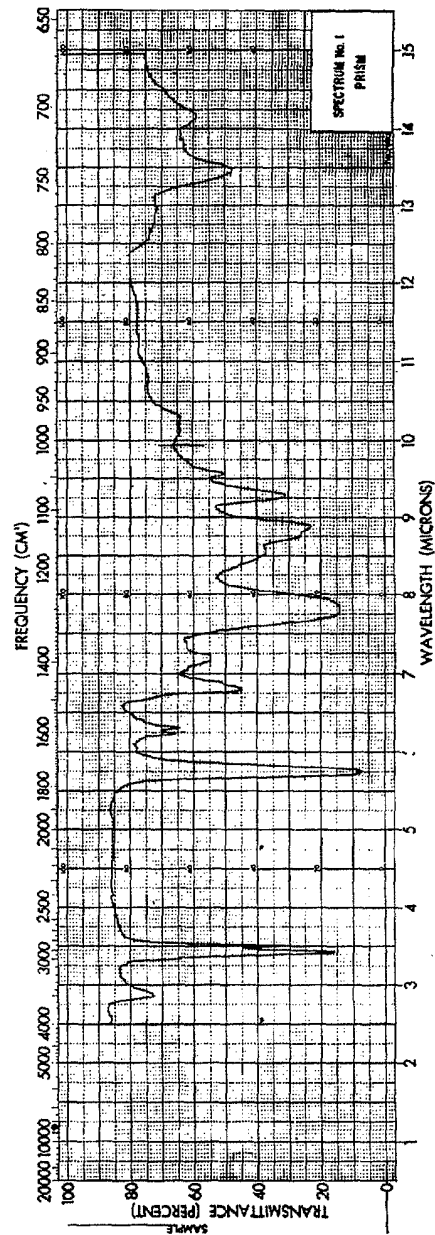
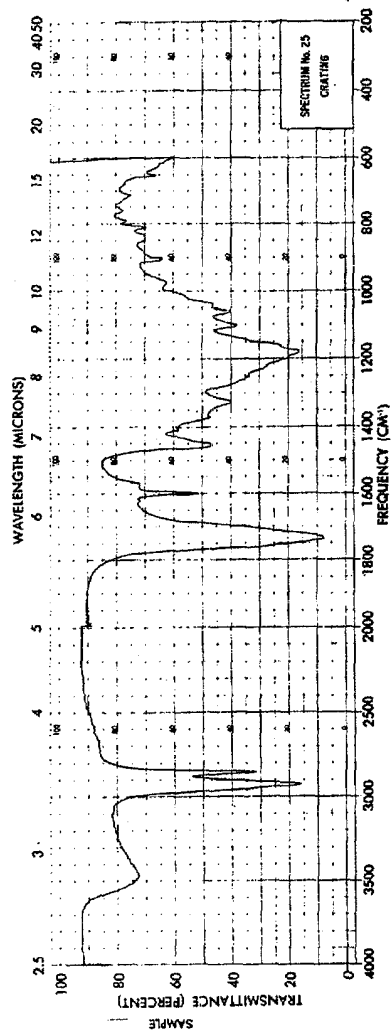


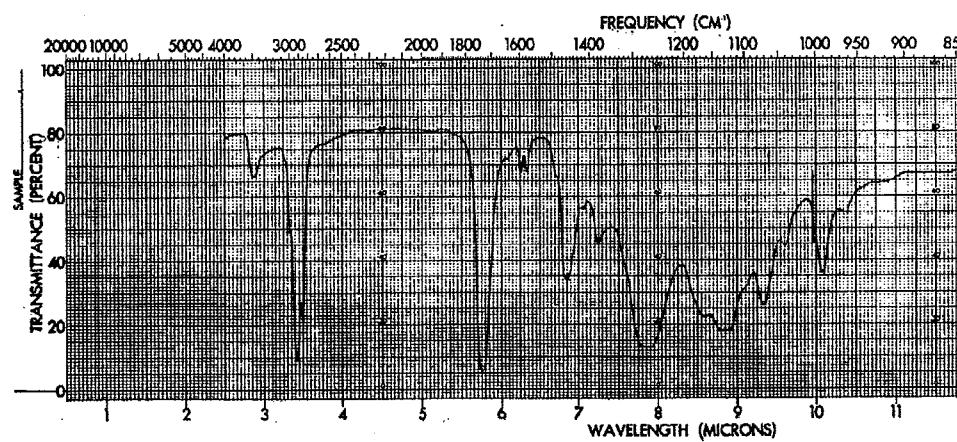
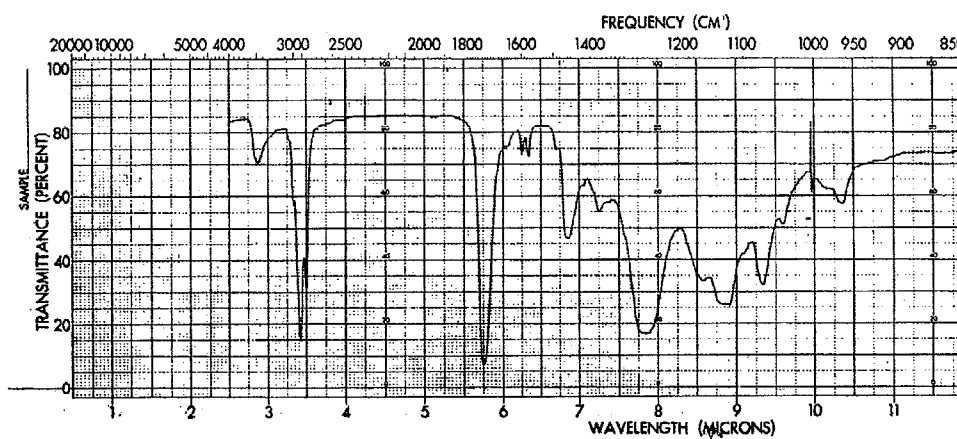




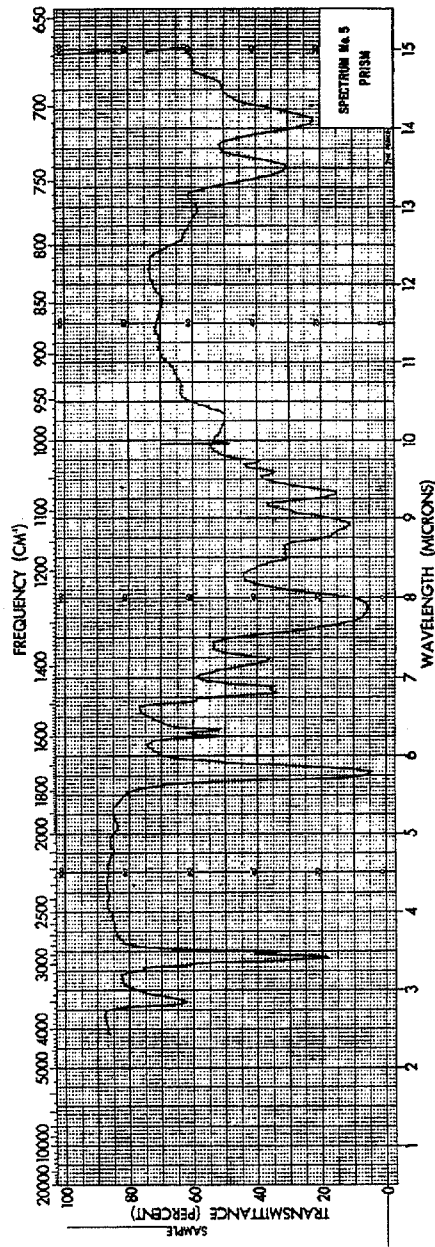
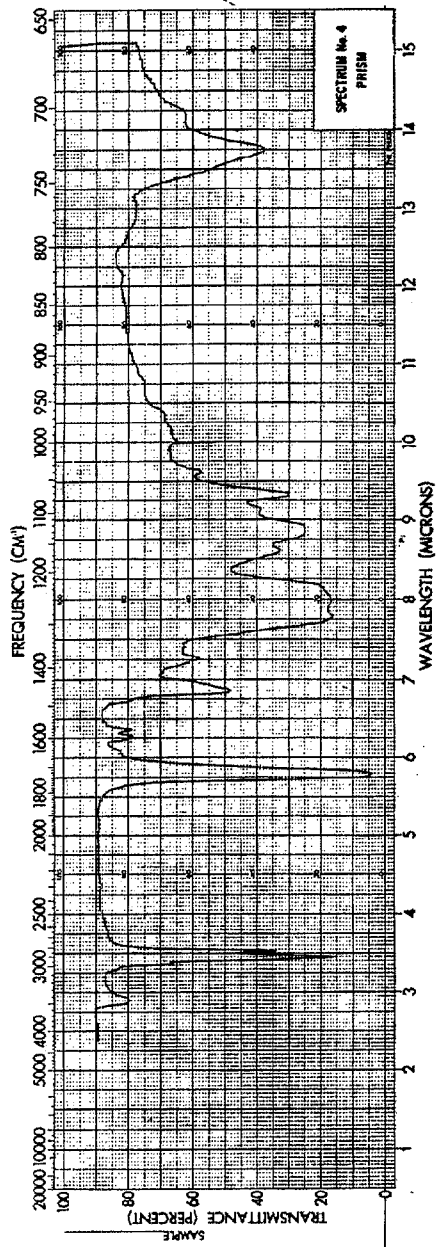


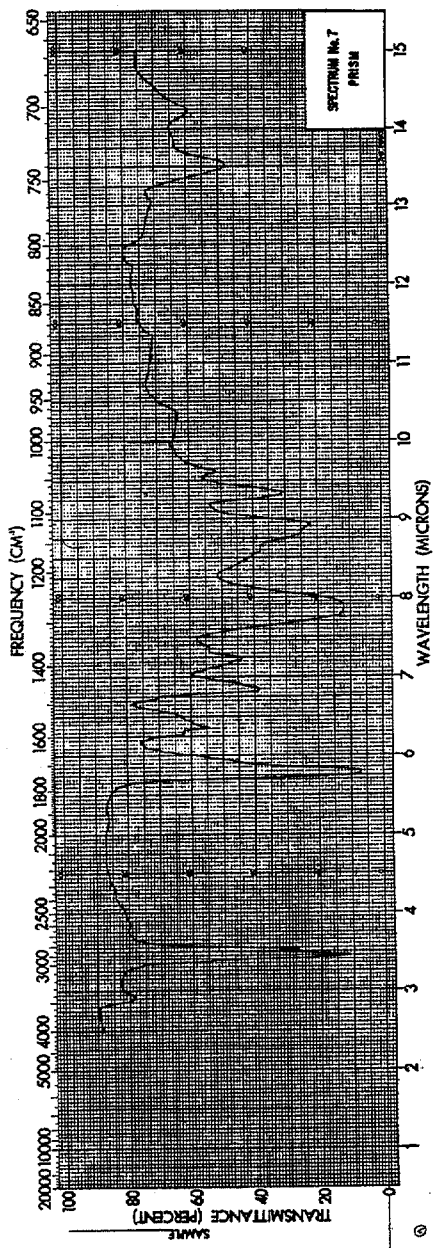
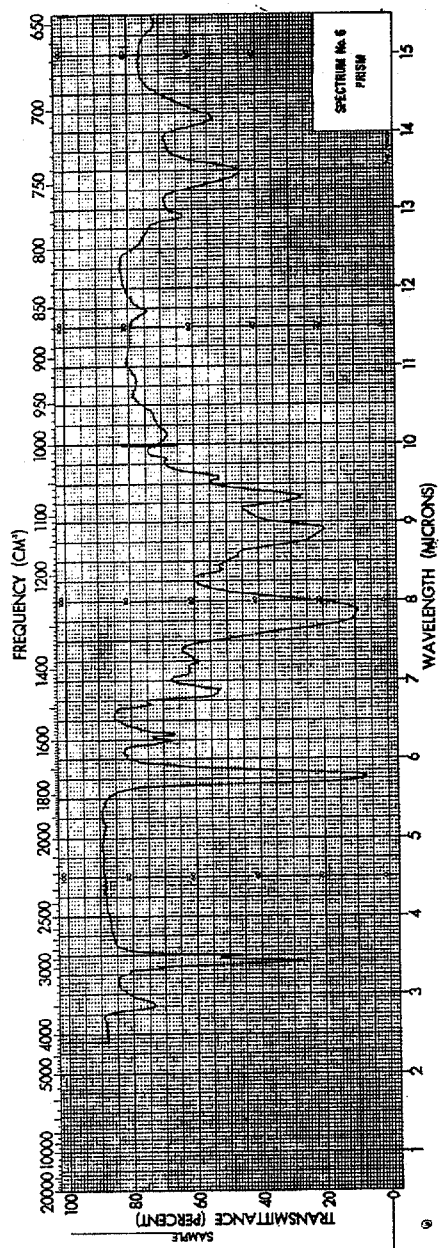


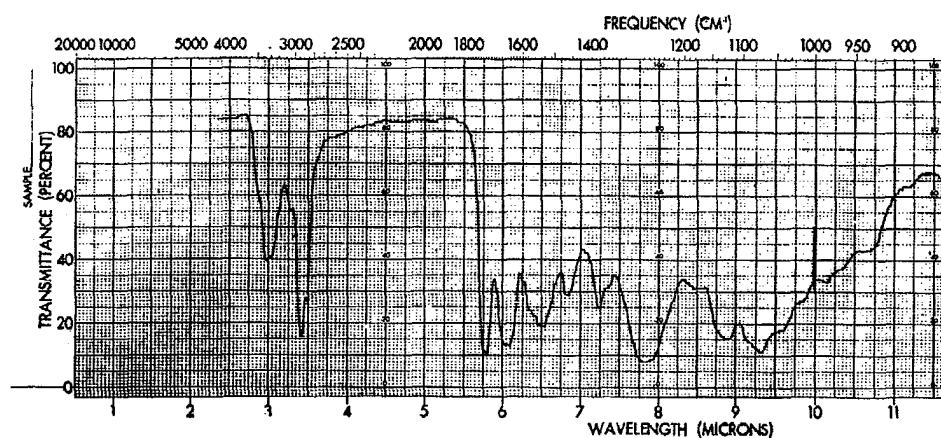
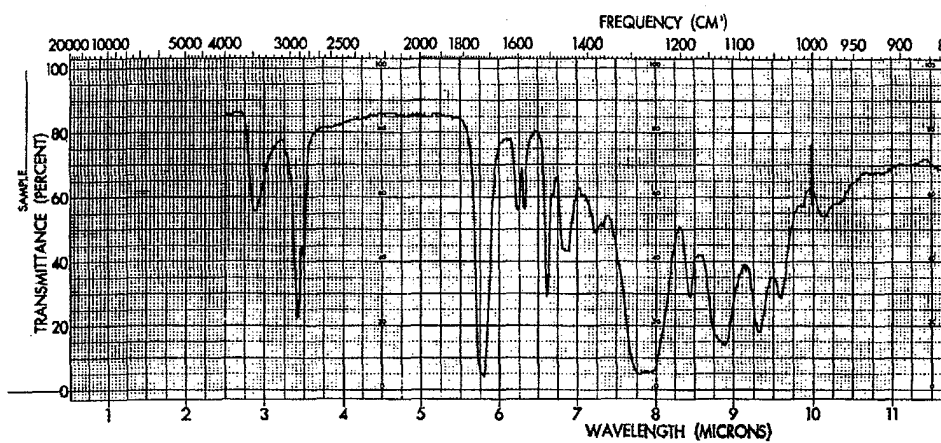


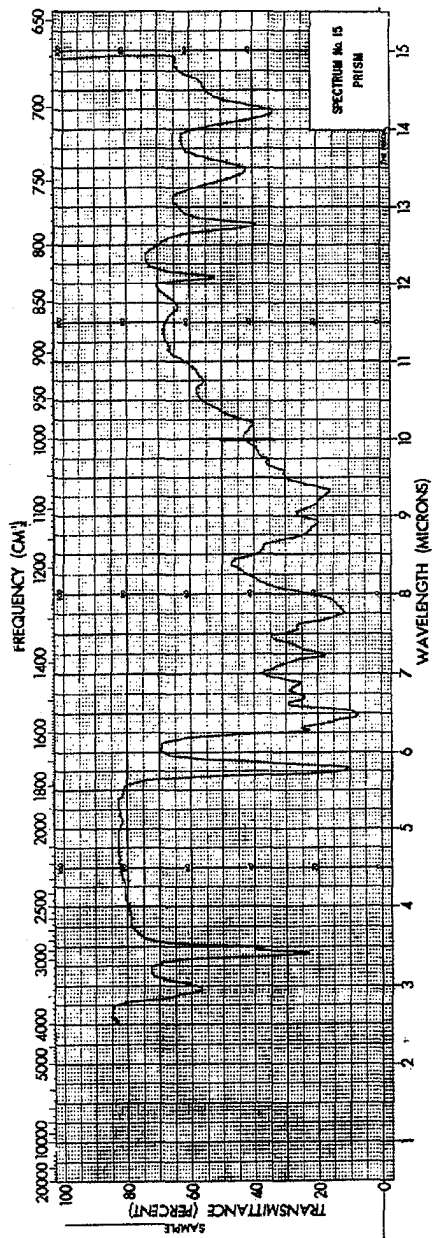
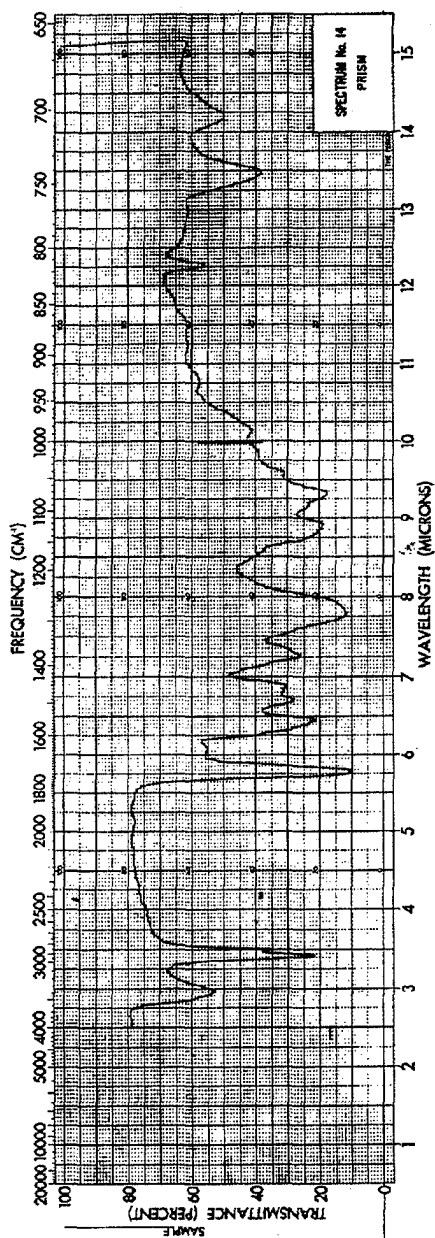


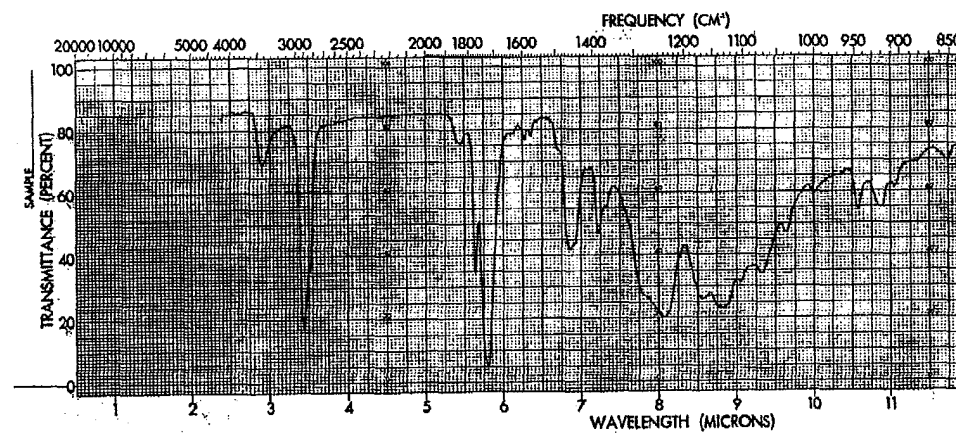
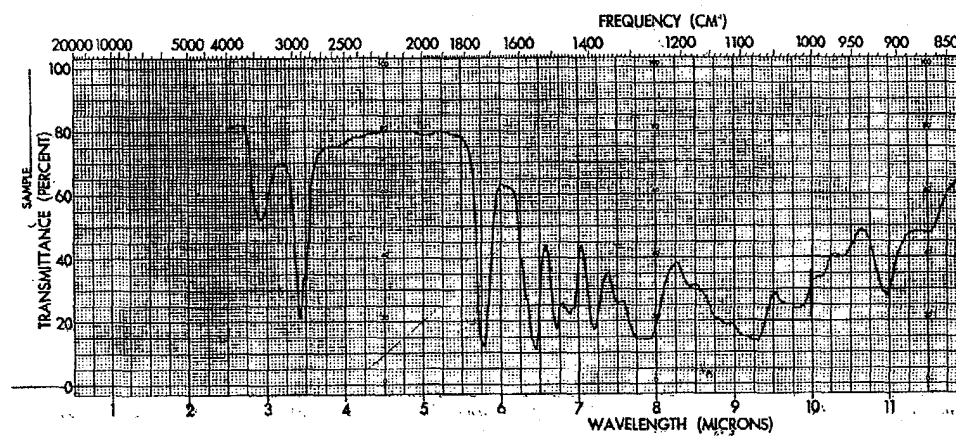
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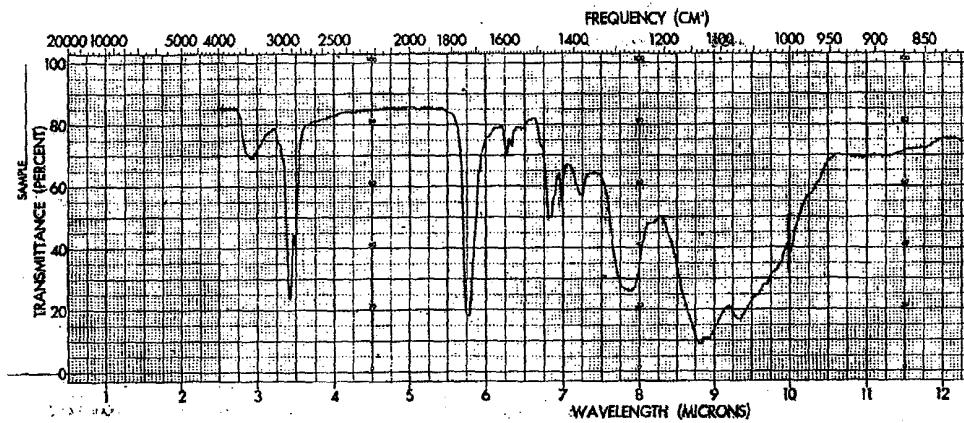
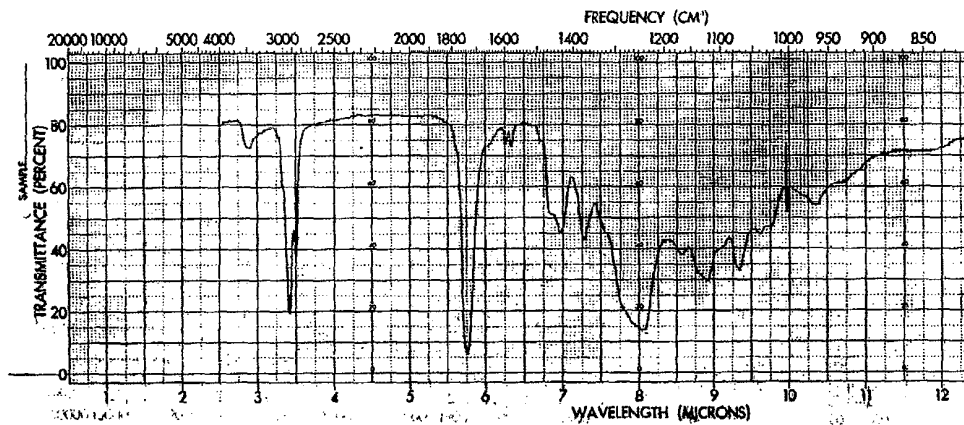




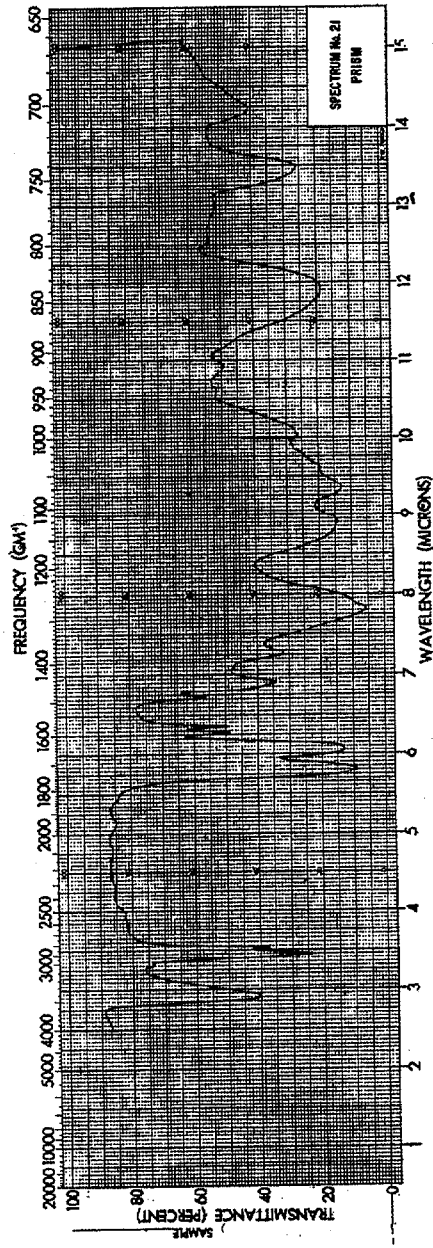
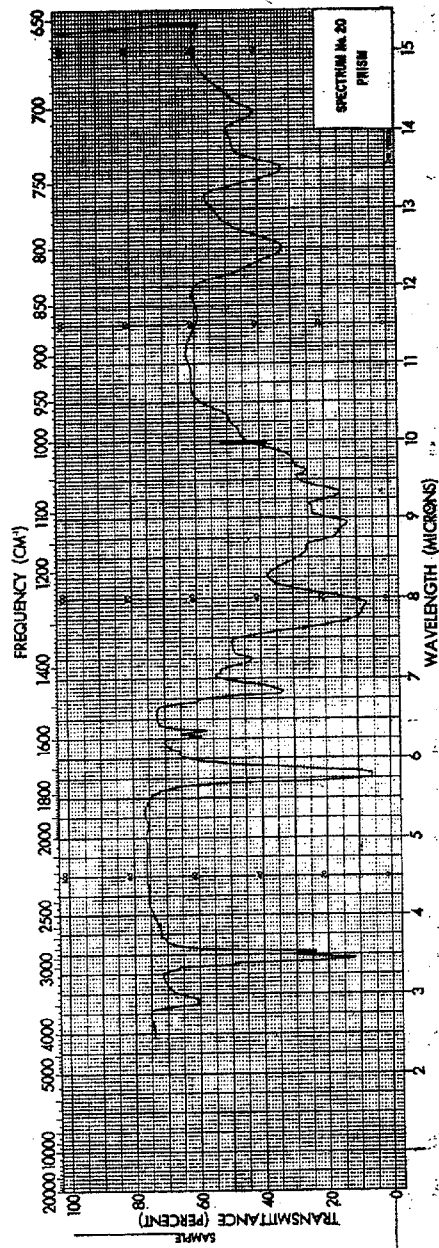


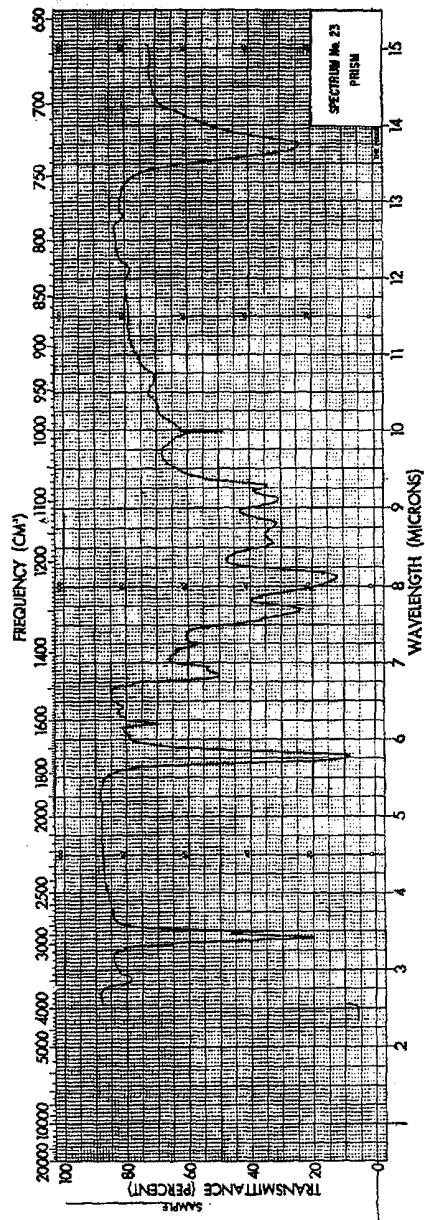
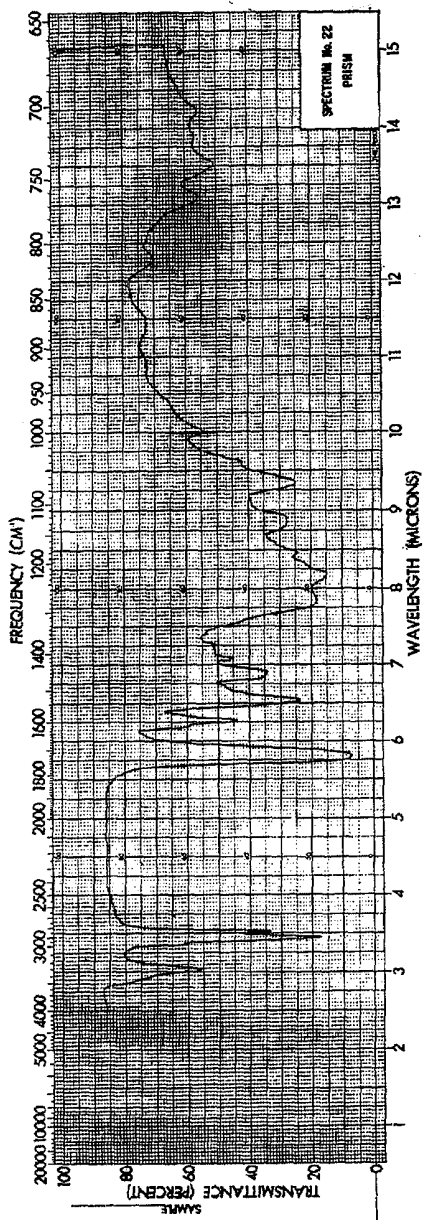
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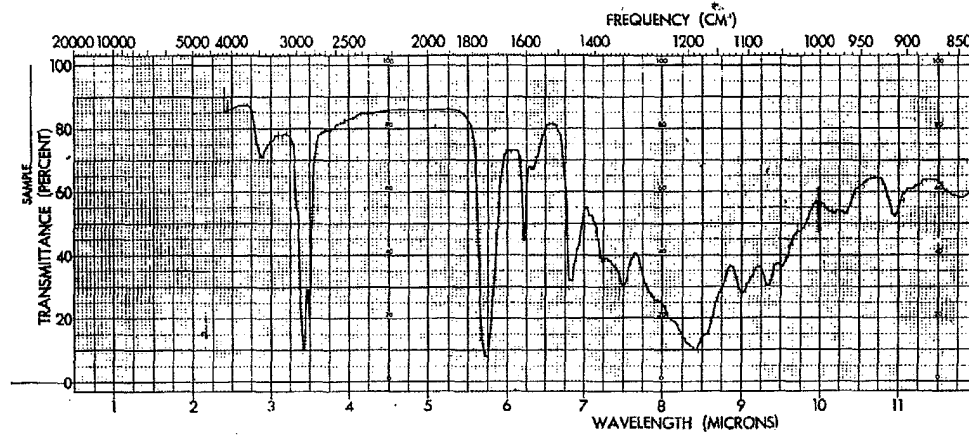
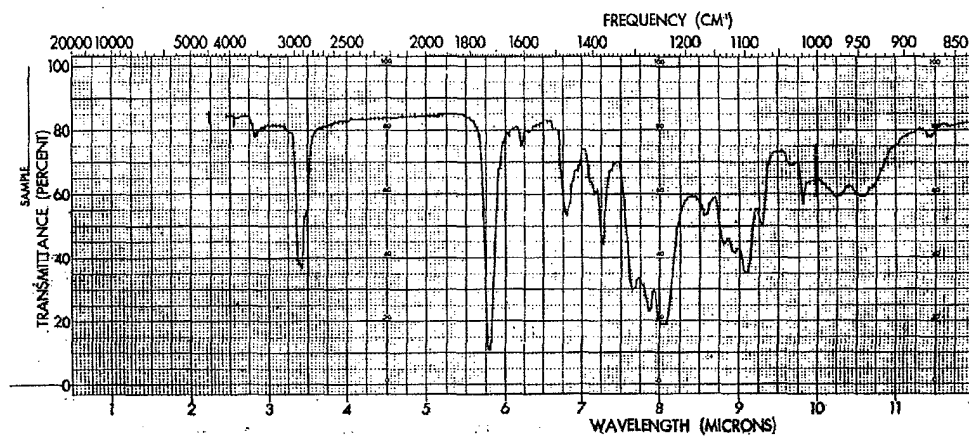
355



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A3. SOURCES OF INFRARED SPECTRA OF KNOWN MATERIALS

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Standard Test Methods for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products^{1,2}

This standard is issued under the fixed designation D 2691; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 These test methods cover the measurement of film thickness of dried coatings applied to a plane rigid substrate of wood or a wood-base material. These test methods apply to both opaque and clear coatings. They are not recommended for metal or glass substrates. Test Method A uses a razor blade and Test Method B a microtome for cross sectioning.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 This test method provides a precise, quantitative measurement of the thickness of a dried coating. It may be used for quality control or monitoring of the production of coated wood products.

3. Apparatus

3.1 *Calibrated Monocular Compound Microscope*, equipped with an optical system sufficient to provide sharp resolution of the cross section to be examined. One system, consisting of a 16-mm objective and a 10-power filar micrometer eyepiece, resulting in a magnification of approximately 100 diameters, has been found satisfactory. Other combinations of objectives and eyepieces and other magnifications may also be suitable, although magnifications above 200 diameters may result in distortion of the viewed cross section.

3.2 *Tool for Extracting Test Specimens*—The tool may be similar in design to an ordinary cork borer, except that it shall be made of thicker walled tubing, hard enough to hold a keen cutting edge, and be fitted with a head suitable for pounding with a hammer (Fig. 1). A leather punch has been found satisfactory for this use. The tool shall make a circular cut approximately $\frac{1}{2}$ in. (12.7 mm) in diameter and $\frac{1}{8}$ in. (3.2 mm) deep through the coating and into the substrate. A sidewise movement of the punch while firmly embedded in the substrate will dislodge the specimen and allow its

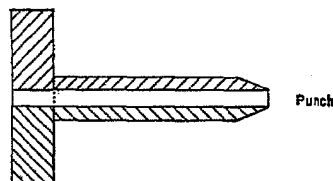


FIG. 1 One Type of Tool for Extracting Coated Wood Specimens

removal intact within the punch.

3.3 *Source of Oblique Illumination.*

3.4 *Hand Microtome (Method B).*

4. Test Specimens

4.1 Specimens for microscopical measurement shall be extracted from the punch tool by means of a push rod inserted through the hollow shaft of its tube. The end of the push rod should be tipped with felt or other soft material so that the coated surface of the disk will not be damaged during extraction.

5. Procedure

5.1 Prepare disks for microscopical examination and measurement by either Method A or Method B. Either method of preparation will expose a cross section of coating and substrate, including the extremes in film thickness resulting from variations in the wood surface or method of application.

5.2 *Method A, Cross Sectioning with Razor Blade*—With a sharp razor blade carefully slice away parallel to the coated surface the substrate adhering to the coating until the disk is approximately $\frac{1}{16}$ in. (1.6 mm) thick. Place the disk coated face down on a smooth, hard surface and cut in half by means of a slicing stroke with a fresh razor blade (Fig. 2). Cut in a direction perpendicular to the grain of the wood substrate and at right angles to the coated surface.

5.3 *Method B, Cross Sectioning with Microtome:*

5.3.1 With a sharp razor blade carefully slice away parallel to the coated surface the substrate adhering to the coating until the disk is approximately $\frac{1}{16}$ in. (1.6 mm) thick.

5.3.2 Embed the thinned disk in molten paraffin³ which is

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.52 on Factory-Coated Wood Products.

Current edition approved March 29, 1988. Published May 1988. Originally published as D 2691 - 68. Last previous edition D 2691 - 70 (1981)⁴.

² This test method is presently under study for revision or replacement with an improved microscopical film thickness method.

³ Paraplast, available from Fischer Scientific, 191 South Gulph Road, P.O. Box J, King of Prussia, PA 19406 melting point about 50 to 60°C, has been found satisfactory for this purpose. For clear films the addition of 0.5 g of Indigo to 200 mL of paraffin improves optical definition.

FIG. 2

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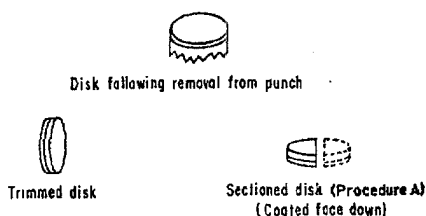


FIG. 2 Coated Wood Specimens Before and After Trimming and Coating

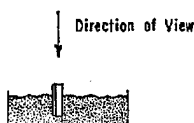


FIG. 3 Specimen Mounted in Clean Sand for Microscopical Examination (Method A)

allowed to harden in a small mold to form a block which surrounds the disk on all sides. (The disk may also be dipped successively in the molten paraffin until a reasonably heavy deposit of wax is obtained.)

5.3.3 Trim the paraffin block with razor blade until the sides are parallel to the woodcoating surface.

5.3.4 Mount the trimmed block in a sliding-knife hand microtome⁴ in such a manner that the embedded disk is vertical with its cross-sectional edge facing and perpendicular to the oncoming knife edge. Cut perpendicular to the grain or to the machine direction of the specimen. The block should be firmly clamped between the jaws of the microtome chuck so that approximately one third of the diameter of the embedded disk protrudes.

5.3.5 Make successive cuts with a single sliding movement of the knife, raising the block by small increments after each cut. Continue sectioning until the clamped paraffin block protrudes no more than 1/16 in. (1.6 mm) above the jaws of the microtome chuck and the embedded disk is sectioned to approximately one half its diameter.

5.3.6 Examine the exposed cross section of the embedded disk with a hand lens to ascertain that a smooth cut has been obtained. If not, resume sectioning until this is accomplished.

6. Microscopical Measurement

6.1 Mount the cross-sectional disk for microscopical examination by carefully mounting in parallel clamps, modeling clay, or in clean sand held in a small container such as a petri dish (Fig. 3). Examine the edge-on view of the specimen by oblique overstage illumination. A focusable microscope lamp and a rotatable microscope stage are recommended for obtaining optimum viewing conditions.

6.2 Determine thickness of the coating above the substrate at ten randomly selected points on the cross section.

Do not measure within 1/32 in. (0.8 mm) of the edges of the disk, nor in areas where obvious deformation of the coating or substrate has occurred.

7. Report

7.1 Report the average film thickness computed from the ten random measurements as well as the maximum and minimum thicknesses measured to the nearest 0.1 mil (2.5 μm).

7.2 If a more extensive sampling procedure is employed, the report shall show the number of disks examined and the number of measurements per disk, in addition to the maximum, minimum, and average film thickness computed from all measurements.

8. Precision

8.1 Opaque Coatings:

8.1.1 *Test Method A*—On the basis of an interlaboratory test of this method in which operators in six laboratories tested 12 materials covering a wide range in film thickness, the within-laboratory standard deviation was found to be 0.26 mils with 72 degrees of freedom, and the between-laboratory standard deviation was found to be 1.03 mils with 60 degrees of freedom. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95% confidence level:

8.1.2 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.7 mils.

8.1.3 *Reproducibility*—Two results, each the mean of duplicate measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 3 mils.

8.1.4 *Test Method B*—On the basis of an interlaboratory test of this method in which operators in three laboratories tested 12 materials covering a wide range in film thickness, the within-laboratory standard deviation was found to be 0.28 mils with 36 degrees of freedom and the between-laboratory standard deviation was found to be 0.66 mils with 24 degrees of freedom. Based on these standards deviations, the following criteria should be used for judging the acceptability of results at the 95% confidence level.

8.1.5 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.8 mils.

TABLE 1 Laboratory Standard Deviations of Opaque Coatings

Substrate	Standard Deviation			
	Between		Within	
	Method A	Method B	Method A	Method B
Wood	0.97	0.66	0.30	0.34
Hardboard or overlaid plywood	1.09	0.25	0.22	0.21

TABLE 2 Repeatability

Substrate	Method A	Method B
Wood	0.8	1.0
Hardboard or overlaid plywood	0.6	0.6

⁴ A Spencer microtome (Catalog No. 900), available from American Optical, Warner-Lambert Co., Reichert Scientific Instrument Division, 201-T Tabor Rd., Morris Plains, NJ 07950 has been found suitable for this purpose.

8.1.6 *Reproducibility*—Two results, each the mean of duplicate measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 2 mils.

8.2 *Clear Coatings*:

8.2.1 *Test Method B*—On the basis of an interlaboratory test of this method in which operators in six laboratories tested 16 materials covering a wide range in film thickness, the within-laboratory standard deviation was found to be 0.07 mils with 96 degrees of freedom, and the between-

laboratory standard deviation was found to be 0.10 mils with 80 degrees of freedom. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95% confidence level:

8.2.2 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.2 mils.

8.2.3 *Reproducibility*—Two results, each the mean of duplicate measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.3 mil.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings¹

This standard is issued under the fixed designation D 2697; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Keywords were added editorially in January 1991.

1. Scope

1.1 This test method is believed to be applicable to the determination of the volume of nonvolatile matter of a variety of coatings. An interlaboratory study to establish the precision of this test method included a water-reducible exterior latex paint and three automotive coatings that included a solvent-reducible primer surfacer, water reducible primer surfacer, water reducible enamel topcoat, and acrylic dispersion lacquer topcoat. Earlier collaborative studies included a gloss enamel, a flat wall paint, a gloss house enamel, an industrial baking enamel, an interior latex paint, and an exterior latex paint.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products²
- D 2369 Test Method for Volatile Content of Coatings²
- D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²
- D 3980 Practice for Interlaboratory Testing of Paint and Related Materials²

3. Summary of Test Method

3.1 The weight and volume of a stainless steel disk are determined. After the disk is coated with the material being tested, the weight and volume of the disk plus dried coating is determined by weighing in air and then by weighing in a liquid of known density, the volume being equal to the quotient of the weight loss of the coated disk (due to the Archimedes buoyancy effect) divided by the density of the liquid displaced. The liquid may be water, organic liquid such as low-solvency mineral spirits or kerosine, or with special modifications not covered specifically in this method, mercury. The choice of liquid depends upon the nature of the coating tested.

NOTE 1—Distilled water is suitable for most paints. Exceptions are coatings that contain ingredients that are readily leached out of the dry film by the water and low-gloss coatings, the surface of which is poorly wet by water even with surfactant added. (Note 2) Low-solvency hydrocarbon solvent (KB below 36) is also practical for most paints and is preferred by some workers.³ It is considered to be particularly good for paint films not readily wet by water. Analogously, organic solvents must not be used if the coating to be tested contains ingredients that will be dissolved readily by the solvent. Lacquers containing monomeric plasticizers would be examples where hydrocarbon solvents should definitely not be used. Coatings formulated much above the CPVC present a special problem, where mercury might be the desired "suspending" liquid (Note 3), and for solvent-reducible paints hydrocarbon solvent might be considered the poorest (unless it is the objective to obtain values closer to "theoretical" spaces between pigment particles not filled with binder, becoming partially filled with solvent during the test).

Note 2—Concentration of surfactant must be kept very low or literature values for the density of the water cannot be used.

Note 3—Details of the mercury displacement techniques can be found in the literature.⁴

3.2 From the measured weights and volumes of the disk before and after coating, the weight and volume of the dried coating film are calculated. Based on the density of the liquid coating and the weight percent nonvolatile matter, the volume of the liquid coating deposited on the coated disk is calculated. The volume of the dried coating divided by the volume of liquid coating, multiplied by 100, provides the volume percent nonvolatile matter in the total liquid coating.

4. Significance and Use

4.1 This test method is intended to provide a measure of the volume of dry coating obtainable from a given volume of liquid coating. This value is useful for comparing the coverage (square feet of surface covered at a specified dry film thickness per unit volume) obtainable with different coating products.

4.2 For various reasons the value obtained may not be equal to that predicted from simple additivity of the weights and volumes of the raw materials in a formulation. One reason is that the volume occupied by a solution of resin in solvent may be the same, greater, or less than the total volume of the separate ingredients: such contraction or expansion in resin solutions is governed by a number of factors, one of which is the extent and direction of spread

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved April 25, 1986. Published June 1986. Originally published as D 2697 - 68. Last previous edition D 2697 - 73 (1979).

² Annual Book of ASTM Standards, Volume 06.01.

³ Bissey, J. E., *Official Digest*, Federation of Paint and Varnish Production Clubs, Vol 35, 1963, p. 1072, and Ashton, H. E., *Materials Research and Standards*, Vol 1, 1961, p. 549.

⁴ Cole, R. J., *Journal, Oil Colour Chemists' Assn.*, Vol. 45, 1962, p. 776.

between solubility parameters of the resin and solvent.

4.3 The spatial configuration of the pigment particles and the degree to which the spaces between the pigment particles are filled with the binder also affect the volume of a dry coating formulation. Above the critical pigment volume concentration, the apparent volume of the dry film is significantly greater than theoretical due to the increase in unfilled voids between pigment particles. The use of volume nonvolatile matter values in such instances should be carefully considered as the increased volume is largely due to air trapped in these voids.

5. Apparatus

5.1 Analytical Balance.

5.2 *Steel Disk*, preferably stainless steel, 2½ in. (60 mm) in diameter and 22 gage (0.65 mm) in thickness with a small hole near the circumference. A fine wire, such as Chromel A, 28 gage (0.32 mm), is attached through the hole and made the appropriate length for subsequent suspension of the disk in a liquid. The wire should have a small loop on the upper end so the disk and wire can be hung by this loop on the balance.

NOTE 4—Instead of steel disks, some analysts use aluminum tubes. In the round-robin results, essentially no difference was found in the precision obtained by both methods. Source and dimensions of these tubes are described in the annex.

5.3 *Counterweight*, to be placed on the balance stirrup after hanger bow and pan are removed.

5.4 *Beaker, 1-L*—For easier manipulation during the weighing of disk in liquid it is advisable to cut the beaker to a height of 4½ in. (115 mm).

5.5 Support for holding the beaker under the balance stirrup without jamming the pan damper in the floor of the balance. A cork or neoprene ring is suitable when a single-pan balance is used.

5.6 *Weight per Gallon Cup*, acrometer, or other suitable means for determining the density of the coating material and the suspending liquids if not known.

6. Volume Determination of Uncoated Disks

6.1 Dry the disk in an oven at $110 \pm 5^\circ\text{C}$ for 10 min. Cool and weigh the disk in air.

6.2 Weigh the disk in the liquid to be used for suspension of the coated disk. If water is used as the suspending liquid, a few drops of wetting agent (Note 2) added to the liquid will help to ensure rapid and thorough wetting of the disk. Be careful that no air bubbles form on the disk or wire. Mark the level of liquid in the 1-L beaker necessary for complete immersion of the disk which should be at least ¾ in. (20 mm) above the disk. Maintain this level in subsequent weighings when the disk is coated.

6.3 Record the temperature of the liquid. Obtain the density of the liquid at the temperature used, from a table, such as is found for pure water in *Handbook of Chemistry and Physics*,⁵ or determine it to 0.001 g/mL.

6.4 Calculate the volume of the disk, G , in millilitres as follows:

$$G = (w_1 - w_2)/D$$

where:

w_1 = weight of disk in air, g

w_2 = weight of disk in liquid, g, and

D = density of liquid at temperature of test, g/mL.

7. Procedure

7.1 Take a representative sample of the liquid coating in accordance with Practice D 3925. Mix thoroughly before taking specimens for the individual tests.

7.2 Determine the weight nonvolatile of the liquid coating by drying 1 h at $110 \pm 5^\circ\text{C}$ in accordance with Test Method D 2369.

NOTE 5—If this method does not apply, then the method used should be agreed upon between producer and user.

7.3 Determine to 0.001 g/mL the density of the liquid coating in accordance with Test Method D 1475.

7.4 Dip the disk in the liquid coating and allow the liquid to come up on the wire a distance from ¼ to ½ in. (5 to 15 mm). Allow about 10 min for draining, and blot the coating material off the bottom edge of the disk so that beads or drops do not dry on the bottom edge of the disk.

NOTE 6—In some cases the paint or varnish may be of such consistency that the amount of solid matter remaining on the disk after drying is too small for an accurate volume determination. The use of a flat pan with a sidewall about 10 mm in height in place of the disk enables the operator to obtain a more desirable volume of solid matter. However, extra care must be observed to prevent trapping of air at the point where the sidewall meets the bottom of the pan. In no case should bubbles be allowed to be present in cast films. This procedure has not been evaluated and no precision statement is available.

7.5 When beads or drips stop forming, hang the disk in the oven for 1 h at 110°C (Note 5). Remove and cool. Weigh the coated disk in air.

7.6 Weigh the coated disk in the chosen medium in the same manner as for the uncoated disk, recording the temperature of the liquid at the time of the test.

8. Calculations

8.1 Calculate the volume of the coated disk, H , in millilitres, as follows:

$$H = (w_3 - w_4)/D$$

where:

w_3 = weight of coated disk in air, g,

w_4 = weight of coated disk in liquid, g, and

D = density of liquid at temperature of test.

8.2 Calculate the volume of the dried coating, F , in millilitres, as follows:

$$F = H - G$$

8.3 Calculate the volume of wet coating, V , in millilitres, from which the dried coating was obtained, as follows:

$$V = (w_3 - w_1)/(w \times \rho)$$

where:

w = nonvolatile matter in 1 g of wet coating, g, and

ρ = density of liquid coating material.

8.4 Calculate the percent volume nonvolatile content in a liquid coating as follows:

$$(F/V) \times 100$$

NOTE 7—The displacement liquid used should be reported with volume percent nonvolatile results. The method of drying the films should also be stated if different from that specified.

⁵ CRC Press, Inc., West Palm Beach, FL, 1986.

9. Precision⁶

9.1 *Precision (In accordance with Practice D 3980)*—In an interlaboratory study of this test method in which one operator in each of five laboratories analyzed in duplicate on two days four coatings (two solvent-reducible and two water-reducible) with nonvolatile contents ranging from 24 to 35 volume %, the pooled within-laboratory standard deviation was found to be 0.444 % with 17 degrees of freedom (DF) and the pooled between-laboratories standard deviation 1.195 % with 16 DF, after discarding one day's results from two laboratories on one sample, one day's results from one laboratory on another sample, and one

duplicate result from one laboratory on a third sample. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 1.32 % absolute at volume nonvolatile contents of 24 to 35 %.

9.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 3.59 % absolute at the same levels.

9.2 *Bias*—Bias has not been determined.

⁶ Supporting data are available from ASTM Headquarters. Request RR:D01-1052.

10. Keywords

10.1 volume nonvolatile content

ANNEX

Nonmandatory Information

A1. Aluminum Tubes

A1.1 Aluminum tubes,⁷ uncoated, plain with no cap or liner, #16 neck and orifice, 1¼ by 6¼ in., (32 mm by 160 mm).

A1.2 Cut two 3-in. (75 mm) lengths of tube from the aluminum tubing. Make a ¾-in. (20 mm) cut on the flattened end of the tube about ¼ in. (6 mm) from the end. Slip the tube over a short length of 1 in. (25 mm) inside diameter electrical conduit and return the tube to a round condition. Remove the tube from the pipe and press 1 in. wide strip at an end of the tube toward the center to serve as a hangar attachment.

⁷ Tubes manufactured by Teledyne; 2290 W. Townsend St., Chester, PA 19016, have been found satisfactory for this purpose.

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Standard Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging¹

This standard is issued under the fixed designation D 2698; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Methods 4022, 4032, 4052 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the separation of pigment from solvent-type paints and the calculation of the percent pigment from the results of nonvolatile determinations on the total paint and the separated vehicle.

NOTE 1—This test method has been proven to be applicable to most solvent-type paints, the exception being those paints containing severely bleeding pigments or pigments such as carbon black that are very difficult or impossible to centrifuge.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

- 2.1 *ASTM Standard:*
D 2369 Test Method for Volatile Content of Coatings²

3. Significance and Use

3.1 This test method describes a rapid method for separating pigment from solvent-reducible paints.

3.2 This test method is used by paint producers and consumers for product acceptance and process control.

4. Apparatus

- 4.1 *Laboratory Centrifuge*, capable of developing 32 000 g or higher.³

5. Procedure

5.1 Shake the sample for 10 min on a mechanical shaker, stir with a paddle to loosen any caked pigment, and shake again for 10 min. If necessary, repeat until there are no lumps present and the sample is thoroughly mixed.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 25, 1990. Published July 1990. Originally published as D 2698 - 68. Last previous edition D 2698 - 73 (1984)⁴.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ A Sharples supercentrifuge or an International centrifuge equipped with a high-speed head have been found satisfactory for this purpose. Any centrifuge capable of developing 32 000 g and having facilities for sealing the specimen to prevent solvent loss should also prove to be satisfactory.

To calculate the gravities developed by a particular centrifuge use the following calculation:

$$g = \text{rotating radius, cm} \times r/\text{min}^2 \times 1.118 \times 10^{-5}$$

5.2 Add the mixed paint to the centrifuge container until sufficient paint is added to permit the recovery of 25 mL minimum of clear vehicle. Revolve the bowl at 32 000 g or higher for 15 min or until a clear vehicle is obtained.

5.3 Transfer the vehicle carefully, so as not to dislodge any of the pigment from the walls, into a jar that is immediately capped to prevent any solvent losses by evaporation. Use this vehicle for the determination of the nonvolatile content of the separated vehicle.

6. Percent Paint and Vehicle Solids

6.1 Determine the percent nonvolatile content of the original paint and the separated vehicle in accordance with Test Method D 2369.

7. Calculation

7.1 Calculate the weight percent pigment of paint, P , as follows:

$$P = (A - B) \times 100/100 - B$$

where:

A = nonvolatile content of paint, %, and

B = nonvolatile content of separated vehicle, %.

8. Recovery of Pigment for Analysis

8.1 The pigment may be completely recovered from the centrifuge bowl and washed to remove residual vehicle. This may be done either by a Soxhlet extraction or washing the pigment with a suitable solvent and recentrifuging.

NOTE 2—It has been noted that the removal of the pigment from the bowl of a Sharples supercentrifuge can be facilitated by lining the bowl with a piece of polyester film prior to adding the sample and subsequently just slipping the polyester film along with the pigment out of the bowl.

9. Precision and Bias

9.1 *Precision*—The precision estimates are based on an interlaboratory study of this test method in which air drying materials were analyzed in duplicate or triplicate. The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—The difference between two results, each the mean of duplicate determinations obtained by the same operator, will approximate 0.1 % absolute. Two such values should be considered suspect if they differ by more than 0.25 % absolute.

9.1.2 *Reproducibility*—The difference between two results, each the mean of duplicate determinations obtained by

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operators in different laboratories, will approximate 0.25 % absolute. Two such values should be considered suspect if they differ by more than 0.5 % absolute.

9.1.3 This precision pertains to air-drying materials. Heat-reactive vehicles will have a repeatability and reproducibility approximately twice as large, that is, 0.5 % and 1.0 % absolute.

9.1.4 It should be noted that when the solvent content of

the total paint is very low (15 % or less) the precision of this test method will not be satisfactory unless the nonvolatile determination is precise to the second decimal place.

9.2 Bias—Bias has not been determined for this test method.

10. Keywords

10.1 high speed centrifuging; pigment content; solvent-reducible paint

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Standard Practices for Uniformity of Traffic Paint Vehicle Solids by Spectroscopy and Gas Chromatography¹

This standard is issued under the fixed designation D 2743; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These practices provide general information on the instrumental techniques available for detecting adulteration or nonuniformity of the chemical nature of the vehicle solids in purchased lots of traffic paints by means of the individual or combined use of infrared and ultraviolet spectroscopy and gas chromatography. The procedures given are applicable when traffic paint is selected and purchased on the basis of pre-qualification laboratory or road performance tests, or both, and a *reference sample* of the original paint so evaluated and selected is retained and compared with test samples representative of subsequent purchased and delivered lots of such paint and which are required to be the same as the original reference sample.

1.2 Although not specifically provided for in these practices, the methods given may also be applied, with appropriate modification, to evaluating the acceptability of traffic paints that have been purchased on the basis of composition specifications. In such cases, application is limited to the vehicle solids as before, as well as the availability of a suitable standard or range of standards representative of the vehicle solids that are acceptable and with which samples of subsequent delivered lots will be compared.

1.3 The techniques provided are wholly adequate for detecting gross adulteration of the vehicle solids where completely different drying oils, resins, or polymers, or combinations of these have been substituted for those originally contained in the reference sample. In cases of lesser adulteration or modification, these methods have been found adequate for detecting vehicle solids, adulterations, or modifications as low as 5 weight % of the vehicle solids.

1.4 These techniques have been developed on the basis of cooperative work with alkyd, chlorinated rubber-alkyd, and poly(vinyl toluene) type paints involving the detection of nonuniformity when such extraneous materials as rosin, fish oil, hydrocarbon resin, and chlorinated paraffin have been added. The procedures given may be, but are not necessarily completely applicable to all other types of vehicle solids or extraneous additions, or both.

1.5 The methods provided appear in the following order:

	Section
Method A—Infrared Spectral Analysis of Total Vehicle Solids.	10 to 12
Method B—Infrared Spectral Analysis of Unsaponifiable Matter from Vehicle Solids	13 to 15

¹ These recommended practices are under jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved Aug. 15, 1968. Published October 1968.

Method C—Gas Chromatographic Analysis of Oils and Oil Acids Separated from Vehicle Solids	16 to 18
Method D—Ultraviolet Spectral Analysis of Total Vehicle Solids	19 to 21

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1259 Test Methods for Nonvolatile Content of Resin Solutions²
- D 1397 Test Method for Unsaponifiable Matter in Alkyd Resins and Resin Solutions²
- D 2245 Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints³
- D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints⁴
- D 2621 Test Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints⁴
- E 105 Recommended Practice for Probability Sampling of Materials⁵
- E 131 Definitions of Terms and Symbols Relating to Molecular Spectroscopy⁶

3. Terminology

3.1 For definitions of terms and symbols, refer to Definitions E 131.

4. Summary of Methods

4.1 Each of the methods given requires both a reference and a test sample of traffic paint and a preliminary separation and removal of the pigment component in each.

4.2 *Method A* involves infrared spectral analysis of cast films of the total vehicle solids to detect spectral differences between the reference and test samples caused by gross or minor adulteration of the test sample.

4.3 *Method B* involves infrared spectral analysis of cast films of the unsaponifiable matter that has been separated from the vehicle solids in order to detect spectral differences between the reference and test samples caused by lesser

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Annual Book of ASTM Standards, Vol 14.01.

adulteration readily evident by 4.4 Method A.

prepared from the differences either gross or with extraneous evident by 4.5 Method B.

analysis of spectral graphs to detect the adulteration be readily apparent.

5. Selection

5.1 All test samples between the test sample and the reference sample. It is not possible to use as a guide to the ratio of the reference sample to the test sample in order to determine the nature of the adulteration causing the difference.

5.2 *Method A* procedures for nonuniformities from indication of other methods is sufficient vehicle solid content spectral sample is corrected for drying oil, addition of resins, and characteristic cases it is by Methods B and C.

5.3 *Method B* unsaponifiable matter that has escaped adulteration in strong spectral differences of the vehicle solids precluding spectroscopic conclusions cause its spectral differences to be evident, failure to

adulterations of an unsaponifiable nature and which was not readily evident when using Method A.

4.4 Method C involves gas chromatographic analysis of prepared methyl esters of the separated fatty acids obtained from the vehicle solids in order to detect chromatographic differences between the reference and test samples caused by either gross or lesser adulteration of the drying oil fraction with extraneous drying oils which may not have been readily evident by the use of Methods A and B.

4.5 Method D involves quantitative ultraviolet spectral analysis of the total vehicle solids dissolved in a nonaromatic spectral grade solvent to give precise concentrations in order to detect ultraviolet spectral absorbance differences between the reference and test samples caused by minor or sophisticated adulterations of the vehicle solids and which may not be readily detected by Method A. Method D is to be used as an alternative to Methods B and C.

5. Selection of Test Methods and Significance and Use

5.1 All of the methods provided involve comparisons between the spectra or chromatograms of the reference and test samples to determine if they show significant differences. It is not possible at this time to establish quantitative limits as a guide to whether a spectral or chromatographic difference is truly significant. Certainly the presence or absence of a moderate or strong peak in the test sample which is not evident in the reference is significant. A persistent difference in the ratios of two peaks of one spectrum as compared to the reference sample is significant. On the whole, some judgment must be exercised in this respect and it is advisable to refer to published data on infrared or gas chromatography in order to establish, where feasible, the possible overall nature of the adulterant or its functional group which might be causing the comparison spectra to differ.

5.2 Method A is rapid and the most convenient of the procedures given. It should be utilized first in order to detect nonuniformity of the test sample. Significant spectral differences from that of the reference sample can be taken as an indication of adulteration and in such cases the use of the other methods is not necessary. As a general rule, Method A is sufficient to detect gross or major adulteration of the vehicle solids. However, where Method A shows no significant spectral differences, it cannot be assumed that the test sample is completely acceptable since changes in the type of drying oil, polyol, and certain dibasic acids in alkyd resins, addition of certain aliphatic or nonfunctional hydrocarbon resins, and many minor adulterations may not always show characteristic infrared spectral differences. Therefore, in such cases it is best to proceed to additional tests as given in Methods B and C or else alternatively directly to Method D.

5.3 Method B is useful in detecting adulterations that are unsaponifiable or else have an unsaponifiable component that has escaped detection in Method A only because the adulterant may have been small in amount and therefore its strong spectral peaks may have been masked over by the rest of the vehicle solids. Some care should be taken in interpreting spectral differences in Method B to avoid an erroneous conclusion that the test sample is unacceptable because its spectrum is different. Apparent but unreal differences can occur as a result of incomplete saponification, failure to remove all saponifiable material, and varying

degrees of contamination of the unsaponifiable fraction with sterols, etc., present in the vehicle solids. After it has thus been firmly established that a real spectral difference does exist, further tests are unnecessary, except that it is wise to resort again to the published literature on infrared to attempt to identify the possible nature of the adulterant. Where Methods A and B indicate acceptability of the test sample, it is still not always possible to rule out adulteration caused by changes or modifications in the saponifiable portion, that is, the type of fatty acid, dibasic acids, and polyol. In such cases, it is best to continue on to Method C for determination of the oil acids, and to other gas chromatographic methods for the polyol and dibasic acids when such equipment is available.

5.4 Method C is extremely sensitive in detecting adulterations and changes that have been made in the oil or fatty acid portion of the vehicle solids. It can, for example, detect whether linseed, coconut, oiticica, etc., has been substituted for soya oil and vice versa, or whether fish or tall oil has partially or wholly replaced some other drying oil, etc. Consequently, when the results of Methods A and B suggest that the test sample is acceptable and where a drying oil component is known to be present, Method C should be used additionally for more complete assurance of product uniformity. Where the results from Method C along with those from Methods A and B indicate product uniformity, it is a fairly safe assumption that the product has not been significantly altered.

5.5 Method D is intended as an alternative to Methods B and C and where the results from Method A indicate apparent product acceptability. Method D, by the use of quantitative ultraviolet spectral absorbance data, is an extremely sensitive procedure for the detection of complete or even partial adulteration of the test sample. However, considerable caution must be exercised in the preliminary pre-drying of the vehicle solids since it is at this stage that the components are extremely sensitive to oxidative changes. Even minor oxidative changes can seriously affect the absorbance data obtained in ultraviolet spectral analysis and may give an impression that the two samples being compared are different when in fact they are the same. When these considerations are provided for, and the comparison spectra are identical in Method D as well as in Method A, then it can be assumed that the sample is acceptable. Significant differences in the spectra from Method D would indicate nonuniformity of the product even though Method A may fail to reveal such nonuniformity.

6. Reference Sample

6.1 The reference sample of traffic paint should be at least ½ pt (250 mL) and should be truly representative of the initial paint found acceptable in pre-qualification laboratory or field service tests, or both, and which paint is subsequently specified for purchase.

6.2 In cases where paint is purchased on the basis of formulation specifications and it is desired to utilize these procedures to detect adulteration in delivered lots of paint, reference samples may be synthetically prepared to represent the extremes of the range that will be permitted in the formulation.

7. Test Sample

7.1 The test samples of the traffic paint should be at least ½ pt (250 mL) and should be representative of each delivered lot of paint that was specified for purchase and which is intended to be the same as the initial reference paint used in the pre-qualifications acceptance tests from which a reference sample was retained.

8. Sampling Reference and Test Samples

8.1 Test and reference samples of the traffic paint should be obtained in accordance with Recommended Practice E 105.

9. Preparation of Samples

9.1 Separate the vehicle from the pigment by centrifuging the paint in accordance with Method D 2372. Transfer and preserve the vehicle in a well-stoppered amber bottle.

METHOD A—INFRARED SPECTRAL ANALYSIS OF TOTAL VEHICLE SOLIDS

10. Apparatus

10.1 *Infrared Spectrophotometer*, recording double-beam, and accessory equipment as described in Test Method D 2621.

11. Procedure

11.1 Obtain the infrared spectra of a cast film of the vehicle solids of both the test and reference samples by utilizing the procedure referred to in 10.1. In all cases, however, the spectral intensity of both samples should be well matched (by adjusting the film thickness) to within 5 % transmission of each other at the strongest peak, and the transmission of this peak shall be between 5 and 15 %. It is also desirable to obtain additional spectra on thicker films or else ordinate scale expansion if available, to bring out spectral differences in the weak to moderate peak areas. Here again, the same degree of care should be taken as above to match the film thickness and thereby the overall spectral intensities of the two samples. If desired, an aid in comparison is to run the test sample against the reference sample in the reference beam in order to show significant differences by means of a single differential spectrum. Considerable caution is required in the use and interpretation of differential spectra as well as proper adjustments of gain, speed, and slit programs.

12. Interpretation of Results

12.1 Compare the companion spectra from the test and reference samples for identity by visual inspection preferably over a light box. Note particularly the presence of an extraneous peak or peaks in one which is (are) not in the other. Also note the ratio of intensities of two adjacent or pairs of peaks on one spectra and determine whether this ratio is similar on the comparison spectra. Any significant difference should be considered as an indication of lack of uniformity between the reference and test samples. Attempt to ascribe this difference to some extraneous component or formulation difference between the comparison samples by referring to available infrared literature and published spectra. Where it is evident that the comparison spectra are significantly different, no further tests are necessary. When

the spectra are identical, proceed to Methods B and C or alternatively to Method D for a fuller evaluation of possible minor or more sophisticated adulteration.

METHOD B—INFRARED SPECTRAL ANALYSIS OF UNSAPONIFIABLE MATTER FROM VEHICLE SOLIDS

13. Apparatus

13.1 Same as in 10.1.

14. Procedure

14.1 Treat a portion of the separated vehicle from both the sample and reference paints so as to obtain an ether solution of the unsaponifiable matter in accordance with Test Method D 1397. Take care to ensure rigorous quantitative separations and similar handling and exposure of the comparison samples. Determine the percentage of unsaponifiable matter. Obtain infrared spectra of cast films of the dried unsaponifiable matter in a manner similar to that described in 11.1.

15. Interpretation of Results

15.1 Compare the percentages of unsaponifiable matter. Compare the spectra and interpret the results in a similar manner as in 12.1. Care should be taken to avoid an erroneous conclusion that the materials are different when in fact they are the same. Such a misinterpretation could be caused by incomplete removal of oil acids and varying degrees of sterol contamination, solvent residues, etc. If it is firmly established that the companion samples are truly different, then further tests are unnecessary. If, on the other hand, the spectra and unsaponifiable contents are identical and the results from Method A were similar, then proceed to Method C to determine adulteration of the oil fraction or by oils if such is present or suspected of being present.

METHOD C—GAS CHROMATOGRAPHIC ANALYSIS OF OILS AND OIL ACIDS SEPARATED FROM VEHICLE SOLIDS

16. Apparatus and Reagents

16.1 Same as in Method D 2245.

17. Procedure

17.1 Proceed as in Method D 2245 utilizing a portion of the vehicles separated from both the test and reference samples.

18. Interpretation of Results

18.1 Compare the two recorded chromatograms and the calculated percentages of individual fatty acids for similarity. Note particularly the absence or presence of extraneous peaks in one and not the other as well as the percentages of individual fatty acids and polymers in one sample as

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METHOD D—ULTRAVIOLET SPECTRAL ANALYSIS OF TOTAL VEHICLE SOLIDS

19. Apparatus

19.1 *Spectrophotometer*, recording double-beam, suitable for use in the ultraviolet region of the electromagnetic spectrum from 190 to 350 nm.

20. Procedure

20.1 On a portion of the previously separated vehicles of the test and reference samples, determine the nonvolatile content by means of Test Method D 1259. On the basis of this determination, quantitatively weigh out, by difference, from the stoppered bottles of the total vehicle, samples to yield 0.2 ± 0.0005 g of vehicle solids and place in a 100-mL beaker. Take care to avoid weighing errors caused by evaporation of solvent in the stoppered bottle during the weighing process.

20.2 Evaporate the volatile material by placing the beaker over an even heat source maintained at 60 to 65°C and under a constant blanket of dry, inert gas, preferably nitrogen, fed through a bell jar in which the beaker is placed. The bell jar should also be fitted with a stoppered separatory funnel with its delivery tube directly over the beaker. Evaporate until all the solvent is completely removed but avoid excessive exposure to these conditions much beyond this point. With the inert gas flow uninterrupted, transfer about 25 to 30 mL of spectral grade cyclohexane to the beaker through the assembled separatory funnel. Remove the beaker and immediately stir to dissolve all the solid material in the solvent. Quantitatively transfer to a 50-mL volumetric flask and fill to mark with additional spectral solvent. This yields a 0.4 % stock solution (wt/vol) of the vehicle solids in solution. If solution is not complete, try gentle warming or else start over using another suitable spectral grade nonaromatic solvent with an ultraviolet cut-off point at least as low as 220 or 230 nm.

20.3 With appropriate calibrated pipets or hypodermic syringes, volumetric flask, and the same lot of solvent used before, prepare several dilute quantitative solutions from aliquots of each stock solution that will enable quantitative measurements of all peak heights in the subsequent ultraviolet analysis (Note 1). In all cases, a specific quantitative dilution of the reference sample should be matched by exactly the same dilution of the test sample. For each such

matched dilution, use the exact same pipet for the test sample as was used for the reference sample to avoid dilution errors of comparison samples.

NOTE 1—The concentrations and numbers of dilute solutions necessary to obtain all the spectral peaks characteristic of a material will vary with the type of vehicle solids under examination. Generally, three dilutions of each stock solution to yield 0.03, 0.01, and 0.005 % solutions should suffice.

20.4 Within 24 h, and after making certain that the dilute solution is perfectly clear and without sediment and insolubles, record the ultraviolet absorbance spectrum of the dilute solution using a 1-cm stoppered quartz cell against the solvent blank in a reference cell. Obtain all absorbance peaks for each material that may occur between 200 and 300 nm. By appropriate use of the various dilutions of each sample, the spectral peaks should be made to fall between 0.3 and 0.8 absorbance unit. In all cases, record the exact absorbance value for each of the peaks for both test and reference samples. For each peak at a specific wavelength, the concentrations used for both the test and reference samples should be identical. It is wise to prepare several replicate standard stock and dilute solutions for each concentration independently of each other in order to obtain an idea of the range in each peak absorbance of the standard sample that might be expected in this procedure, especially as a result of the preliminary removal of original solvent by heating and its possible oxidative effects.

21. Interpretation of Results

21.1 Compare the nature and shape of the ultraviolet absorption curves obtained for both the test and reference samples. Any significant difference is an indication of adulteration or nonuniformity. Also compare the absorbance value of each peak given by the test sample with that given by the comparison reference sample. Any significant difference should be an indication of adulteration or nonuniformity. It is difficult to fix precise criteria for spectral differences that apply to all materials. As a general guide, the following criteria may be useful for evaluating comparable absorbance peaks. Comparison peaks should be within 0.05 absorbance units when occurring between 220 and 350 nm, and within 0.08 at lower wavelengths.

22. Report

22.1 Indicate uniformity or lack of uniformity of the vehicle solids of the test sample with respect to the reference sample and the method or methods (A, B, C, and D) used for this judgment. If possible, attempt to report the nature of possible adulterant(s) present or the cause of the nonuniformity.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Solvent and Fuel Resistance of Traffic Paint¹

This standard is issued under the fixed designation D 2792; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a laboratory test for determining the resistance of a dried film of traffic paint to the action of a specified hydrocarbon solvent or gasoline fuel test fluid.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 471 Test Method for Rubber Property—Effect of Liquids²

2.2 Federal Standard:

TT-S-735 Standard Test Fluids for Hydrocarbons³

3. Summary of Method

3.1 The test paint is applied to a specified, properly prepared, tin panel. After aging, the coated panel is immersed in the hydrocarbon test fluid for a specified period of time at a specified temperature. The paint is examined for blistering, wrinkling, and loss of adhesion immediately upon removal and for complete hardness after a specified recovery time.

4. Significance and Use

4.1 Traffic paints must have good resistance to motor oil and fuel drippings on the highway. This test method describes the procedure necessary to measure the resistance of traffic paint to reference test fluids in order to simulate this type of action.

5. Apparatus

5.1 *Tin Panel*—Panels shall be cut from bright tin plate weighing not more than 25 g and not less than 19 g/dm² (0.51 to 0.39 lb/ft²). The panel should be about 75 by 130 mm (3 by 5 in.).

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved Oct. 3, 1969. Published December 1969.

² *Annual Book of ASTM Standards*, Vol 09.01.

³ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPQDS.

5.2 *Film Applicator*, which will produce a 3 or 6-mil (0.003 or 0.006-in.) (76 or 142- μ m) wet film thickness.

6. Reagents and Materials

6.1 *Test Liquid*—The testing liquid shall be specified by the purchaser and shall be selected from the following, dependent on the paint tested:

6.1.1 Type I of Federal Specification TT-S-735 which is the same as ASTM Reference Fuel A (ASTM Test Method D 471).

6.1.2 Type III of Federal Specification TT-S-735 which is the same as ASTM Reference Fuel B (Test Method D 471).

7. Procedure

7.1 Clean the tin panels thoroughly with a suitable solvent, and buff lightly with fine steel wool. Prepare at least 3 panels for each paint to be tested.

7.2 Examine the test paint, remove any skins that are present, stir with a paddle to loosen any settled pigment, and mix thoroughly.

7.3 Draw down the material on the panels using a film applicator that will produce a 3 or 6-mil (76 or 142- μ m) wet film as specified by the purchaser.

7.4 Air dry the coating for the time specified by the purchaser (usually 90 h) and then immerse the panels to half their length in the test liquid contained in a covered glass beaker at the temperature specified by the purchaser (generally 70 to 90°F (21 to 32°C)). Use a separate beaker for each paint tested.

7.5 At the end of the specified time (usually 4 to 18 h, depending on the type of paint) remove the panels and examine immediately for blistering, wrinkling, and loss of adhesion.

7.6 Allow the panels to dry at normal temperatures for the purchaser-specified time (generally 24 h), and examine for film defects and softening in comparison with the unimmersed portion of the panels.

8. Report

8.1 Report whether blistering, wrinkling, or loss of adhesion is evident immediately upon removal of panel from the test fluid as determined in 7.5.

8.2 Report whether any softening of the film is evident after the panel has recovered as determined in 7.6.

9. Precision

9.1 No statement of precision, in terms of percentages, can be made, although repeatability and reproducibility are exceptionally good.

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Standard Test Method for Block Resistance of Organic Coatings on Wood Substrates¹

This standard is issued under the fixed designation D 2793; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers the determination of the block resistance of organic coatings on wood and wood-based substrates. Block resistance is the ability of a coating to resist sticking to another surface and to resist any change in appearance, when it is pressed against that surface for a prolonged period of time.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Method

2.1 The coatings to be tested are applied to the desired substrate. They are cured in a manner duplicating the condition of end use as much as possible. Then a stack of these painted substrates is formed and subjected to a specified pressure for a sufficient time to develop any sticking tendencies that exist. The pressure is released and the painted surfaces are examined for any signs of sticking. If blocking (forming a block by panels sticking together) occurs, the material is unsatisfactory. If no sticking or damage to the film surface occurs, the material is satisfactory.

2.2 When the conditions of production finishing are established and known, the method of application, the substrate, film thickness, and cure of the film should duplicate those conditions as closely as possible.

2.3 When the conditions of storage and shipment are established and known, the temperature, relative humidity, duration, and pressure used for the test should duplicate those conditions as closely as possible.

3. Apparatus

3.1 *Rigid Platens*, 4-in. (100-mm) square, capable of being maintained at $120 \pm 2^\circ\text{F}$ ($49 \pm 1^\circ\text{C}$).

4. Test Specimens

4.1 For those cases where the intended use conditions are not established or known, the following specifications or some set of specifications agreed upon by the purchaser and seller apply: The test coating shall be applied by normal spray, using air, to a 4 by 4-in. (100 by 100-mm) panel. The panel shall be a piece of $\frac{1}{4}$ -in. (6.4-mm) SIS (smooth one

side) standard hardboard. The dry film thickness of pigmented coatings shall be 1.5 ± 0.2 mils ($38 \pm 5 \mu\text{m}$), and of clear coatings shall be 1.2 ± 0.2 mils ($30 \pm 5 \mu\text{m}$). Cure of the coating shall be as agreed and should be standardized for each coating.

NOTE 1—Film thickness can be measured by weight difference before and after painting, but this requires careful conditioning of the substrate under standard temperature and humidity conditions prior to each weighing. Film thickness can also be determined by using an aluminum tab alongside the test panel and measuring the film thickness with a standard eddy-current gage or using a steel panel with a magnetic film thickness gage.

5. Procedure

5.1 After 90 s cooling under ambient conditions or as agreed upon, make a stack of six panels and place under a uniform pressure of 35 ± 2 psi (240 ± 20 kPa) for Class I, 20 ± 1 psi (140 ± 10 kPa) for Class II, and $5 \pm \frac{1}{4}$ psi (35 ± 1.7 kPa) for Class III. These classes are arbitrary and simply designate three groups of coatings or possibly three groups of end uses. Stack the panels in the following order from the top to the bottom:

- (a) Face down.
- (b) Face down
- (c) Face up.
- (d) Face down.
- (e) Face down.
- (f) Face up.

This provides for two face-to-face contacts and two face-to-back contacts. Apply the load through rigid 4-in. square (100-mm square) pressure plates. Apply the load for a minimum of 24 h. Other pressures, types of pressure plates, and times may be used if agreed between the purchaser and seller. Conduct the test under ambient conditions but use press platens heated to $120 \pm 2^\circ\text{F}$ ($49 \pm 1^\circ\text{C}$). Slip sheets should not be used unless they are known to be used during storage of shipment.

NOTE 2—The most difficult portion of this test method to standardize on intelligently is the proper pressure to employ. Stacking pressure in production would be calculated as follows:

$$(DHA_1/A_2) \times F$$

where:

D = board density,
 H = height of stack,
 A_1 = area of stack,
 A_2 = area of support, and
 F = safety factor.

The area of support is difficult to establish. In some instances board is stacked smoothly on the floor while in other instances the stack is supported on 2 by 4 or 4 by 4 stringers which cut the support to very

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials.
Effective Oct. 3, 1969.

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small areas. It has been found that pressures of 20 to 50 psi (140 to 340 kPa) are suitable on hardboard substrates while 2 to 5 psi (14 to 35 kPa) are suitable on solid wood surfaces.

NOTE 3—Many coatings are plasticized if they are contaminated with water. If possible, therefore, it is advisable to run blocking tests at as high relative humidities as might be encountered in production of warehousing.

NOTE 4—Blocking tests shall be run with panels wet with water, if water quenching is used for cooling the boards and the boards are wet when stacked in production.

5.2 At the end of the test period remove the pressure and pick up the stack in such a way that first the bottom panel, then the next to the bottom panel, and eventually all of the panels are free to fall of their own weight. This falling free is considered as passing the test if there also is no change in the appearance of the coated surfaces of any of the panels.

6. Report

6.1 This is a go/no-go test for both face-to-face and face-to-back conditions. In addition to the separate statement of satisfactory or not satisfactory under both of these conditions, the nature of the failure should be reported. The report should also include the particulars of the test as follows:

- 6.1.1 Type of substrate,
- 6.1.2 Method of application,
- 6.1.3 Curing cycle,
- 6.1.4 Film thickness,
- 6.1.5 Temperature, humidity, and pressure of the blocking test,
- 6.1.6 Type of stacking including identification and type of slip sheets if any, and
- 6.1.7 Duration of test.

7. Precision

7.1 A round robin will be made to establish precision.

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Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)¹

This standard is issued under the fixed designation D 2794; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for rapidly deforming by impact a coating film and its substrate and for evaluating the effect of such deformation.

1.2 This test method should be restricted to testing in only one laboratory when numerical values are used because of the poor reproducibility of the method. Interlaboratory agreement is improved when ranking is used in place of numerical values.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 609 Practice for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

3. Terminology

3.1 Description of Term Specific to This Standard:

3.1.1 *impact resistance, of a coating*—the number of inch-pounds (kilogram-metres) required to produce cracking in the deformed coating.

4. Summary of Test Method

4.1 The organic coatings under test are applied to suitable thin metal panels. After the coatings have cured, a standard weight is dropped a distance so as to strike an indenter that deforms the coating and the substrate. The indentation can be either an intrusion or an extrusion. By gradually increasing the distance the weight drops, the point at which failure usually occurs can be determined. Films generally fail by cracking, which is made more visible by the use of a magnifier, by the application of a copper sulfate (CuSO_4)

solution on steel, or by the use of a pin hole detector.

5. Significance and Use

5.1 Coatings attached to substrates can exhibit cracking when subjected to rapid deformation, produced by impacts of objects during manufacturing and service. This test method has been very useful in evaluating attached coatings for their ability to resist cracking caused by impacts.

6. Apparatus

6.1 *Tester*, consisting of a vertical tube to guide a cylindrical weight that is dropped on a punch resting on the test panel.³

6.1.1 *Guide Tube*, 24 to 48 in. (0.6 to 1.2 m) long mounted vertically in a base plate. A slot is cut lengthwise on one side of the tube to act as a guide for a cylindrical weight that fits inside the tube. Graduations are marked in inch-pounds along the slot. The base is constructed so that a thin flat panel can be inserted at 2 in. (50 mm) below the tube.

6.1.2 *Weight*, metal cylinder, made to fit inside the guide tube. A pin is fitted into one side of the weight to act as a guide by riding in the slot of the tube and to serve as a handle by which the weight can be raised and released and serve as the indicator of inch-pounds (kilogram-metres).

6.2 *Indenter*—A steel punch with a hemispherical head having a diameter of either 0.500 in. (12.7 mm) or 0.625 in. (15.9 mm). The head rests on the test panel and the punch is held vertically by a guide ring.

6.3 *Panel Support*—A steel fixture with a 0.64-in. (16.3-mm) diameter cylindrical hole centered under the indenter for supporting the test panel.

6.4 *Magnifier*.

6.5 *Pin Hole Detector*.⁴

7. Reagents

7.1 An acidified copper sulfate (CuSO_4) solution prepared by dissolving 10 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 90 g of 1.0 N hydrochloric acid (HCl).

8. Test Specimens

8.1 Apply uniform coatings of the material to be tested to

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved July 27, 1990. Published November 1990. Originally published as D 2794 - 69. Last previous edition D 2794 - 84¹.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Suitable instruments are the Gardner-SPI Modified Impact Tester, available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910, or the Universal Impact Tester Model No. 172, available from Paul N. Gardner Co., Inc., 316 N.E. First St., PO Box 10688, Pompano Beach, FL 33061-6688. Equivalent apparatus may be used.

⁴ Suitable instruments are the Elcometer Pinhole Detector Model 104 and Zorelco Model 169 Pin Hole Detector, available from Zorelco Corp., PO Box 25500, Cleveland OH 44125, and the K-D Bird Dog Holiday Detector.

24-gage (0.001 in.) conversion of Practice D 609 minimum of four

NOTE 1—This test method is in accordance with Methods D 823 and D 1186. Other gage steel producer and the

8.2 Cure time and temperature user.

NOTE 2—This test method is in accordance with

9. Condition

9.1 Unless otherwise specified and the user, at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$). Conduct the test on removal of

10. Procedure

10.1 Install or agreed upon coated side of the test panel. Ensure the panel indenter is lightly placed on the tube so that weight up to failure will occur. indenter.

10.2 Remove the indenter and observe the test panel. If cracking is evident, creasing is observed, retest slightly above the first trial. Test one height at

10.3 Examine the following

10.3.1 Use

10.3.2 Heat

acidified copper

24-gage (0.025 in. or 0.63 mm) steel panels treated with a conversion coating in accordance with Procedure A of Practice D 609, unless otherwise specified. Prepare a minimum of four coated panels for the material.

NOTE 1—The coatings should be applied in accordance with Test Methods D 823, or as agreed upon between the producer and the user. Other gage steel panels may be used if agreed upon between the producer and the user.

8.2 Cure the coated panels under conditions of humidity and temperature agreed upon between the producer and the user.

NOTE 2—The thickness of the dry coatings should be measured in accordance with Test Methods D 1186.

9. Conditioning

9.1 Unless otherwise agreed upon between the producer and the user, condition the coated test panels for at least 24 h at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity. Conduct the test in the same environment or immediately on removal therefrom.

10. Procedure

10.1 Install the punch having the head diameter specified or agreed upon. Place the test panel in the apparatus with the coated side either up or down as specified or agreed upon. Be sure the panel is flat against the base support and that the indenter is in contact with the top surface of the panel. Lightly place the weight on the indenter and adjust the guide tube so that the lifting pin is at the zero mark. Raise the weight up the tube to a height where it is expected that no failure will occur. Release the weight so that it drops on the indenter.

10.2 Remove the test panel from the apparatus and observe the impact area for cracks in the coating. If no cracks are evident, repeat the procedure at a greater height, increasing 1 in. (25 mm) at a time. Once visible cracks are observed, repeat the test five times at each of three heights; slightly above, slightly below, and at that determined in the first trial. Test in a random fashion so that all impacts from one height are not made in succession or on one panel.

10.3 Examine the impacted areas for cracking by one of the following methods:

10.3.1 Use a magnifier to examine the area for cracks.

10.3.2 Hold a white flannel-type cloth saturated with the acidified copper sulfate (CuSO_4) solution (7.1) over the

impacted areas for at least 15 min. Remove the cloth and examine both the test areas and cloth for evidence of copper deposition or iron-rust staining respectively.

NOTE 3—The copper sulfate solution will not perform properly on zinc-phosphate-treated metal unless the conversion coating cracks.

10.3.3 To detect breaks in the film with a pin hole detector, first connect the ground lead from the instrument to the bare substrate and connect the instrument to an electrical power source. Moisten the probe sponge with tap water and slowly draw the probe over the impact area. The presence of cracks will be indicated by an audible alarm.

10.4 For each inch-pound (kilogram-metre) level, tabulate the number of times the coating passed or failed. The value where the results change from mainly passing to mainly failing is the impact failure end point.

11. Report

11.1 Report the following for each coating tested:

11.1.1 The inch-pounds (kilogram-metres) at the impact failure end point,

11.1.2 Whether intrusion or extrusion was used,

11.1.3 Diameter of the punch used,

11.1.4 Thickness of coating,

11.1.5 Substrate thickness and type of metal,

11.1.6 Method of panel preparation, and

11.1.7 Atmospheric conditions under which the coated panels were conditioned and tested.

NOTE 4—Because of the poor reproducibility of this method, the reporting of inch-pounds (kilogram-metres) in comparing coatings for impact resistance should be restricted to one laboratory. For interlaboratory comparisons, rankings of coatings for impact resistance should be reported.

12. Precision

12.1 On the basis of an interlaboratory test in which operators in six laboratories tested three paints having a broad range of impact resistance on two metal substrates, the between-laboratories coefficients of variation were found to be as follows:

	Coefficient of Variation	
	Intrusion, %	Extrusion, %
Brittle coating (less than 6 in.-lb)	25	100
Average coating (between 6 and 140 in.-lb)	80	100
Flexible coating (more than 140 in.-lb) (0.625 in.-diameter punch)	10	25

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Method for Leveling Characteristics of Paints by Draw-Down Method

This method covers the laboratory determination of the relative leveling characteristics of liquid water-reducible or solvent-reducible coatings in white and light tints.

Formerly under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials, this test method was discontinued in 1990.

1. Scope

1.1 This method covers the susceptibility of the coating to leveling.

1.2 This method covers whether the coating will level after application.

1.3 This method covers the leveling characteristics of the coating, and addresses all the responses appropriate to the application of the coating.

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Standard Test Method for Filiform Corrosion Resistance of Organic Coatings on Metal¹

This standard is issued under the fixed designation D 2803; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers two procedures for determining the susceptibility of organic-coated metal substrates to formation of filiform corrosion.

1.2 This method is limited to the determination of whether filiform corrosion will occur between the organic coating and substrate.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
 B 117 Method of Salt Spray (Fog) Testing²
 D 609 Methods for Preparation of Steel Panels For Testing Paint, Varnish, Lacquer, and Related Products³
 D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³
 D 1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers³
 D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base³
 D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base³
 D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments³
 D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴
 E 104 Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions⁵
 2.2 *International Standard:*
 ISO 4623 Paint and Varnishes—Filiform Corrosion Test on Steel⁶

3. Summary of Method

3.1 Coated metal specimens are scribed and placed in a

corrosive atmosphere to initiate corrosion. The specimens are then exposed to controlled temperature and humidity conditions known to be conducive to filiform corrosion.

3.2 In Procedure A, panels are subjected to a preliminary exposure in the salt spray cabinet to initiate corrosion, rinsed, and placed in a humidity cabinet. In Procedure B, based on ISO 4623, panels are either exposed to salt spray or dipped in a salt solution but not rinsed before placing in the humidity cabinet operated at a higher temperature than in Procedure A.

4. Significance and Use

4.1 Some organic coatings applied to metal substrates exhibit filiform corrosion when there is a break in the coating film and the relative humidity is in the 70 to 95 % range.

4.2 This method can be used to determine the susceptibility of organic coated metal substrates to the formation of filiform corrosion.

5. Definitions

5.1 *filiform corrosion*—a special type of corrosion that occurs under coatings on metal substrates that is characterized by a definite threadlike structure and directional growth. Filiform corrosion usually occurs between 70 and 95°F (20 and 35°C), with a corresponding relative humidity range of 60 to 95 %; above 95 % humidity, blistering rather than filiform corrosion may occur.

6. Apparatus

6.1 *Salt-Spray Chamber* as described in Method B 117, for salt-spray (fog) testing.

NOTE 1—The preferred initiator of corrosion at the scribe is the salt-fog atmosphere described in Method B 117. Exposure of the specimens for 2 to 4 h in a closed cabinet to the atmosphere above a 1 % acetic acid solution is an acceptable alternative.

6.2 *Humidity Cabinet*—Any cabinet with suitable temperature and humidity controls with air circulation may be used. The size and detailed construction of the apparatus are optional, provided the conditions meet the requirements of this method.

7. Test Specimen

7.1 The composition, surface preparation, and number of test specimens shall be agreed upon between the producer and the user. Steel is the preferred substrate, but other metals such as aluminum, copper, and stainless steel may be used. Zinc and zinc-coated steel are not recommended because filiform corrosion generally does not occur on zinc.

NOTE 2—Applicable test panel description and surface preparation methods are given in Method D 607 and Practices D 1730.

7.2 *Preparation of Test Specimens*—The method of application, film thickness, curing, and conditioning shall be agreed upon between the producer and user.

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Tests for Protective Coatings.

Current edition approved June 25, 1982. Published September 1982. Originally published as D 2803 - 69 T. Last previous edition D 2803 - 70 (1974).

² *Annual Book of ASTM Standards*, Vols 03.02 and 06.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vols 02.05 and 06.01.

⁵ *Annual Book of ASTM Standards*, Vols 08.03, 10.01, and 11.03.

⁶ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

NOTE 3—Test Methods D 823 gives application methods that can be used to produce films of uniform film thickness.

NOTE 4—Test Methods D 1005, D 1186, and D 1400 give procedures that can be used for measurement of dry film thickness.

7.3 Scribe the test specimen in accordance with Method D 1654 or as agreed upon between the producer and user.

7.4 The back and edges of the panel should be protected with an anti-corrosion coating or tape, unless edge failure is being evaluated.

8. Procedure A

8.1 Expose the test specimens to the salt-fog atmosphere in accordance with Method B 117 for at least 4 h and not more than 24 h, or as agreed upon between the producer and user.

8.2 Remove from the salt-fog cabinet and thoroughly rinse with distilled or demineralized water. Do not permit specimens to dry before placing in the humidity cabinet.

8.3 Place the specimens in the humidity cabinet so they are no closer together than 1.5 in. (40 mm) and not in contact with any metal. Operate the cabinet at $77 \pm 3^\circ\text{F}$ ($25 \pm 2^\circ\text{C}$) and $85 \pm 2\%$ relative humidity unless otherwise agreed upon between the producer and user.

NOTE 5—The recommended means of maintaining the specified relative humidity is to utilize trays of saturated potassium chromate solution (650 g/L). Recommended Practice E 104 describes other methods for maintaining relative humidity, but they may affect the degree and rate of filiform corrosion.

8.4 Inspect the specimens at intervals of approximately 168 h for development of threadlike filaments from the scribe. Exercise care to maintain wetness of specimens during inspection, since drying stops the original pattern of filiform corrosion. New filiform may develop on continued exposure but at different points.

8.4.1 Filiform corrosion will be noted as threadlike filaments (see Note 6), initiating at the scribe. A standard method of rating failure due to filiform is not available. The uniformity of filament growth in direction, width, height and frequency precludes a precise rating system. Photographs of filiform failure are preferred for recording test results. Otherwise, a brief description of the filament growth and their frequency can be made.

NOTE 6—The nature of grinding and grain direction on panels can influence the direction of filiform growth.

8.5 The normal test period is 6 weeks (1008 h). Other periods may be used at the option of the producer and user.

9. Procedure (ISO 4623)

9.1 Two alternative initiations of corrosion are available, based on initiation by dipping in sodium chloride solution (see Note 7) or exposure to salt fog. The dipping technique is

generally preferable for air drying and low-durability materials. For high-durability systems such as automotive finishes applied to phosphated steel, the dipping technique may not produce filiform corrosion and for these systems, it is preferable to adopt the salt-fog technique. In such cases, the necessary period of exposure to salt fog depends on the durability of the material under test but should rarely need to exceed 24 h.

NOTE 7—Sodium chloride 0.1 % (w/v) solution (for dipping technique) is prepared by dissolving 1 g of reagent grade sodium chloride (NaCl) in 1 L of distilled or demineralized water. Place this solution in a vessel suitable for complete immersion of the test panel.

9.2 Dipping Technique:

9.2.1 Immerse the scribed specimen for 30 to 60 s in the sodium chloride solution (see Note 7).

9.2.2 Take the specimen out of the solution and remove any drops of liquid adhering to it, taking care not to remove the solution from the scribed marks.

9.2.3 Place the specimen in a humidity cabinet maintained at $104 \pm 3.5^\circ\text{F}$ ($40 \pm 2^\circ\text{C}$) and $80 \pm 5\%$ relative humidity.

9.2.4 Repeat the immersion procedure every 3 or 4 days until the end of the test cycle (21 days) or as agreed upon between the producer and the user.

9.3 Salt-Fog Technique:

9.3.1 Expose the scribed test specimen to the salt-fog atmosphere in accordance with Method B 117 for at least 4 h or not more than 24 h or as agreed upon between the producer and the user.

9.3.2 Remove the test specimen from the salt-fog cabinet and remove any drops of liquid adhering to it, taking care not to remove the solution from the scribed marks.

9.3.3 Place the specimen in a humidity cabinet maintained at $104 \pm 3.5^\circ\text{F}$ ($40 \pm 2^\circ\text{C}$) and $80 \pm 5\%$ relative humidity for the specified period agreed upon between the producer and the user.

9.4 Follow the procedures in 8.4 and 8.4.1.

10. Report

10.1 The report shall include the following:

- 10.1.1 Initiation procedure,
- 10.1.2 Humidity cabinet operating temperature,
- 10.1.3 Exposure period, and
- 10.1.4 Rating of coatings as described in 8.4.1.

11. Precision

11.1 *Procedure A*—Twelve laboratories, using three types of corrosion initiations, three coatings and three substrates, were in excellent agreement in their ranking of the susceptibility to filiform corrosion by this procedure.

11.2 *Procedure B*—Has been judged reproducible by Working Group 2 of ISO/TC35/SC9.

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Standard Test Method for Hiding Power of Paints by Reflectometry¹

This standard is issued under the fixed designation D 2805; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

INTRODUCTION

Using equations derived from Kubelka-Munk turbid media theory (1-4)², the reflectance of a coating can be predicted for any film thickness from measurements made at only one. On this basis several easy and accurate test methods (5,7) have been developed for determining hiding power. In the past such test methods have been considered difficult due to complexities, apparent and actual, in the treatment of data. The present test method has been simplified in this respect, primarily by adapting it fully for computer calculations.

Although the use of broad-band reflectometry makes this test method theoretically valid only for nonchromatic (white or gray) colors, good agreement has been obtained with chromatic paints as well. This is undoubtedly because the experimental measurements are made fairly close to the hiding power end point so that the Kubelka-Munk extrapolation and thus any associated error is relatively small.

This test method is therefore recommended without restriction as to color.

1. Scope

1.1 This test method covers the determination, without reference to a material paint standard, of the hiding power of air dry coatings with Y tristimulus values greater than 15 %. With appropriate modification, it can also be used to test baking finishes.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts³

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products³

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates³

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials³

E 97 Test Method for Directional Reflectance Factor, 45-deg, 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁴

E 179 Practice for Selection of Geometric Conditions for Measurement of Reflectance and Transmission Properties of Materials⁵

E 284 Definitions of Terms Relating to Appearance of Materials⁵

3. Terminology

3.1 *Definitions*—For definitions used in this test method, see Definitions E 284.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *contrast ratio*—the ratio of the reflectance of a film on a black substrate to that of an identical film on a white substrate.

3.2.1.1 C_W —the contrast ratio with a white substrate of reflectance W .

$$\text{Thus: } C_W = R_0/R_W$$

3.2.1.2 C —the contrast ratio with a white substrate for which $W = 0.80$.

$$\text{Thus: } C = R_0/R_{0.80}$$

3.2.2 *reflectance*—the daylight luminous diffuse reflectance factor (specular reflection excluded). Also referred to in this test method as the Y-tristimulus value. This value may be expressed as a percent or a decimal fraction, the latter being preferred and usually required for mathematical calculations.

3.2.2.1 *reflectivity*, R_∞ —the reflectance of film thick enough to have the same reflectance over both a black and a white substrate.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved March 25, 1988. Published May 1988. Originally published as D 2805 - 69 T. Last previous edition D 2805 - 85.

² The boldface numbers in parentheses refer to the list of references at the end of this standard.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

3.2.2.2 R_0 —the reflectance of a film on a black surface with a reflectance of 1 % or less, which is effectively zero for the purpose of this test.

3.2.2.3 W —the reflectance of a white substrate.

3.2.2.4 R_w —the reflectance of a film applied on a white substrate of reflectance W .

3.2.2.5 $R_{0.80}$ —the reflectance of a film applied on a substrate having a reflectance of 80 %, which is the standard white-substrate reflectance in paint technology.

3.2.3 *scattering coefficient, S* —the ability of a material to internally scatter and thereby reflect light; expressed in this test method in the same units as spreading rate.

3.2.4 *spreading rate, H* —film area per unit volume of coating, in this test method expressed in square metres per litre (m^2/L). It is also frequently expressed in square feet per gallon (ft^2/gal).

3.2.4.1 *spreading rate, H_x* —an experimentally determined value of H .

3.2.4.2 *spreading rate, H_C* —value of H at a specified contrast ratio C .

3.2.4.3 *hiding power, $H_{0.98}$* —the spreading rate at the contrast ratio $C = 0.98$.

NOTE 1—It should be emphasized that this contrast ratio does not represent visually complete hiding, nor does it indicate that the same contrast ratio holds at every wavelength.

4. Summary of Test Method

4.1 The reflectivity R_∞ of the coating is determined from reflectance measurements on black and white hiding power charts.

4.2 The scattering coefficient S of the coating is determined from R_∞ and the reflectance R_0 and spreading rate H_x of a film applied on black glass.

4.3 The hiding power, $H_{0.98}$ of the coating is calculated from the reflectivity R_∞ and the scattering coefficient S .

4.4 As an optional procedure the contrast ratio C at a specified spreading rate H_C is calculated from R_∞ and S .

5. Significance and Use

5.1 This is a precise instrumental method giving results having an absolute physical significance without reference to a comparison paint. It should be used when maximum precision and minimum subjectivity are required, as in testing specification coatings or evaluating the hiding efficiency of pigments.

5.2 Hiding power Test Method D 344 is visual instead of instrumental, and gives results that are relative to a material standard instead of absolute. It is less precise than Test Method D 2805 but more closely aligned with practical painting procedures.

6. Apparatus and Materials

6.1 Substrates:

6.1.1 *Black Glass Panels*,⁶ minimum size 200 by 200 mm, and approximately 13 mm thick.

⁶ Black Vitrolite and Carrara structural glasses were used originally and may still be obtainable from some local glaziers but, in general, have been replaced with black float glass. If this is not available from local glaziers it may be purchased from The Leneta Co., Box 86, Ho-Ho-Kus, NJ 07423.

6.1.2 *Black and White Paper Charts*⁷—The surface shall be smooth and level, and impervious to paint liquids. The black area shall have a maximum reflectance of 1 % and the white area a minimum reflectance of 76 %.

6.2 *Balance*, accurate to 0.1 mg.

6.3 *Glass Slides*⁸—Round or square plates of thickness similar to that used for microscope specimen slides, with a minimum area of 40 cm^2 .

6.4 *Reflectance-Measuring Instrument*—One that allows only diffusely reflected, radiant flux to be incident upon the measuring element. It shall employ a photometric system, including source, filters, and receptor, that provides a response closely similar to the product of the spectral luminous efficiency function of the CIE standard observer and source C . It shall provide readings to at least the third decimal place and permit estimation to the fourth.

6.5 *Template*,⁹ with a film area approximately 100 cm^2 determined to the nearest tenth. Record the exact value on Line C of the worksheet shown in Fig. 1.

6.6 *Doctor Blade Film Applicators*, width 150 mm, clearances 50, 75, 100, 125, 150, 175, and 200 μm .

NOTE 2—With doctor blades made in the United States, estimate 25 mm/in. and 25 $\mu m/mil$. Bird-type applicators are usually marked with their half clearance.

6.7 *Computer and Software*,¹⁰ for solving the relevant Kubelka-Munk equations.

7. Procedure

7.1 General Instructions:

7.1.1 *Film Application*—Drawdowns should be made manually with a smooth uniform motion, at the rate of about 6 cm/s. Paper charts should be held flat by a vacuum plate or other suitable device while making drawdowns.

7.1.2 *Reflectance Measurements*—Measure the reflectance of each test area at a minimum of three locations, reading or estimating to four decimal places and calculating mean values to the same. Charts should be placed over a white surface and black glass over a black surface while measurements are being made.

7.1.3 *Record Keeping*—All data should be recorded on a copy of the worksheet form specified in Fig. 1. Typical data entries are shown in Fig. 2.

7.1.4 *Weight Measurements*—Make all weighings to 0.1 mg on the analytical balance.

7.2 *Paint density, D* —Determine the density in g/mL to four decimal places in accordance with Test Method D 1475 and record on Line A of the worksheet (Fig. 1). If the density is first determined in pounds per gallon, divide by 8.3454 to obtain it in grams per millilitre.

7.3 *Nonvolatile Content, N* —Sandwich 0.3 mL of paint (dispensed with a 1-mL syringe) between two previously

⁷ Leneta Form 3B, available from The Leneta Co., has been found satisfactory for this purpose. An equivalent may be used.

⁸ If glass slides of this type are not readily available, use clear or translucent plastic slides instead. Leneta Form P420NV, available from the Leneta Co., have been found satisfactory for this purpose.

⁹ A template available from Precision Gage and Tool Co., 28 Volkenand Ave., Dayton, OH 45410, has been found suitable for this purpose. An equivalent may be used.

¹⁰ Most computers can be programmed with the relevant equations. A small pre-programmed computer is available from The Leneta Co.

- A. Paint Density, D : _____ g/mL
 B. Nonvolatile Content, N : _____ (decimal fraction)
 C. Template Film Area, A : _____ cm²
 D. Drawdowns on Charts:

Chart	R_0	R_w	W	C_w	R_{∞}
1					
2					
3					
4					
Mean	—	—	—		

$$C_w = R_0/R_w \quad R_{\infty} = f(R_0, R_w, W) \quad \text{Eq A1.1 of Annex A1}$$

- E. Drawdowns on Glass:

Panel	R_0	M , g	H_x , m ² /L	S , m ² /L	$H_{0.98}$, m ² /L
1					
2					
3					
4					
Mean	—	—	—		

$$H_x = \frac{AND}{10M} = \bar{M} \quad S = f(R_0, R_{\infty}, H_x) \quad \text{Eq A1.2 of Annex A1}$$

$$H_{0.98} = f(S, C, R_{\infty}) \text{ where } C = 0.98 \quad \text{Eq A1.3 of Annex A1}$$

- G. Report

- (1) Hiding power $H_{0.98}$ _____ m²/L _____ ft²/gal
 (2) Reflectivity, R_{∞} _____
 (3) Scattering coefficient, S _____ m²/L
 (4) Applicator clearance _____ μm _____ mils
 (5) Contrast ratio, C_w _____
 (6) Sample identification
 Type of paint _____ Color _____
 Formula No. _____ Batch or sample No. _____
 (7) Reflectometer description
 Manufacturer _____ Model name _____
 Model No. _____ Type _____
 Geometry _____ Aperture _____

- H. Alternative Hiding Power Report:

- Specified spreading rate, H _____ m²/L _____ ft²/gal
 Contrast ratio, C _____

FIG. 1 Work Sheet Form

weighed slides, squeezing them together so that the paint spreads to a diameter of about 45 mm. Reweigh to obtain the weight of paint sample. Separate the slides, allow the films to dry, and weigh a third time to determine the dry weight. Then calculate the nonvolatile content of the paint as a decimal fraction to four places. Run in duplicate and report the mean value to three decimal places on Line B of the worksheet (Fig. 1).

7.4 *Applicator Selection*—Make trial drawdowns on black and white charts at clearances of 100, 150, and 200 μm. Allow them to dry overnight and measure their contrast ratios. Based on the results select the best applicator for a contrast ratio of 0.97 ± 0.01 , which may be one of the trial applicators or one of intermediate clearance. If it seems necessary, make an additional trial drawdown to confirm the correct applicator.

7.5 *Drawdowns on Glass and Charts*—Using the applicator selected from 7.4, make four drawdowns each on black glass and on black and white charts in accordance with 7.1.1.

Leave enough of the white area of each chart uncoated to permit measurement of the reflectance W .

7.6 *Drying of the Films*—Immediately after application place each drawdown horizontally in a well-ventilated dust-free location, with all drawdowns in close proximity to one another to assure identical drying conditions, and allow to dry a minimum of 40 h before testing.

7.7 *Reflectance Measurements*—On the second or third day after application measure the reflectances on all of the drawdowns within as brief a time span as possible, alternating the measurements between charts and glass rather than doing first all one and then the other. Enter the values of R_0 , R_w , and W for charts into Section D of the worksheet, and the values of R_0 for glass into Section E.

7.8 *Weight of Dry Film, M* —After all of the reflectances are measured, position the template on the glass panels, each in its turn, and scrape off the surplus paint with a razor blade, to leave film areas defined by the template. If the template area is not already specified, measure one of the

- A. Paint Density, D : 1.246 g/mL
 B. Nonvolatile Content, N : 0.499 (decimal fraction)
 C. Template Film Area, A : 100.2 cm²
 D. Drawdowns on Charts:

Chart	R_0	R_w	W	C_w	R_∞
1	0.8755	0.9118	0.8157	0.9602	0.9452
2	0.8723	0.9112	0.8158	0.9573	0.9473
3	0.8758	0.9117	0.8123	0.9606	0.9463
4	0.8768	0.9117	0.8123	0.9617	0.9449
Mean	—	—	—	0.9600	0.9459

$$C_w = R_0/R_w \quad R_\infty = f(R_0, R_w, W) \quad \text{Eq A1.1 of Annex A1}$$

- E. Drawdowns on Glass:

Panel	R_0	M , g	H_x , m ² /L	S , m ² /L	$H_{0.98}$, m ² /L
1	0.8760	0.4803	12.97	98.04	9.180
2	0.8785	0.4980	12.51	97.09	9.092
3	0.8793	0.4921	12.66	99.11	9.281
4	0.8798	0.5082	12.26	96.50	9.036
Mean	—	—	—	96.72	9.147

$$H_x = \frac{AND}{10M} = \frac{M}{M} \quad S = f(R_0, R_\infty, H_x) \quad \text{Eq A1.2 of Annex A1}$$

$$H_{0.98} = f(S, C, R_\infty) \text{ where } C = 0.98 \quad \text{Eq A1.3 of Annex A1}$$

- G. Report

- (1) Hiding power $H_{0.98}$ 9.15 m²/L 373 ft²/gal
 (2) Reflectivity, R_w 0.946
 (3) Scattering coefficient, S 96.7 m²/L
 (4) Applicator clearance 125 μm 5 mils
 (5) Contrast ratio, C_w 0.96
 (6) Sample identification
 Type of paint Exterior Latex Gloss Color White
 Formula No. SF1436 Batch or sample No. 875-1234
 (7) Reflectometer description
 Manufacturer Photovolt Model name
 Model No. 670 Type Colorimeter
 Geometry 45-deg 0-deg Aperture 20 mm

- H. Alternative Hiding Power Report:

- Specified spreading rate, H 15.46 m²/L 630 ft²/gal
 Contrast ratio, C 0.949

FIG. 2 Typical Work Sheet

resultant films carefully to determine its area in square centimeters to the nearest tenth. Record this value on Line C of the worksheet (Fig. 1) as the characteristic area of the template. Then scrape off each paint film carefully into a weighing dish, weight to 0.1 mg, and record the weights M in Section E of the worksheet, each value entered adjacent to the corresponding value of R_0 for the same glass panel.

8. Calculation

8.1 In general, calculations should be made and recorded to four significant figures and final report values to three. Enter calculated results in the worksheet (Fig. 1). Typical entries are shown in Fig. 2.

8.2 Contrast Ratio C_w and Reflectivity R_∞ —From the data entered in Section D of the worksheet calculate C_w and R_∞ for each of the four charts, thus:

$$C_w = R_0/R_w \quad R_\infty = f(R_0, R_w, W) \quad (\text{A1.1})$$

Enter the values, as they are calculated, in the appropriate columns of Section D, then calculate and likewise enter their means.

8.3 Spreading Rate H_x —From the data recorded in the worksheet on Lines A, B, and C and in Section E, calculate the spreading rate $H_{0.98}$ for each of the four glass panels, thus:

$$H_x = \frac{A \times N \times D}{10M}$$

where:

- H_x = spreading rate, m²/L,
 A = template area, cm²,
 N = nonvolatile content of the paint,
 D = density of the paint, g/mL, and
 M = dry film weight, g.

and enter the values in the appropriate column of Section E.
 8.4 Scattering Coefficient S and Hiding Power $H_{0.98}$ —Continuing on Section E of the worksheet (see Fig. 1), and

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using the mean value of R_{∞} from Section D, calculate for each of the glass panels, first S and then $H_{0.98}$ in square millimetres per litre as follows:

$$S = f(R_0, R_{\infty}, H_X) \quad (A1.2)$$

$$H_{0.98} = f(S, C, R_{\infty}) \text{ given } C = 0.98 \quad (A1.3)$$

Enter the values as they are calculated in the appropriate columns of Section E, then calculate and likewise enter their means.

8.5 *Contrast Ratio C at a Specified Spreading Rate H*—If desired, calculate the contrast ratio at a specified spreading rate using the mean values of R_{∞} and S from worktable Sections D and E, of the worksheet (Fig. 1), thus:

$$C = f(S, H, R_{\infty}) \quad (A1.4)$$

9. Report

9.1 Enter the following information on the indicated line in Fig. 1, Section G:

9.1.1 *Hiding Power, $H_{0.98}$* —Enter the mean value of $H_{0.98}$ from Section E, rounded to the second decimal place, onto Line G(1). If desired, multiply this value by 40.746 to obtain the value in square feet per gallon to the nearest whole number and enter it on the same line.

9.1.2 *Reflectivity, R_{∞}* —Enter the mean value of R_{∞} from Section D, rounded to three significant figures, onto Line G(2).

9.1.3 *Scattering Coefficient, S* —Enter the mean value of S from Section E, rounded to three significant figures, onto Line G(3).

9.1.4 *Applicator Clearance*—On Line G(4) enter the clearance of the applicator, expressed in μm and if desired also in mils.

9.1.5 *Contrast Ratio, C_W* —Enter the mean value of C_W from Section D, rounded to the third decimal place, onto Line G(5).

9.1.6 On Line G(6) enter the identification and brief description of the coating.

9.1.7 On Line G(7) enter the identification and description of the reflectometer employed.

9.2 *Alternative Hiding Power Report: Contrast Ratio at a Specified Spreading Rate*—Enter the specified spreading rate H and the value of C calculated from 8.5, into Section H of the worksheet. This report may be in addition to or instead of conventional hiding power $H_{0.98}$.

10. Precision

10.1 *Precision*—The precision values given for this test method are based on two series of interlaboratory tests. For gray and chromatic paints (6), five operators in five laboratories tested four gloss enamels covering a broad range of colors. For white paints (5), five operators in five laboratories tested three white finishes covering the range of gloss to flat. The between-laboratory coefficients of variation were found to be those shown in Table 1.

11. Index Terms

11.1 This test method is indexed under the following terms: hiding power (of paints/coatings); spreading rate; contrast ratio; scattering coefficient.

TABLE 1 Precision of Hiding Power, $H_{0.98}$

	Coefficient of Variation	Maximum Acceptable Differences for Two Results	
		Repeatability	Reproducibility
Gray, and chromatic gloss finishes, %	3.5	<10.4	10.4
White finishes:			
Gloss, %	1.2	<4.7	4.7
Semigloss and flat, %	5.0	<16.3	16.3

ANNEX

(Mandatory Information)

ANNEX A1. KUBELKA-MUNK EQUATIONS USED IN THIS TEST METHOD

$$R_{\infty} = f(R_0, R_W, W) \quad (A1.1)$$

$$a = \frac{1}{2} \left(R_W + \frac{R_0 + W - R_W}{WR_0} \right) \quad (A1.1.1)$$

$$R_{\infty} = f(a) = a - (a^2 - 1)^{1/2} \quad (A1.1.2)$$

$$S = f(R_0, R_{\infty}, H_X) \quad (A1.2)$$

$$b = f(R_{\infty}) = \frac{1}{2} \left(\frac{1}{R_{\infty}} - R_{\infty} \right) \quad (A1.2.1)$$

$$S = \frac{H_X}{2b} \ln \left(\frac{1 - R_0 R_{\infty}}{1 - R_0 / R_{\infty}} \right) \quad (A1.2.2)$$

$$H_C = f(S, C, R_{\infty}) \quad (A1.3)$$

$$a = f(R_{\infty}) = \frac{1}{2} \left(\frac{1}{R_{\infty}} + R_{\infty} \right) \quad (A1.3.1)$$

$$b = f(R_{\infty}) = \frac{1}{2} \left(\frac{1}{R_{\infty}} - R_{\infty} \right) \quad (A1.3.2)$$

$$U = \left[\left(a + \frac{1-C}{1.60 C} \right)^2 - \frac{1}{C} \right]^{1/2} + \frac{1-C}{1.60 C} \quad (A1.3.3)$$

$$P = f(U, R_{\infty}) = \frac{1}{2b} \ln \left(\frac{U+b}{U-b} \right) \quad (A1.3.4)$$

$$H = S/P \quad (A1.3.5)$$

$$C = f(S, H, R_{\infty}) \quad (A1.4)$$

$$P = S/H \quad (A1.4.1)$$

$$a = f(R_{\infty}) = \frac{1}{2} \left(\frac{1}{R_{\infty}} + R_{\infty} \right) \quad (A1.4.2)$$

$$b = f(R_{\infty}) = \frac{1}{2} \left(\frac{1}{R_{\infty}} - R_{\infty} \right) \quad (A1.4.3)$$

$$C = \frac{a + b \coth bP - 0.80}{(a + b \coth bP) [1 - 0.80(a - b \coth bP)]} \quad (A1.4.4)$$

REFERENCES

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- (5) Jacobsen, A. E., Mitton, P. B., and Switzer, M. H., "Determination of the Hiding Power of Nonchromatic Paints," *Materials Research and Standards*, Vol 2, No. 12, 1962, pp. 1004-1005.
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- (7) Mitton, P. B., "Easy Quantitative Hiding Power Measurements," *Journal of Paint Technology*, Vol 42, No. 542, 1970, pp. 159-183.

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Standard Test Method for Durability and Compatibility of Factory-Primed Wood Products with Representative Finish Coats¹

This standard is issued under the fixed designation D 2830; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides for the determination of the relative durability and compatibility of factory-primed wood and wood-based substrates with representative finish coats when exposed to the weather.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 659 Method of Evaluating Degree of Chalking of Exterior Paints²
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints²
- D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood²
- D 3274 Test Method of Evaluating Degree of Surface Disfigurement of Paint Films by Microbial (Fungal or Algal) Growth or Soil and Dirt Accumulation²
- D 3359 Test Methods for Measuring Adhesion by Tape Test²

2.2 U. S. Federal Standards:³

- TT-P-96b
- TT-P-102

3. Summary of Test Method

3.1 This test method consists of the atmospheric exposure of at least three types of conventional exterior white house paints applied to panels of factory-primed wood or wood-base substrates that have been exposed to atmospheric

weathering conditions for periods of 0, 3, and 6 months.

3.2 The finish coats are applied by brush.

3.3 An additional panel of original factory-primed substrate, without finish coat, is exposed for the duration of the test.

4. Significance and Use

4.1 This test method is useful for evaluating the weathering performance of factory-applied primers on wood or wood-based substrates overcoated with a field-applied topcoat.

4.2 This test method is useful for evaluating the weathering compatibility of commercial topcoats over factory-applied primers on wood or wood-based substrates.

5. Apparatus

5.1 A vertical (90° from horizontal) test fence constructed in accordance with requirements of Practice D 1006.

6. Materials

6.1 Exterior Finishes:

6.1.1 *Paint, Oil, Alkyd*, fume resistant, conforming to Federal Specification TT-P-102.

6.1.2 *Resin Latex Paint, Synthetic*, of the emulsion type designed for use on exterior wood surfaces (such as TT-P-96b).

6.1.3 *Flat Alkyd, Solvent-Thinned, Exterior*.

6.1.4 Any other type of exterior finish paint mutually agreed upon.

6.2 *Bristle Brush (Natural or Synthetic)*.

7. Procedure

7.1 The minimum length of panel is 3 ft (910 mm) with a minimum width of 6 in. (150 mm) of a particular preprimed substrate.

7.2 Mark off one third of the panel area (in a manner which will not damage the prime coat) and apply by brush to the right one third panel area a single coat of one of the white finishes at a spreading rate as recommended by the manufacturer or by the appropriate Federal specification. Paint the panel and dry indoors for 7 days under prevailing conditions of temperature and humidity, taking care to avoid exposure to drafts or excessive heat.

7.3 Determine the spreading rate of the finish paints by the weight difference method or other suitable methods. Record the spreading rate, film thickness, and method used.

7.4 Fasten the panel to a south exposure rack.

NOTE 1—Panel mounting should be the same as normal practice for the mounting of a particular substrate.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.52 on Factory-Coated Wood Building Products.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 2830 - 69. Last previous edition D 2830 - 82 (1987).

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

7.5 After 3 months (± 1 week) of exposure of the panel, coat the center one third section adjacent to the section already coated with exterior finish as specified in 7.2.

NOTE 2—If possible, the application should be made in the laboratory as in 6.2. No washing or cleaning should be done to the surface to be coated.

7.6 At completion of 6 months (± 1 week) of exposure to the weather, apply finish coat to the final third section of the panel as specified in 7.2.

7.7 Examine each section of the panels visually for appearance, mildew, and film failure at intervals of not more than 3 months (preferably 1 month) beginning 1 month after initial exposure.

7.8 At the end of each 6 months exposure of a topcoated section, test for intercoat adhesion between the factory-applied primer and the topcoat as follows:

7.8.1 Cut a small "X" in the film with a sharp knife or razor blade. The cuts should penetrate the substrate slightly but not distort the film unduly. Apply pressure-sensitive tape⁴ so that it crosses the "X" in the direction of the acute angles. Press the tape on carefully with the flat surface of the

finger nail or with a rubber eraser. Remove the tape with a quick yank, at a 90° angle to the surface.

7.9 Exposure of the topcoated preprimed substrate may be terminated 12 months after application of the finish coat to the third section. Additional exposure may be agreed upon between buyer and seller.

8. Report

8.1 Report the following properties:

8.1.1 Chalking—Method D 659.

8.1.2 Checking—Test Method D 660.

8.1.3 Cracking—Test Method D 661.

8.1.4 Erosion—Test Method D 662.

8.1.5 Flaking—Test Method D 772.

8.1.6 Blistering—Test Method D 714.

8.1.7 Intercoat Adhesion—Test Methods D 3359.

8.1.8 Mildew—Test Method D 3274.

9. Precision and Bias

9.1 Varying weather conditions from year to year and at several exposure sites preclude development of precision and bias data.

10. Keywords

10.1 adhesion; factory primed wood; primer performance; topcoat compatibility; weathering

⁴ 3-M Company's No. 600 Cellophane Tape has been found satisfactory. Tape shall be stored in an airtight container and shall not be more than 6 months old.

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Standard Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings¹

This standard is issued under the fixed designation D 2832; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Keywords were added editorially in June 1991.

1. Scope

1.1 This guide is intended to aid in the selection of the proper ASTM standard for determining the volatile and nonvolatile content of paint and related coatings.

NOTE—Test methods for determining the composition of the volatile fraction are not covered by this guide.

1.2 The standards included are as follows:

Type of Coating	Section	ASTM ² Designation
Aerosol coatings	4.1	D 3062
Architectural wall coatings, interior, high performance	4.3	D 1644
Asphalt roof coatings	4.4	D 2823
Asphalt roof coatings, aluminum-pigmented	4.5	D 2824
Bitumens, emulsified	4.6	D 2939
Bleached lac varnish	4.7	D 1650
Coil coatings	4.8	D 1353
		D 2697
Electrical insulation varnishes	4.9	D 115
House paints, gloss	4.10	D 2697
Industrial baking enamel	4.10	D 2697
Lacquers, clear and pigmented	4.11	D 1644
		D 333
Latex paint, exterior	4.10	D 2697
Latex paint, interior	4.10	D 2697
Magnet wire enamels	4.12	D 3288
Plastics, coatings for	4.13	D 1644
Powder coatings	4.14	D 3451
Shellac varnish, orange	4.7	D 1650
Solvent-reducible coatings	4.2	D 2369
Traffic paints	4.15	D 2205
Varnishes	4.16	D 1644
Wall and trim enamels, interior semigloss, solvent-based	4.17	D 2697
Wall paints, flat	4.10	D 2697
Water-reducible coatings	4.2	D 2369

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

Current edition approved March 9, 1983. Published September 1983. Originally published as D 2832 - 69. Last previous edition D 2832 - 69 (1980).

D 115 Methods of Testing Varnishes Used for Electrical Insulation²

D 333 Test Methods for Clear and Pigmented Lacquers³
D 1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products⁴

D 1644 Test Methods for Nonvolatile Content of Varnishes³

D 1650 Methods of Sampling and Testing Shellac Varnish⁵

D 2205 Guide for Selection of Tests for Traffic Paints³

D 2369 Test Method for Volatile Content of Coatings³

D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings³

D 2823 Specification for Asphalt Roof Coatings⁶

D 2824 Specification for Aluminum-Pigmented Asphalt Roof Coatings⁶

D 2939 Method of Testing Emulsified Bitumens Used as Protective Coatings⁶

D 3062 Test Method for Solids Content of Aerosol Coatings⁷

D 3288 Method of Testing Magnet-Wire Enamels⁸

D 3451 Practices for Testing Polymeric Powders and Powder Coatings³

3. Significance and Use

3.1 The nonvolatile content of paint and related coatings is useful to producers and users and to environmental and health and safety interests in comparing the coverage of competing products and in estimating the volatile organic content.

4. Procedure

4.1 *Aerosol Coatings*—Test Method D 3062 covers the determination of solids content (weight %) in aerosol coatings.

4.2 *Volatile Content of Coatings (Test Method D 2369)*—This test method covers the determination of the volatile content of coatings. It is considered to be applicable to most

² Annual Book of ASTM Standards, Vol 10.01.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 06.03.

⁵ Annual Book of ASTM Standards, Vol 06.02.

⁶ Annual Book of ASTM Standards, Vol 04.04.

⁷ Annual Book of ASTM Standards, Vol 15.09.

⁸ Annual Book of ASTM Standards, Vol 10.02.

solvent-reducible and water-reducible paints.

4.3 High Performance Interior Architectural Wall Coatings (HIPAC)—Determine the nonvolatile content of HIPAC coatings in accordance with Test Methods D 1644. Calculate the volatile content (weight %) by difference.

4.3.1 Method A—3 h at 105°C for paints with nonsolvent components that decompose at higher temperature.

4.3.2 Method B—10 min at 149°C for most paints with nonsolvent components that are reasonably stable at 149°C.

4.4 Asphalt Roof Coatings—Determine the nonvolatile content (weight %) of asphalt roof coatings of brushing or spraying consistency in accordance with 8.2 of Specification D 2823.

4.5 Aluminum-Pigmented Asphalt Roof Coatings—The nonvolatile content (weight %) of asphalt-based aluminum roof coatings suitable for application to roofing or masonry surfaces by brush or spray is determined in accordance with 8.2 of Specification D 2824.

4.6 Emulsified Bitumens Used as Protective Coatings—Section 8 of Test Methods D 2939 contains a method for determining residue by evaporation (weight %) of emulsified bitumens used in relatively thick films as protective coatings for metals and built-up roofs.

4.7 Shellac Varnish—Determine the nonvolatile matter in orange shellac and bleached lac varnishes in accordance with Sections 14 through 16 of Methods D 1650.

4.8 Coil Coatings—Although stated to be for solvents, determine the nonvolatile matter (weight %) in accordance with Test Method D 1353. Determine volume solids in accordance with Test Method D 2697.

4.9 Electrical Insulation Varnishes:

4.9.1 Sections 18 through 22 of Methods D 115 on nonvolatile matter by weight, are applicable to the following classifications of varnishes used for electrical insulation: alcohol-soluble varnishes, oxidizing air-drying varnishes, thermosetting varnishes, oxidizing baking varnishes, air-drying asphaltic varnishes, silicone varnishes, and thermosetting laminating varnishes.

4.9.2 Determine nonvolatile matter in electrical insulating varnishes intended for electrical equipment operating at 180°C and above in accordance with Methods D 115 except that the temperature used shall be $275 \pm 5.5^\circ\text{F}$ ($135 \pm 3^\circ\text{C}$) or at a temperature agreed upon between the producer and the user.

4.10 Volume Nonvolatile Matter in Clear or Pigmented Coatings (Test Method D 2697)—This test method is applicable to the determination of the volume nonvolatile matter of coatings. A gloss enamel, a flat wall paint, a gloss house paint, an industrial baking enamel, an interior latex paint,

and an exterior latex paint included in formal collaborative studies of this test method.

4.11 Lacquer Coatings—Determine the nonvolatile content of clear and pigmented lacquers as described in Test Methods D 1644. As an additional requirement, the specimen shall be reheated and reweighed until the weight is constant to within 1 mg. Method A of Test Methods D 1644 is preferred since Method B is potentially dangerous when used with lacquers.

4.12 Magnet Wire Enamels—Sections 17 through 23 of Test Method D 3288 cover the determination of nonvolatile content (weight %) in magnet wire enamels.

4.13 Coatings for Plastics—Determine nonvolatile matter in clear and pigmented coatings designed for use on rigid or semirigid plastic substrates in accordance with Test Methods D 1644.

4.14 Polymeric Powders and Powder Coatings—Determine nonvolatile content (weight %) in accordance with Section 12 of Practices D 3451. Determine volatile content at baking or fusion temperature in accordance with Section 13 of Practices D 3451.

4.15 Traffic Paints—Determine the nonvolatile content of traffic paints, ready-mixed, of spraying consistency of the premix, drop-in, or combination type in accordance with Test Methods D 1644, and state any necessary larger specimen size for beaded paint. Either of the two methods can be used as follows:

4.15.1 Method A—3 h at 105°C for paints with nonsolvent components that decompose at higher temperature.

4.15.2 Method B—10 min at 149°C for most paints with nonsolvent components that are reasonably stable at 149°C.

4.16 Varnishes—Nonvolatile content (weight %) of varnishes is determined using Test Methods D 1644. These test methods may give high results due either to incomplete elimination of volatile matter or to absorption of oxygen by oxidizing-type varnishes.

4.17 Solvent-Based Interior Semigloss Wall and Trim Enamels—Use Test Method D 2369 to determine volatile content. Volume nonvolatile matter is determined in accordance with Test Method D 2697.

5. Precision

5.1 Some of the referenced ASTM standards have precision limits. Reference to the individual standards for precision statements is recommended.

6. Keywords

6.1 nonvolatile content of paints and related coatings; volatile content of paints and related coatings

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Standard Index of Methods for Testing Architectural Paints and Coatings¹

This standard is issued under the fixed designation D 2833; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This index is provided for reference in the selection of procedures, test methods, and specifications to be used in the evaluation of architectural paints and coatings designed for application in the field. Composition specifications and analytical methods in general, also tests on raw materials, are not included, the scope being limited to procedures required in the evaluation of finished coatings.

1.2 Methods suitable for both interior and exterior coatings of either the water-thinned or solvent-thinned type are listed by ASTM designation primarily. Where comparable (but not necessarily identical) methods exist in U.S. Federal Test Method Standard No. 141, the Federal Method numbers are also listed.

1.3 Where more than one test method is listed for the same characteristic in this index, no attempt is made to indicate superiority of one test method over another. Selection of the test methods to be followed must be governed by experience and the requirements in each individual case,

¹ This index is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved March 31, 1989. Published May 1989. Originally published as D 2833 - 69. Last previous edition D 2833 - 83.

together with agreement between the purchaser and the seller.

2. Precision and Bias

2.1 If available, precision for any procedure or test method selected is given in the latest issue of the subject standard.

3. Test Categories

3.1 For convenience in selection, the procedures, methods, and specifications listed in this index are classified into three groups and three classes. The category of each listing is indicated in a column preceding the reference. The classifications are as follows:

3.1.1 *Group I—Applicable to Either Water- or Solvent-Reducible Coatings:*

Class A—For both interior and exterior products.

Class B—For interior products only.

Class C—For exterior products only.

3.1.2 *Group II—Water-Thinned Coatings:*

Class A—For both interior and exterior products.

Class B—For interior products only.

Class C—For exterior products only.

3.1.3 *Group III—Solvent-Thinned Coatings:*

Class A—For both interior and exterior products.

Class B—For interior products only.

Class C—For exterior products only.

INDEX OF METHODS

Category		ASTM Volume No.	ASTM Designation	Federal Standard No. 141	Category
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IA	Primers and Primer Surfacers over Preformed Metal	06.01	D 3322		
IA	Sampling Liquid Paints and Related Pigmented Coatings	06.01	D 3925	1022, 3011	IA
IB	Testing High-Performance Interior Architectural Wall Coatings	06.01	D 3730		IA
IIA	Resistance of Emulsion Paints in the Container to Attack by Microorganisms	06.01	D 2574		IA
IIA	Water-Borne Floor Paints	06.01	D 3358		
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IIIB	Interior Latex Semigloss and Gloss Paints	06.01	D 4540		IA
IIIC	Testing Asphalt Emulsions for Use as Protective Coatings for Metal	^a	D 1010		IA
IIIC	Exterior Latex House Paints	06.01	D 3129		IA
IIIA	Testing Varnishes	06.01	D 154		IA
IIIA	Clear and Pigmented Lacquers	06.01	D 333		IA
IIIA	Solvent-Reducible Floor Paints	06.01	D 3383		IA
IIIB	Performance Tests of Clear Floor Sealers	06.01	D 1546		IA
IIIB	Sampling and Testing Shellac Varnish	06.02	D 1650		IA
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IIIB	Solvent-Reducible Interior Semigloss Wall and Trim Enamels	06.01	D 3425		
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IIA	Particle Size of Multicolor Lacquers	06.01	D 2338		IIA
IIIA	Flash Point by Tag Closed Tester	06.03	D 56		IIA
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IA	Preparation of Hot-Dipped Nonpassivated Galvanized Steel for Testing Paint	06.01	D 2201		IC
IA	Pictorial Surface Preparation Standards for Painting Steel Surfaces	06.01	D 2200		
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IIIA	Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting	06.01	D 1730		IC
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^a Discontinued; see 1980 Annual Book of ASTM Standards, Part 27.

^a Discontinued
^c Discontinued

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IA	Drying, Curing or Film Formation of Organic Coatings at Room Temperature	06.01	D 1640	4061.1
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IA	Hiding Power of Paints by Reflectometry	06.01	D 2805	
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IA	Specifying Color by the Munsell System	06.01	D 1535	
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^a Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

^c Discontinued; see 1976 Annual Book of ASTM Standards, Part 27.

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IIIA	Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood	06.01	D 1211	
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IIIA	Detergent Resistance of Organic Coatings	06.01	D 2248	
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IIC	Reporting Paint Film Failures Characteristic of Exterior Latex Paints	06.01	D 1848	
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IIIC	Conducting Exterior Exposure Tests of Paints on Steel	06.01	D 1014	
IIIC	Exterior Durability of Varnishes	06.01	D 1641	

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Standard Test Method for Qualitative Tests for the Presence of Water Repellents and Preservatives in Wood Products^{1,2}

This standard is issued under the fixed designation D 2921; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes simple qualitative field or laboratory tests to determine water repellency or the presence of chlorinated phenol³ preservative chemicals in wood products that are specified to be water repellent preservative treated.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 U.S. Federal Specification:

TT-W-572 Wood Preservative Water-Repellent⁴

2.2 NIST Standard:

262-63 Water Repellent Preservative Non-Pressure Treatment for Mill Work⁵

3. Significance and Use

3.1 Although chlorinated phenol-treated wood has become less common due to environmental concerns, repellent-treated wood is commonly specified in construction. This test method provides a means to verify the presence of a significant level of water repellent protection.

4. Apparatus

4.1 *Eyedropper*, plastic squeeze bottle or similar means for metering drops of water.

4.2 *Flame Source*, such as bunsen burner, butane torch, or alcohol burner.

4.3 *Copper Wire Coil Specimen Holder (or Other Suitable Copper Holder)*—A suitable copper wire coil can be made by using a lead pencil as a mandrel to form a helix using copper wire of about $\frac{1}{16}$ to $\frac{3}{32}$ in. (1.6 to 2.4 mm) in diameter. Leave a space of approximately the diameter of the wire between each loop. The helix should be $\frac{3}{4}$ to 1 in. (19 to 25 mm) in length. Leave a pigtail of about 6 in. (152 mm) of wire at one end of the helix and form a loop of approxi-

mately 1 in. (25 mm) in diameter to be used as a holder for the coil.

4.4 *Sharp Knife*.

5. Water Repellent Test

5.1 Place uncut wood items to be tested so that the end grain is exposed as a horizontal surface. If the end grain cannot be so positioned, comparisons can be made on the flat grain.

5.2 With an eyedropper, or similar device, allow several drops of water to fall from about $\frac{1}{2}$ in. (13 mm) on the end grain of the wood. Wait 5 min and then observe the degree of penetration. With flat grain or vertical grain surfaces, waiting periods of 10 to 15 min may be necessary.

5.3 Water drops that immediately flatten out, penetrate and darken the wood, indicate that the wood has not been treated with a water repellent.

5.4 Water drops that "bead-up" and remain as spheres, with little or no color change or penetration, indicate that the wood has been treated with a water repellent. Water repellent preservatives, meeting Fed. Spec. TT-W-572 and NBS Standard 262-63, impart sufficient water repellency to the end grain of wood to cause water drops to bead up and form spheres.

6. Preservative Test

6.1 Chlorinated phenol based wood preservatives emit a characteristic green flame upon pyrolysis within a ventilated copper envelope due to the release of chlorine and its consequent reaction with copper (Beilstein's test). Untreated wood produces a yellow-orange flame but any chlorine containing compound will give a positive test.

6.2 Using the copper wire specimen holder, described in 3.3, hold the coil portion in the flame until it burns with a characteristic yellow-orange color. This preheated coil is then allowed to cool.

6.3 With a knife, cleaned by heating after each use, cut a splinter from the specimen of wood to be tested. Samples should be taken near the end of the wood items where end grain is exposed and where normal concentration of the preservative chemicals is greatest. The size of the wood sample should be smaller than the length and inner diameter of the coil.

6.4 With the copper coil held horizontally, place the splinter in the center of the coil. Put the copper coil with wood splinter in the flame and burn. If chlorinated phenols are present, the characteristic green flame is produced. Untreated wood produces the yellow-orange flame.

7. Accuracy

7.1 The tests are less accurate on flat grain and the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.52 on Factory-Coated Wood Products.

Current edition approved March 25, 1988. Published May 1988. Originally published as D 2921 - 70. Last previous edition D 2921 - 70 (1979)⁶.

² An improved quantitative test method for water repellents is under development by Subcommittee D01.42.

³ Pentachlorophenol, tetrachlorophenol and other chlorinated phenols.

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁵ Available from the National Institute of Standards and Technology, Gaithersburg, MD 20899.

heartwood of some wood species, so that end grain and sapwood should be used whenever possible.

7.2 Tests on both a known untreated sample and a treated sample of the same wood species will provide comparison standards for the operator and will improve the accuracy of the tests.

7.3 Test results are improved if tests are made with wood samples taken from the center of a stack or from a protected area where the possibility of contamination is least. Untreated items which come in physical contact with pre-

servative treated products during handling and shipping may show slight evidence of the preservative chemical.

7.4 More than one wood sample obtained from different areas from the unknown piece should be tested.

8. Precision and Bias

8.1 No numerical statement of precision is possible in this qualitative test method. One hundred percent agreement was obtained in a round-robin test.

8.2 Bias—Bias has not been determined.

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1. Scope

1.1 This standard covers the methods for testing wood preservative treated products.

1.2 The test methods apply to application means, previous surfaces.

1.3 The test methods address the responsibility of the user of the test method.

2. Refer

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¹ These methods are the property of the American Society for Testing and Materials and are not to be used for other than the purposes for which they were developed.
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Standard Guide for Testing Latex Flat Wall Paints¹

This standard is issued under the fixed designation D 2931; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—The title was editorially changed in March 1989.

1. Scope

1.1 This guide covers the selection and use of procedures for testing flat latex paints for use on interior walls. The test methods included are listed in Tables 1 and 2.

1.2 This guide applies to ready-mixed flat latex paints for application by brushing, roller coating, spraying, or other means, on plaster, masonry surfaces, wood, wallboard, previously painted surfaces and other interior architectural surfaces.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁴
- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁴
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁴
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁴
- D 1554 Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials⁵

- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁴
 - D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁶
 - D 1736 Test Method for Efflorescence of Interior Wall Paints⁴
 - D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁷
 - D 1849 Test Method for Package Stability of Paint⁴
 - D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁴
 - D 2243 Test Method for Freeze-Thaw Resistance of Water-Borne Paints⁴
 - D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
 - D 2369 Test Method for Volatile Content of Coatings⁴
 - D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints⁴
 - D 2574 Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms⁴
 - D 2805 Test Method for Hiding Power of Paints by Reflectometry⁴
 - D 2831 Test Method for Evaluating the Ability of a Latex Paint to Resist Efflorescence from the Substrate⁸
 - D 3258 Test Method for Porosity of Paint Films⁴
 - D 3450 Test Method for Washability Properties of Interior Architectural Coatings⁴
 - D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings⁴
 - D 4062 Test Method for Leveling of Paints by Draw-Down Method⁴
 - D 4213 Test Method for Wet Abrasion Resistance of Interior Paints⁴
 - E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁹
 - E 105 Recommended Practice for Probability Sampling of Materials⁶
- 2.2 *U. S. Federal Test Methods Standard 141:*¹⁰
- 2112 Application by Roller
 - 2131 Application of Sprayed Films
 - 2141 Application of Brushed Films

¹ These practices are under the jurisdiction of ASTM Committee D-1 on Paint, and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Aug. 31, 1984. Published January 1985. Originally published as D 2931 - 70 T. Last previous edition D 2931 - 79.

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 04.09.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

⁸ Discontinued; see 1980 Annual Book of ASTM Standards, Part 27.

⁹ Annual Book of ASTM Standards, Vol 15.05.

¹⁰ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

3011 Condition in Container
4321 Brush Properties
4541 Working Properties and Appearance of Dried Film

3. Definitions

3.1 For definition of terms used in this practice, refer to Definitions D 16 and D 1554.

4. Conditions Affecting Latex Wall Paints

4.1 *Substrate Type*—The substrate to be painted can affect not only the application properties and the physical properties of a latex paint, such as sheen and uniformity, but may also be a factor in determining the type of latex paint to be used. For instance, a latex paint with good sealing properties may be required for porous surfaces, such as new dry wall, bare plaster, new wood or porous masonry surfaces.

4.2 *Substrate Condition*—Dirty, chalky, alkaline, or water-soluble surfaces may affect the practical requirements and performance of interior latex flat paints.

4.3 *Application Properties*—The application properties of interior latex paints are affected by temperature, humidity, and the texture and porosity of the surface to be painted. Application will also depend upon the type and quality of equipment used.

4.4 *Physical Requirements*—The type of latex wall paint to be used will depend not only upon the surface to be painted, but also upon the desired physical properties of the finish. For instance, if maximum light diffusion and uniformity of appearance are the prime requirements, such as when painting ceilings, then a low sheen paint should be used. However, if cleanability or stain removal properties are desired for walls that become soiled, then a nonporous paint should be used.

5. Selection of Tests

5.1 It has been pointed out that there are many conditions that affect interior latex wall paints so that different types of latex paints have been developed specifically to meet the requirements of these various conditions. Although the recommended test methods presented in Tables 1 and 2 cover most of the properties of interior latex wall paints, all of these tests may not be required for each paint. If a paint is to be used only in a warm climate, for instance, then freeze-thaw tests or low temperature coalescence tests need not be considered.

5.2 The purchaser should first determine which properties a latex paint must have and then select only those test methods that will measure or evaluate these properties. After selecting the desired tests, the purchaser should determine which of these properties are the most important and then establish the requirements or specifications of these tests to get the most desired properties. Since some paint properties tend to oppose each other, such as low sheen versus good cleanability, or high viscosity versus good leveling, some properties may need to be attenuated if others are to be accentuated. This balance of properties must be considered when selecting the tests and establishing the requirements of these tests. The properties that these tests measure and the normal range of values have been presented for many of the tests, however.

5.3 Some tests cannot be selected as being more impor-

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
Liquid Paint Properties:			
Condition in container	7.1	...	3011.1
Coarse particles and foreign matter	7.2	D 185	...
Density (weight per gallon)	7.3	D 1475	...
Fineness of dispersion	7.4	D 1210	...
Odor	7.5	...	...
Consistency (viscosity)	7.6	D 562	...
Rheological properties, non-Newtonian liquids	7.7	D 2196	...
pH	7.8	E 70	...
Color compatibility	7.9	...	...
Freeze-thaw stability	7.10	D 2243	...
Package stability	7.11	D 1849	...
Microorganism resistance	7.12	D 2574	...
Paint Application and Film Formation:			
Application properties	8.1	...	4541, 4321
Application by brush	8.1.1	...	2141.1
Application by roller	8.1.2	...	2112
Application by spray equipment	8.1.3	...	2131
Drying time	8.2	D 1640	4061.1
Flow and leveling	8.3	D 4062	...
Low-temperature coalescence of paints	8.4	D 3793	...
Producing uniform thickness of films	8.5	D 823	2162
Touch-up	8.6	...	...
Appearance of Dry Paint Film:			
Color difference of opaque materials, visual evaluation	9.1	D 1729	...
Color difference of opaque materials, instrumental evaluation	9.2	D 2244	...
Hiding power	9.3	D 344, D 2805	...
Specular gloss	9.4	D 523	...
Uniformity of appearance	9.5	...	4541
Properties of Dry Paint Film:			
Efflorescence of paint film	10.1	D 1736	...
Efflorescence from substrate	10.2	D 2831	...
Elongation (flexibility)	10.3	D 1737, D 522	6221
Film porosity	10.4	D 3258	...
Scrubability (wet abrasion)	10.5	D 2486	6142
Stain removal (cleanability)	10.6	D 3450	...
Wet Abrasion Resistance	10.7	D 4213	...
Analysis of Paint:			
Chemical analysis	11.1	...	...
Volatile content	11.2	D 2369	...

tant than others and specific values for each test cannot be recommended since the properties that are important to one purchaser may not be important to another.

6. Sampling

6.1 Prior to sampling, the condition of the container should be established since damage to it may cause evaporation, skinning, or other undesirable effects in the coating. Determine the condition of the coating in accordance with 7.1 and 7.2.

6.2 Sample in accordance with Practice D 3925. Determine the weight per gallon in accordance with Test Method D 1475. Repeat this procedure until successive readings agree within 0.2 lb (90 g) or as agreed upon between the purchaser and the seller. Samples for testing may then be taken.

6.3 Specify the amount of sample, the package sizes, and identification codes to assure a representative sample. A

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TABLE 2 Alphabetical List of Test Methods

Federal Test Method Standard No. 141	Test Methods	Section	ASTM Test Method	Federal Test Method Standard No. 141
3011.1	Application by brush	8.1.1	...	2141.1
...	Application properties	8.1	...	4541, 4321
...	Application by roller	8.1.2	...	2112
...	Application by spray equipment	8.1.3	...	2131
...	Chemical analysis	11.1	...	...
...	Coarse particles and foreign matter	7.2	D 185	...
...	Color compatibility	7.9	...	...
...	Color difference of opaque materials, instrumental evaluation	9.2	D 2244	...
...	Color difference of opaque materials, visual evaluation	9.1	D 1729	...
...	Condition in container	7.1	...	3011.1
...	Consistency (viscosity)	7.6	D 562	...
...	Density (weight per gallon)	7.3	D 1475	...
...	Drying time	8.2	D 1640	4061.1
4541, 4321	Efflorescence of paint film	10.1	D 1736	...
2141.1	Efflorescence from substrate	10.2	D 2831	...
2112	Elongation (flexibility)	10.3	D 1737, D 522	6221
2131	Film porosity	10.4	D 3258	...
4061.1	Fineness of dispersion	7.4	D 1210	...
...	Flow and leveling	8.3	D 4062	...
...	Freeze-thaw stability	7.10	D 2243	...
2162	Hiding power	9.3	D 344, D 2805	...
...	Low-temperature coalescence of paints	8.4	D 3793	...
...	Microorganism resistance	7.12	D 2574	...
...	Odor	7.5	...	...
...	Package stability	7.11	D 1849	...
...	pH	7.8	E 70	...
...	Producing uniform thickness films	8.5	D 823	2162
...	Rheological properties of non-Newtonian liquids	7.7	D 2196	...
4541	Scrubability (wet abrasion)	10.5	D 2486	...
...	Specular gloss	9.4	D 523	...
...	Stain removal (cleanability)	10.6	D 3450	...
...	Touch-up	8.6	...	...
6221	Uniformity of appearance	9.5	...	4541
...	Volatile content	11.2	D 2369	...
6142	Wet Abrasion Resistance	10.7	D 4213	...

1-gal (4-L) sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling plan, consult Recommended Practice E 105.

7. Liquid Paint Properties

7.1 Condition in Container—Thickening, settling, and separation are undesirable and objectionable if the paint cannot be reconditioned with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties of paints after storage. Determine the condition in the container in accordance with Method 3011 of Federal Test Method Standard No. 141.

7.2 Coarse Particles and Foreign Matter:

7.2.1 Paints must be free of coarse particles to form uniform films of good appearance, a typical maximum being 2 weight % of total paint. The specified test with a 325-mesh (45- μ m) screen and water as the wash liquid gives the percent of these particles in a latex paint. Determine coarse particles and foreign matter in accordance with Test Methods D 185.

7.2.2 Another test method used in industry to determine whether coarse particles are present in a dry film is to scrape the surface of the film with a spatula or metal edge of a ruler.

Any particles larger than 325 mesh can be clearly seen after the surface has been scraped.

7.3 Density (Weight per Gallon)—The density as measured by weight per gallon (kilograms per litre) is used to assure product uniformity from batch to batch. It does not necessarily measure the quality of a paint. In the referenced method, the density is expressed as the weight in pounds of 1 U.S. gal (kg/L) of the paint at a specified temperature. Most interior flat latex paints have densities of about 10 to 12 lb/gal (1.2 to 1.4 kg/L). Determine density in accordance with Test Method D 1475.

7.4 Fineness of Dispersion:

7.4.1 The more finely a pigment is dispersed, the more efficiently it is being used. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the material down a calibrated tapered groove varying in depth from 4 to 0 mils (100 to 0 μ m). The point at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the liquid is taken as the fineness reading. Lower readings in mils or micrometres or higher readings in Hegman units indicate better fineness of dispersion.

7.4.2 The referenced method was designed primarily for coatings with good fineness of dispersion, such as enamels. Most interior flat latex paints have finenesses of about 70 to 90 μ m (1 to 2.5 Hegman). Although these paints may contain pigments so coarse that it is impractical to measure the fineness with a grind gage, most of the pigments are so finely dispersed that particles or agglomerates cannot be seen by the unaided eye or felt on the surface of the dry film. In addition, the fast drying of some flat latex paints may make it difficult to make accurate measurements of fineness of dispersion.

7.4.3 Determine fineness of dispersion with Test Method D 1210.

7.5 Odor—One of the advantages of latex paints is that they do not have odors characteristic of solvent-based coatings. However, other ingredients, such as ammonia, may be used which might also be objectionable in confined spaces. Thus, interior latex paints should be tested for odor acceptability. Although there is no adequate ASTM test method to measure odor for nonorganic solvent paints, test methods are used by industry to measure this property. Examine to determine if the paint has an unpleasant or irritating odor during application or drying.

7.6 Consistency (Viscosity)—Paints of a given type should fall within a stated consistency for satisfactory reproduction of a specific formula. While consistency is an important property it does not determine the quality of a paint and should be used mainly to assure product uniformity. In the referenced method, consistency is defined as the load in grams to produce a specified rate of shear. Although the consistency of most latex wall paint is about 150 to 300 g/100 revolutions, a much wider range is possible because of the wide variations in rheological properties of these paints. Also two paints of the same consistency may have quite different rheological properties. Determine the consistency in accordance with Test Method D 562.

7.7 Rheological Properties, Non-Newtonian Liquids—Rheological properties are related to application and leveling properties of the liquid paint. The referenced method covers

the determination of rheological properties and is particularly suited for use with paints that display thixotropic characteristics. It actually measures viscosity under varying conditions of time and rotational speed of the spindle. Determine rheological properties in accordance with Test Methods D 2196.

7.8 pH—Latex paints with low (acidic) pH may corrode the container. pH may vary from about 5 to 10 depending upon the type of latex used and the general formulation. pH does not determine the quality of a latex paint and should only be used to assure product uniformity. A change in pH during storage may indicate poor stability or a change in properties of a latex paint. Determine pH in accordance with Test Method E 70.

7.9 Color Compatibility—A test method to determine how well colorants can be dispersed in a paint so that the paint will have uniformity of color when applied is now in preparation and will be included in this guide when adopted by ASTM.

7.10 Freeze-Thaw Stability—Water-based paints may be subjected to freezing conditions during shipping and storage. Suitably stabilized paints will resist several cycles of freezing and thawing without showing deleterious changes such as coagulation, graininess, or excessive viscosity increase. Many latex paints will increase in viscosity but can still be considered usable if other properties, which may be affected by a higher viscosity, such as leveling and brushability, are satisfactory. Determine freeze-thaw stability in accordance with Test Method D 2243.

7.11 Package Stability—Since paints cannot normally be used immediately after manufacture, they must remain stable in the can for some time. At normal temperatures, most latex paints can be stored for over a year with little change in properties. Although package stability can usually be determined in several weeks at an elevated temperature such as 122°F (50°C), occasionally the results of the accelerated test do not coincide with those of prolonged room temperature storage. The referenced method covers the change in consistency and in certain other properties in packaged latex paint when stored at temperatures above freezing. Determine package stability in accordance with Test Method D 1849.

7.12 Microorganism Resistance—Bacteria in a latex paint can cause gassing, putrefactive, or fermentative odors and loss of viscosity. Determine if the paint contains living bacteria or if it is resistant to attack by bacteria in accordance with Test Method D 2574.

8. Paint Application and Film Formation

8.1 Application Properties—Determine the ease with which a paint can be applied to various wall surfaces with brush, roller, or spray equipment, in accordance with Method 4541 of Federal Test Method Standard 141. Application properties are generally compared to a standard, or described by requirements in a product specification.

8.1.1 Brushing Properties—The specified method covers the determination of the brushing properties of coatings. The test is quite subjective although someone experienced in the art can produce quite consistent results, particularly in the evaluation of "drag" properties. Determine brushing proper-

ties in accordance with Method 4321 of Federal Test Method Standard No. 141.

8.1.2 Roller Application—Determine the ease with which a paint can be roller applied in accordance with Method 2112 of Federal Test Method Standard 141. Since roller foam and roller spatter are often serious problems when latex paints are roller applied, these properties can also be determined in the test method for roller application. The amount of foam produced, the time that it takes for the bubbles to break, and the number of craters that remain after the bubbles have broken can be determined by visual examination of the test panel. Roller spatter can be determined by placing a strip of paper or a panel at the bottom of the test panel to catch the paint spatters from the roller. The degree to which a paint will spatter when roller applied can be determined by the density of the paint spatter.

8.1.3 Sprayed Film Application—Interior latex paints are sometimes applied by spray. Determine the spray application properties in accordance with Method 2131 of Federal Test Method Standard No. 141. The method can be modified to include application by airless spray equipment.

8.2 Drying Time—The drying time of an interior latex paint is important in determining when a freshly painted room can be put back to use. Under average conditions most flat latex paints are dry to touch in 1 or 2 h when the water has evaporated from the film. They can usually be recoated from within a few hours to 18 h. Curing to obtain the ultimate properties may take only a few days for some latex paints while others may require 1 or 2 weeks depending upon the composition. Determine drying time in accordance with Test Methods D 1640.

8.3 Flow and Leveling—These properties are usually evaluated by visually examining the test panel after a paint has been applied by brush or roller to see if any brush or roller marks can be seen in the dry film. Determine the ability of the paint to flow out after application in accordance with Test Method D 4062.

8.4 Low-Temperature Coalescence of Paints—Determine how well the latex particles in a paint will fuse together or coalesce, to form a continuous film at low temperatures in accordance with Test Method D 4062.

8.5 Producing Films of Uniform Thickness—The following method covers the preparation of various films of uniform thickness essential in conducting tests. Prepare films in accordance with Test Methods D 823.

8.6 Touch-Up—Although there is no test method published in Federal Test Method Standard 141 to measure touch-up, test methods are used by the paint industry. The usual method is to apply the test paint to a small section of a test panel that has previously been painted with the test paint and allowed to dry. The touch-up area is usually applied with a small brush. When the touch-up area has dried, it is examined to see if there is any difference in color or sheen between the touched-up area and the initial coat of paint.

9. Appearance of Dry Film

9.1 Color Differences of Opaque Materials by Visual Evaluation—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of light source, illuminating and

viewing conditions to be used of opaque materials in accordance with Practice 9.2 Color D

9.2 Color D Evaluation—Color standard can tolerance is agreed upon. Although color eye, they frequently compare method covers differences of nonfluorescent coated specimens (9.1) standard instrumental method

9.3 Hiding—Measure of the hiding power, however, depends upon practical test thickness is usually as affected by film paint. Test method precise and accurate standard. Paint the effects of measured, determine hiding in accordance with D 2805.

9.4 Specular—85° geometric appearance of 85° sheen of uniformity of paints with sheen, this should not be determined in D 523.

9.5 Uniformity—Method 4541 examine the color or sheen

10. Properties

10.1 Efflorescence—method measures itself, not efflorescence of formulations of ter sufficient soil deposit on the surface in accordance with

10.2 Efflorescence—strates may be

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viewing conditions, size of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color difference in accordance with Practice D 1729.

9.2 Color Differences of Opaque Material by Instrumental Evaluation—Color difference between a product and the standard can be measured by instrument. Generally, the tolerance is agreed upon by the purchaser and the seller and may also be required if a product specification is involved. Although color instruments are not more sensitive than the eye, they provide numerical values which can be subsequently compared to later measurements. The referenced method covers the instrumental determination of small color differences observable in daylight illumination between nonfluorescent, nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (9.1) should be used to verify the results. Make instrumental measurement of color difference in accordance with Method D 2244.

9.3 Hiding Power (Dry Opacity)—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness which is influenced by flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, opacity is evaluated visually as compared to a standard paint, and results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test that does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Determine hiding power in accordance with Test Methods D 344 or D 2805.

9.4 Specular Gloss (Sheen)—The method given, using the 85° geometry, is useful in characterizing the low angle appearance of flat paints. Most flat latex wall paints have an 85° sheen of about 1 to 10. Although paints with good uniformity of appearance are often paints of lower sheen and paints with good cleanability are often ones with higher sheen, this is not always the case, and the sheen of a paint should not be used as a measure of other paint properties. Determine the 85° gloss in accordance with Test Method D 523.

9.5 Uniformity of Appearance—Apply the paint as in Method 4541, Federal Test Method Standard 141, and examine the dry paint to see if there is any nonuniformity of color or sheen.

10. Properties of Dry Film

10.1 Efflorescence of the Paint Film—The referenced method measures efflorescence that comes from the paint itself, not from the substrate. Few interior latex paints effloresce due to improvements in latex and latex paint formulations. Salt formation is produced by specific conditions of temperature and humidity if a paint contains sufficient solid water-soluble material to cause a noticeable deposit on the film. Determine efflorescence resistance in accordance with Test Method D 1736.

10.2 Efflorescence from Substrate—Cementitious substrates may contain sufficient solid water-soluble materials to

cause a surface deposit through leaching and evaporation. Determine if a latex paint applied over an alkaline masonry surface will allow soluble salts to pass through the film in accordance with Test Method D 2831.

10.3 Elongation—Elongation is a measure of the flexibility of a paint film. Most interior latex paints can be bent over a ¼ in. (6.4-mm) mandrel without affecting the film. Determine elongation in accordance with Test Methods D 522 or D 1737.

10.4 Film Porosity—The more porous a paint is, the worse will be its cleanability and enamel holdout. Determine the film porosity in accordance with Test Method D 3258.

10.5 Scrubbability—The ability of an interior finish to resist scrubbing is an important property. The referenced method provides a measure of the wet abrasion resistance of a film. However, wet abrasion resistance is not necessarily a measure of how well soils or stains can be removed since some paints have good scrubbability but poor stain cleanability because they are porous. The scrubbability of interior latex flat paints can vary from less than 100 to more than 1000 cycles. Determine the scrubbability in accordance with Test Method D 2486. A control paint should always be tested at the same time because of the variability of the method.

10.6 Stain Removal (Cleanability)—The ability to remove marks satisfactorily without damaging the film is an important property of interior finishes. A test method to determine how well stains and soils can be removed from a cured paint film is now in preparation and will be included in this guide when it is approved by ASTM. Test methods used by the paint industry consist of applying various household soils or stains to the cured paint film and attempting to remove these soils or stains by means of a washing or scrubbing operation. The attempted removal of the soils or stains can be done by hand or with a mechanical apparatus. Determine the relative ease of removal of soil from the dried film by washing with either an abrasive or nonabrasive cleaner in accordance with Test Method D 3450.

10.7 Wet Abrasion Resistance (Weight Loss Method)—This method differs from Test Method D 2486, scrub resistance (10.5), in that the loss in weight or volume of the paint film is recorded for each 100 scrub cycles. In Test Method D 2486 the number of scrub cycles required to wear through the paint film is recorded. Determine the wet abrasion resistance of the paint in accordance with Test Method D 4213.

11. Analysis of Paint

11.1 Chemical Analysis—If a specification requires certain raw materials or certain components in a given amount, then chemical analysis is required to determine whether the specified materials are present and in what amounts. Analysis does not necessarily establish paint quality which can also be greatly affected by manufacturing techniques. Most ASTM analytical methods apply to solvent-based coatings. However, some of them can be adapted for analysis of latex paints.

11.2 Volatile Content—The amount of volatile material in a paint cannot be used as an indication of the quality of a paint. This value is useful, however, in determining the

similarity of two samples. Determine volatile content in accordance with Test Method D 2369.

12. Field Testing

12.1 Although the recommended test methods attempt to

duplicate actual conditions under which latex flat wall paints are applied, it is not possible to duplicate all possible conditions. It is therefore necessary to test latex flat wall paints under actual existing conditions for a final evaluation of the quality of a paint.

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Standard Guide for Testing Exterior Solvent-Reducible House and Trim Coatings¹

This standard is issued under the fixed designation D 2932; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Editorial changes were made throughout, including the title, in October 1988.

1. Scope

1.1 This guide covers the selection and use of procedures for testing exterior solvent-reducible house paints and trim coatings in the laboratory and in the field. The test methods included are listed in Tables 1 and 2. All of these tests may not be required for each paint. Selection of the test methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.

1.2 This guide covers the testing of ready-mixed solvent-reducible house paint or trim coatings for application by brush, roller, or spray on exterior surfaces. The paint may be any of the following types:

1.2.1 *Type I Paint, Oil*—Titanium, lead, zinc, and oil, nonfume-resistant, white and colors. For general exterior use, particularly on wooden structures.

1.2.2 *Type II Paint, Oil*—Titanium, zinc, and oil, fume-resistant, white and colors. Suitable for use on exterior surfaces such as wood and primed steel or sealed concrete, where resistance against discoloration from hydrogen sulfide is required.

1.2.3 *Type III Paint, Oil-Alkyd*—Combination of oil and alkyd in the vehicle and pigments, composed of chalk-resistant and chalking titanium dioxide, white and colors. For general exterior use, particularly on wooden surfaces, trim, primed steel, or sealed concrete.

1.2.4 *Type IV Paint, Miscellaneous Vehicles*—Combination of pigments and vehicle, white and colors. Suitable for use on exterior wooden surfaces, trim, primed steel, or sealed concrete.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 16 Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products²

D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester³

D 154 Guide for Testing Varnishes⁴

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints⁵

D 215 Methods of Chemical Analysis of White Linseed Oil Paints⁴

D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁴

D 358 Specification for Wood to Be Used as Panels in Weathering Tests of Coatings⁴

D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings⁴

D 523 Test Method for Specular Gloss⁴

D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴

D 659 Method of Evaluating Degree of Chalking of Exterior Paints⁴

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints⁴

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁴

D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints⁴

D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints⁴

D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood⁴

D 1038 Definitions of Terms Relating to Veneer and Plywood⁶

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁴

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁴

D 1554 Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials⁶

D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room⁶ Temperature⁴

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁷

D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁸

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials, and is the direct responsibility of Subcommittee D 01.42 on Architectural Finishes.

Current edition approved Aug. 1, 1980. Published November 1980. Originally published as D 2932 - 70 T. Last previous edition D 2932 - 79¹.

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vols 05.01, 06.01, and 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁶ Annual Book of ASTM Standards, Vol 04.09.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Discontinued; see Annual Book of ASTM Standards, Vol 06.01.

- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁴
- D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates⁴
- D 2245 Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints⁸
- D 2366 Test Method for Accelerated Testing and Moisture Blister Resistance of Exterior House Paints on Wood⁷
- D 2369 Test Method for Volatile Content of Coatings⁴
- D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁴
- D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints⁴
- D 2621 Test Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints⁴
- D 2698 Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging⁴
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁴
- D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus⁹
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry¹⁰
- E 105 Recommended Practice for Probability Sampling of Materials⁷
- 2.2 U. S. Federal Test Method Standard 141:¹¹
- 141/1021 Sampling (General)
- 141/2131 Application of Sprayed Films
- 141/2141.1 Application of Brushed Films
- 141/3011.1 Condition in Container
- 141/4203.1 Reducibility and Dilution Stability
- 141/4421 Absorption Test
- 141/4541 Working Properties and Appearance of Dried Film

3. Definitions

3.1 For definition of terms used in these practices, refer to Definitions D 16, D 1554, and D 1038.

4. Conditions Affecting Solvent-Based House and Trim Coatings

4.1 Practical requirements and performance for solvent house or trim paints may vary with:

4.1.1 Substrate type, such as type and quality of wood or hardboard; type of grain of wood; knots; and type, quality, and alkalinity of concrete substrates.

4.1.2 Condition of previously painted substrates such as degree of chalk, adhesion of film, dirt, mold, and general condition of old coating.

4.1.3 Type and quality of primer and time before topcoating.

4.1.4 Climatic conditions at the time of coating application and immediately after application.

⁹ Annual Book of ASTM Standards, Vol 06.03.

¹⁰ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

¹¹ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Method	Federal Test Method Std. No. 141
Liquid paint properties:			
Skimming	6.1	D 154	3021
Condition in container	6.2	...	3011.1
Coarse particles and foreign matter	6.3	D 185	4081
Weight per gallon or density	6.4	D 1475	4184.1
Fineness of dispersion	6.5	D 1210	4411.1
Consistency	6.6	D 562	4281
Dilution stability	6.7	...	4203.1
Rheological properties of non-Newtonian liquids	6.8	D 2196	...
Absorption	6.9	...	4421
Flash point	6.10	D 93, D 3278	...
Paint application and film formation:			
Working properties	7.1	...	4541
Application by brush	7.2	...	2141.1
Application by spray	7.3	...	2131
Drying properties	7.4	D 1640	4081.1
Appearance of dry paint film:			
Color difference of opaque materials, visual evaluation of	8.1	D 1729	4249.1
Color difference of opaque materials, instrumental evaluation of	8.2	D 2244	6123.1
Directional reflectance	8.3	E 97	6121
Gloss (60-deg specular)	8.4	D 523	6101
Hiding power	8.5	D 2805, D 344	4121
Properties of dry paint film:			
Elongation (flexibility)	9.1	D 1737	6221
Moisture blister resistance	9.2	D 2366, D 522	...
Fume resistance	9.3	...	...
Exterior exposure resistance	9.4	D 1006	8181.1
Wood panel description	9.4.1	D 358	2031
Chalk resistance ratings	9.4.2	D 659	6411
Checking resistance ratings	9.4.3	D 660	6421
Cracking resistance ratings	9.4.4	D 661	6471
Erosion resistance ratings	9.4.5	D 662	6431
Flaking resistance ratings	9.4.6	D 772	6441.1
Analysis of paint:			
Chemical analysis	10.1	D 215	...
Volatile content	10.2	D 2369	4041.1
Pigment content	10.3	D 2371, D 2698	4021.1
Pigment analysis	10.4	D 215	7261 ¹⁰
Nonvolatile vehicle content	10.5	D 215	4059
Identification of vehicle solids	10.6	D 2621	...
Identification of oils	10.7	D 2245	7501

4.1.5 Environmental conditions after application, both general for the area and specific, such as under eaves, behind shrubbery, and north- and south-side exposures, and

4.1.6 Structural aspects of the building. If construction defects due to age are such that excessive moisture from the inside or from the outside makes its way through to the substrate, blistering, flaking, or peeling may result.

5. Sampling

5.1 To ensure an adequate and representative sample of paint to conduct tests agreed upon between the purchaser and the seller, proper sampling is important.

5.2 Sample the coating in accordance with Method 102 of U. S. Federal Test Method Standard 141. Prior to

TABLE 2 Alphabetical List of Test Methods

Federal Test Method Std. No. 141	Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
3021	Absorption	8.9	...	4421
3011.1	Application by brush	7.2	...	2141.1
4091	Application by spray	7.3	...	2131
4184.1	Chalk resistance ratings	9.4.2	D 669	6411
4411.1	Checking resistance ratings	9.4.3	D 660	6421
4281	Chemical analysis	10.1	D 215	...
4203.1	Coarse particles and foreign matter	6.3	D 185	4091
...	Color difference of opaque materials, instrumental evaluation of	8.2	D 2244	6123
4421	Color difference of opaque materials, visual evaluation of	8.1	D 1729	4249.1
...	Condition in container	6.2	...	3011.1
4541	Exterior exposure resistance	9.4	D 1006	6161.1
2141.1	Consistency viscosity	6.6	D 562	4201
2131	Cracking resistance ratings	9.4.4	D 661	6471
4061.1	Directional reflectance	8.3	E 97	6121
...	Dilution stability	6.7	...	4203.1
...	Drying properties	7.4	D 1640	4061.1
...	Elongation (flexibility)	9.1	D 1737, D 522	6221
4249.1	Erosion resistance ratings	9.4.5	D 662	6431
6123	Fineness of dispersion	6.5	D 1210	4411.1
...	Flaking resistance ratings	9.4.6	D 772	6441
6121	Flash point	6.10	D 93, D 3278	...
6101	Fume resistance	9.3	...	...
4121	Gloss (60-deg specular)	8.4	D 523	6101
...	Hiding power	8.5	D 2805, D 344	4121
6221	Identification of oils	10.7	D 2245	7501
...	Identification of vehicle solids	10.6	D 2621	...
...	Moisture blister resistance	9.2	D 2366	...
...	Nonvolatile vehicle content	10.5	D 215	4053
...	Pigment analysis	10.4	D 215	7261
6161.1	Pigment content	10.3	D 2371, D 2698	4021.1
2031	Rheological properties of non-Newtonian liquids	6.8	D 2196	...
6411	Sampling	5	...	1021
6421	Skinning	6.1	D 154	3021
6471	Volatile content	10.2	D 2369	4041.1
6431	Weight per gallon or density	6.4	D 1475	4184.1
6441	Wood panel description	9.4.1	D 358	2031
...	Working properties	7.1	...	4541

sampling, stir the paint as in 4.21 of Method 1021. Determine the weight per gallon (or kilograms per litre) in accordance with Test Method D 1475. Stir again and recheck the weight per gallon. The two readings should agree within 0.10 lb (45 g).

5.3 Specify the amount of sample, the package sizes, and identification codes to assure a representative sample. A 1-gal (4-L) sample is usually sufficient for the recommended tests but for guidance in selecting a sampling plan consult Recommended Practice E 105.

6. Liquid Paint Properties

6.1 *Skinning*—Paints containing a binder, that dries by oxidation are subject to skin formation in a partially filled can or by diffusion of air into the can. Since skins are insoluble in the paint, they must be removed before use. This test in a partially filled container indicates the tendency of a paint to skin. Examine the original sample for skins, both on the surface and in its mass. Using a well-mixed, skin-free

portion of the sample, perform a skinning test in accordance with Guide D 154.

NOTE 1—A typical minimum time for skinning in accordance with Guide D 154 is 48 h.

6.2 *Condition in Container*—After storage, paints should be suitable for application with minimum stirring. Characteristics that are undesirable and objectionable in a stored paint are given in the referenced test method. Determine condition in container in accordance with Method 3011.1 of Federal Test Method Standard No. 141.

6.3 *Coarse Particles and Foreign Matter*—Paints must be free of oversize particles and foreign matter to form a uniform film of good appearance. This test, with a 325-mesh (45- μ m) screen, gives the percent of these particles in the paint. Determine coarse particles and foreign matter in accordance with Test Methods D 185.

NOTE 2—A typical maximum for coarse particles and foreign matter is 1 weight % of total paint.

6.4 *Density or Weight per Gallon*—The density of a paint as measured by weight per gallon (or kilograms per litre) provides a check against the theoretical weight calculated from the formula and is useful for determining similarity of two samples. The density is expressed as the weight in pounds of 1 U. S. gal (or kilograms per litre) of the liquid at a specified temperature. A calibrated weight per gallon cup is used. Determine weight per gallon in accordance with Test Method D 1475.

6.5 *Fineness of Dispersion*—The more finely a pigment is dispersed the more efficiently it is being utilized. One method for measuring degree of dispersion is to draw a paint down a tapered groove in a hardened steel block with the groove varying in depth from 4 to 0 mils ((100 to 0 μ m) 0–8 Hegman units). The point at which continuous groupings of particles or agglomerates protrude through the surface of the wet film is taken as the dispersion reading. Higher readings (Hegman units) indicate better dispersion. Determine the fineness of dispersion in accordance with Test Method D 1210.

NOTE 3—A typical reading for house paint is 60 μ m (approximately 2½ mils) and for trim paint 25 μ m (1 mil). Several arbitrary scales and modifications of the gage are used by industry. The approximate relationship of these scales to micrometre and mil readings is shown in Test Method D 1210.

6.6 *Consistency (Viscosity)*—Paints of a given type should fall within a stated consistency range for satisfactory reproduction of a specific formula. While consistency is an important property, it does not determine the quality of a paint and should be used mainly to assure product uniformity. In the referenced method consistency is defined as the load in grams to produce a specified rate of shear. Although the consistency of most solvent-based house and trim coatings is about 150 to 300 g/100 revolutions, a much wider range is possible because of the wide variations in rheological properties of these paints. Also two paints of the same consistency may have quite different rheological properties. Determine the consistency in accordance with Test Method D 562.

6.7 *Dilution Stability*—Dilution with a specified thinner shows whether the materials are compatible and whether the reduced paint is stable. The referenced test is a measure of

the stability of paint that has been reduced to a desired viscosity, for example, for spray application. The diluent suggested for reduction should be readily incorporated into the paint without excessive stirring or shaking. Determine dilution stability in accordance with Method 4203 of Federal Test Method Standard No. 141.

6.8 Rheological Properties of Non-Newtonian Liquids—Rheological properties are related to application and flow properties of the liquid coating. The referenced test method covers the determination of the rheological properties of a paint and is particularly suited for use with paints that display thixotropic characteristics. It actually measures viscosity under varying conditions of time and rotational speed of the spindle. Determine rheological properties in accordance with Test Methods D 2196.

6.9 Absorption—On porous surfaces, binder penetration can result in pigment volume concentration changes as the film dries. This may cause performance to vary. The referenced method provides a rapid means for measuring the relative penetration of the binder into a porous surface. It provides a rough measure of the wetting and penetrating quality of liquid materials. Determine the absorption in accordance with Federal Test Method Standard No. 141, Method 4421.

6.10 Flash Point—When the flash point of a material is required for shipping information, use Test Methods D 93, Part B, or D 3278.

7. Paint Application and Film Formation

7.1 Working Properties—Working properties of a paint are generally compared to a standard or described by requirements in the product specification. Test working properties in accordance with Method 4541 of Federal Test Method Standard No. 141.

7.2 Application by Brush—Brushed films should be smooth and free of seeds and on vertical surfaces should show no sagging, streaking, or excessive brush marks. For proper method of application of a brushed film for test purposes, refer to Method 2141.1 of Federal Test Method Standard No. 141.

7.3 Application by Spray—This method covers application of a paint by spray, which is sometimes used for house and trim paint. Refer to Method 2131 of Federal Test Method Standard No. 141.

7.4 Drying Properties—The drying time of a coating is determined by its composition and by atmospheric conditions during drying. Insufficient drying may result in dirt or insect pickup resulting in a poor appearance, or a nonuniform appearance caused by dew or rain falling on the paint film. Testing a paint for the required drying properties can determine whether the paint has lost its drying properties through aging or whether insufficient drier was included in the product at the time of manufacture. The specific category of drying should be selected to suit the types of house paint being tested, since drying times vary widely. Determine the drying time in accordance with Test Methods D 1640.

8. Appearance of Dry Paint Film

8.1 Color Difference of Opaque Materials, Visual Evaluation of—Visual comparison is fast and often acceptable although numerical values are not obtained. The referenced

practice covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, sizes of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine the color difference in accordance with Practice D 1729.

8.2 Color Difference of Opaque Materials, Instrument Evaluation of—Color difference between a product and the standard can be tested by instrument. Generally the tolerance is agreed upon between the purchaser and the seller, and may also be required if a product specification is involved. Determine the color difference in accordance with Method D 2244.

8.3 Directional Reflectance—This property is a measure of light reflected from the surface of the paint. It usually is assigned a value in specifications for white and pastel shades. Determine daylight directional reflectance in accordance with Test Method E 97.

8.4 Gloss (60-deg Specular)—Determine the specular gloss in accordance with Test Method D 523. Oil house paints are typically in a gloss range from 30 to 70, while trim enamels are in a gloss range from 70 to 90.

8.5 Hiding Power (Dry Opacity)—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness which is influenced by the flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, opacity is evaluated visually as compared to a standard paint, and results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test that does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Determine hiding power in accordance with Test Methods D 344 or D 2805.

9. Properties of Dry Paint Film

9.1 Elongation (Flexibility)—Elongation is a measure of flexibility of a paint film. Generally, gloss house paints and trim paints will have no problem in passing a mandrel bend test. Flat house paints, however, may fail a sharp bend. Determine elongation in accordance with Test Methods D 1737 or D 522.

9.2 Moisture Blister Resistance—Blister resistance is related to the ability of a dry paint film to resist the formation of blisters caused by water from the wood substrate. The water can come from either the interior of the home or from structural defects that permit entry of the water behind the wood. Moisture blister resistance can be qualitatively evaluated in a laboratory test. Determine resistance to moisture blistering in accordance with Test Method D 2366.

9.3 Fume Resistance—Fume resistance is the ability of a dry paint film to resist discoloration in a moist hydrogen sulfide atmosphere. This type of atmosphere may be present near industrial or other polluted areas and can cause paint to yellow or darken in as little time as overnight exposure. There are no ASTM or Federal test methods for evaluating this property, but one procedure used by the industry is as follows:

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9.3.1 Apply a sufficient number of coats of the paint to two glass plates to hide the surface completely, allow to dry for 6 h and expose one of the plates in a moist atmosphere of hydrogen sulfide for 18 h. Compare the color with the unexposed plate. The exposed plate should be practically no darker than the unexposed one. The color difference should not exceed that between plates that have been coated with a paint made with titanium dioxide pigment, lead-free zinc oxide, raw or refined linseed oil, and sufficient cobalt added for drying, and which has been similarly treated.

9.4 *Exterior Exposure Resistance*—In conducting exterior exposures, refer to Practice D 1006.

9.4.1 *Wood Panel Description*—In establishing exposure performance, use the panels as described in Specification D 358.

9.4.2 *Chalk-Resistance Ratings*—Determine chalk-resistance rating using Method D 659.

9.4.3 *Checking-Resistance Ratings*—Determine checking-resistance rating using Method D 660.

9.4.4 *Cracking-Resistance Ratings*—Determine cracking-resistance rating using Test Method D 661.

9.4.5 *Erosion-Resistance Ratings*—Determine erosion-resistance rating using Test Method D 662.

9.4.6 *Flaking-Resistance Ratings*—Determine flaking-resistance rating using Test Method D 772.

10. Analysis of Paint

10.1 *Chemical Analysis*—If a specification requires specific ingredients, chemical analysis may be required. Select test procedures from Methods D 215, and other ASTM

methods that are pertinent to the components of exterior house paints.

NOTE 5—No single schematic analysis is comprehensive enough to cover the wide variety of house paint compositions.

10.2 *Volatile Content*—Thinner is removed from the paint at 110°C. The percentage of volatile matter calculated from this loss in weight indicates the thinner loss from the film as the paint dries. Determine volatile content of the paint in accordance with Test Method D 2369.

10.3 *Pigment Content*—The pigment gives paint its hiding and color and influences many other properties of the paint. Determine the percent pigment in accordance with Test Method D 2371.

10.4 *Pigment Analysis*—The analysis of pigment may be required if the product is covered by a specification, or upon agreement between the purchaser and the seller. Determine analysis of pigments in accordance with the selected test procedure from Method D 215.

10.5 *Nonvolatile Vehicle Content*—The nonvolatile vehicle is that portion of the film-forming solids in a paint other than the pigment. Water, volatile thinner, and pigment are determined and the sum subtracted from the total weight to give the nonvolatile vehicle content. Determine the nonvolatile vehicle content in accordance with Method D 215. Separate the vehicle for further analysis in accordance with Method D 2372.

10.6 *Identification of Vehicle Solids*—Determine identity of vehicle solids in accordance with Methods D 2621.

10.7 *Identification of Oils*—Determine identity of oils in accordance with Methods D 2245.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Method for Corrosion Resistance of Coated Steel Specimens (Cyclic Method)

This test method is intended for use in determining the resistance to failure of coatings of paint, varnish, lacquer, and related products on steel surfaces when exposed cyclically to salt spray, humidity, and cold to induce coating failure more representative of the type found in outdoor corrosive environments.

Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials, this test method was discontinued in 1992.

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Standard Practice for Evaluation of Coatings for Plastics¹

This standard is issued under the fixed designation D 3002; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is intended for the evaluation of clear and pigmented coatings designed for use on rigid or semi-rigid plastic substrates. Coated film and sheeting are not covered by this practice.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 523 Test Method for Specular Gloss²
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive²
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive²
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes²
- D 1474 Test Methods for Indentation Hardness of Organic Coatings²
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)³
- D 1644 Test Methods for Nonvolatile Content of Varnishes²
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁴
- D 2091 Test Method for Print Resistance of Lacquers²
- D 2199 Test Method for Measurement of Plasticizer Migration from Vinyl Fabrics to Lacquers²
- D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates²
- D 2246 Test Method for Finishes on Primed Metallic Substrates for Humidity-Thermal Cycle Cracking²
- D 2247 Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity²
- D 3170 Test Method for Chipping Resistance of Coatings²
- D 3359 Test Methods for Measuring Adhesion by Tape Test²
- G 24 Practice for Conducting Natural Light Exposures Under Glass⁴

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint, and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 3002 - 71. Last previous edition D 3002 - 78.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

⁴ Annual Book of ASTM Standards, Vol 14.02.

3. Significance and Use

3.1 This practice is designed to set up a series of screening tests that will indicate the performance level to be expected of a coating or coating system on a given plastic substrate.

3.2 Rigid and semirigid plastic substrates vary widely in their acceptance characteristics for a given coating.

3.3 Surface cleaning or preparation prior to application of the coating can be critical to the proper performance of the coating.

4. Precaution

4.1 This standard may involve the use of hazardous materials, operations, and equipment. It is the responsibility of whoever uses this standard to establish appropriate safety practices and to determine the applicability of regulatory limitations prior to use.

5. Test Panels and Panel Preparation

5.1 Unless directed otherwise, conduct performance tests on coatings of specified thickness applied to a normally molded sample of the designated plastic. Applied coatings cannot correct surface defects from improper molding procedures.

5.2 Preparation of test samples shall include any cleaning, metalizing, or priming operations agreed upon by the producer and the user.

5.3 Air-dry or bake the system according to the established schedule and aged as agreed between the producer and the user.

6. Nonvolatile Matter

6.1 Test for nonvolatile matter in the coating material in accordance with Test Methods D 1644.

7. Color

7.1 *Clear (on liquid sample)*—Determine the Gardner color in accordance with Test Method D 1544.

7.2 *Pigmented (on dry film)*—Evaluate color differences visually in accordance with Practice D 1729 and instrumentally in accordance with Method D 2244.

8. Gloss

8.1 Test in accordance with Test Method D 523.

9. Print Test

9.1 Test in accordance with Test Method D 2091.

10. Hardness

10.1 Apply a film to a clean steel panel at a thickness agreed upon by the purchaser and the seller. Test in accordance with Test Methods D 1474.

11. Tape Adhesion

11.1 Test for tape adhesion in accordance with Methods D 3359.

12. Resistance to Plasticizer Migration

12.1 Test in accordance with Test Method D 2199. Time and temperature may be varied as agreed between the producer and the user.

12.2 If migration from a plastic substrate into a coating is being determined, the vinyl contact pad should be omitted.

13. Resistance to Temperature-Humidity Cycling

13.1 Expose the finished panel in accordance with Test Method D 2246.

13.2 After the number of cycles agreed between the producer and the user, determine whether adhesion (Section 11) and appearance (Sections 7 and 8) have changed.

14. Resistance to Continuous 100 % Humidity

14.1 Expose the finished panel in accordance with Practice D 2247.

14.2 After exposure for the length of time agreed between the producer and the user, determine whether adhesion (Section 11) and appearance (Sections 7 and 8) have changed.

15. Resistance to Household Chemicals

15.1 Test in accordance with Test Method D 1308.

16. Wear Resistance

16.1 Conduct an abrasion test in accordance with Test Method D 658 or Test Methods D 968 and determine the abrasion coefficient.

17. Light Stability Test

17.1 Expose to sunlight in accordance with Recommended Practice G 24. Duration of test and maximum acceptable change in color shall be as agreed between the producer and the user.

18. Weather Resistance

18.1. Expose panels in Florida or other area agreed between the producer and the user, 45° South for 12 months and determine changes in appearance (Sections 7.2 and 8.1), and adhesion (Section 11).

19. Resistance to Chipping

19.1 Using the plastic, pretreatment, primer, and topcoat system agreed between the producer and the user, test with the gravimeter in accordance with Test Method D 3170.

20. Effect on Substrates

20.1 Determine the physical properties of the substrate such as hardness, tensile strength, flexibility, or surface smoothness before and after applying the coating under test. Select the appropriate tests for the substrate from Volumes 08.01, 08.02, and 08.03 of the *Annual Book of ASTM Standards*.

21. Precision

21.1 The precision of this practice is described by each individual method cited.

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Standard Test Method for Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates¹

This standard is issued under the fixed designation D 3003; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers determination of the pressure mottling and sticking, or blocking resistance of organic coatings applied to coil-coated or factory-coated metal prior to postformed.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Method

2.1 The coated metal is cut into suitably sized panels. A stack of these panels is then subjected to a specified pressure and temperature for a specified time to permit any pressure mottling and sticking or blocking to develop. The heating elements of the test apparatus are turned off and, after cooling, the specimens are examined for any signs of sticking or blocking, and mottling. The results are rated on the 0 to 10 scale and may be used in accepting or rejecting the coating according to standards established by the purchaser and the seller.

3. Description of Terms

3.1 *pressure mottling*—film distortion or uneven pattern giving a change of gloss and nonuniform appearance. It is usually caused by pressures within a painted coil or stacked painted sheets or other painted products.

3.2 *blocking or sticking*—the condition wherein coated surfaces adhere to each other.

4. Apparatus²

4.1 A suitable hydraulic or mechanical press or vise may be used. The equipment shall be capable of producing the required test pressure in pounds-force per square inch (or kilopascals) and be equipped with a suitable device for measuring the force applied.

5. Test Specimens and Conditions

5.1 At least four, and preferably six, flat panels shall be cut from the coated stock, the age of which shall be within the limits agreed upon by the purchaser and the seller.

5.2 Panels should be at least 4 by 2.5 in. (100 by 70 mm) to provide an adequate area for assessing the results. Where the equipment does not provide adequate pressure, smaller panels may be used. The minimum recommended size is 2 by 2 in. (50 by 50 mm).

5.3 Use only flat panels. If necessary, file the edges smooth to ensure maximum contact between the surfaces. When the equipment permits, panels larger than the pressure plates may be used, thus eliminating any effect from uneven edges. With this method, the pounds-force per square inch (or kilopascals) is calculated using only the panel area within the pressure plates.

5.4 The film thickness of the coating under test shall be as specified or agreed upon by the purchaser and the seller.

5.5 The coated stock shall be tested under the conditions of pressure, temperature, and time mutually agreed upon by the purchaser and the seller. Pressures ranging from 110 to 350 psi (750 to 2400 kPa), temperatures from 110 to 140°F (43 to 60°C), and times of 2 to 16 h have been used.

5.6 In the absence of agreed or specified test conditions, a pressure of 110 ± 5 psi (750 ± 35 kPa), a temperature of $110 \pm 3^\circ\text{F}$ ($43 \pm 1.5^\circ\text{C}$), and a time of 16 h shall be used.

5.7 The total force applied is measured by a suitable gage and the pounds-force per square inch (or kilopascals) is calculated by dividing the force (pounds-force or newtons) by the area in square inches (or square millimetres) of one side of one panel.

6. Procedure

6.1 Stack the panels face to back as used in production with both face and back coatings applied. Limit the number of panels in the test to the quantity that can be heated rapidly to and maintained at the correct panel temperature throughout the test.

6.2 For presses having heating units, place the test panels in the press and bring the heating platens in contact but with light pressure. Turn on the electrical heating unit, and heat the panels to the required test temperature. When this temperature is reached, the indicating light will shut off. Apply the required pressure and maintain it for the specified time (6.4).

6.3 Where the apparatus does not have built-in heating units, preheat the mechanical press or vise. Place the panels in the press at room temperature with very light contact and heat the unit in an oven to the required temperature. Then

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.53 on Factory-Precoated Strip Metal.

Edition effective Dec. 22, 1971.

² Suitable equipment includes Pasadena or Studebaker Hydraulic Presses; Gardner Laboratories Cam-activated Pressure Mottling Tester; and drill press vises modified for use with a torque wrench if agreed between purchaser and seller (Hensley, W. L., "Pressure Mottling Test," *Journal of Paint Technology*, Vol. 40, No. 517, February 1968, p. 544).

TABLE 1 Classification

NOTE 1—Intermediate grades are permissible to distinguish small differences.

NOTE 2—Reasonable force is defined as that which can be exerted by the fingers only, using the fingernails as the only prying instrument.

NOTE 3—The transfer of components of one coating onto or into another coating is also known as offsetting or pickoff.

Grade	Sticking or Blocking	Grade	Mottling
10	Panels fall apart—no sticking.	10	No mottling immediately after separation.
8	Slight tackiness or panels pull apart easily.	8	No mottling after 24 h separation.
6	Panels pull apart with effort. No flexing or prying required to aid separation.	6	Light permanent mottling after 24 h separation.
4	Panels pull apart using reasonable force. Flexing may be required (Note 2).	4	Heavy permanent mottling after 24 h separation.
2	Panels require prying apart with a spatula or similar instrument.	2	Light pickoff or transfer of one or both coatings (Note 3).
0	Panels block together so they cannot be separated with a spatula or similar instrument, without using extreme force.	0	One or both coatings show heavy pickoff down to the metal or basecoat; or block together (Note 3).

apply the proper pressure and return the unit to the oven for the specified time.

6.4 Record the time only after the specified temperature and pressure are reached. Check the temperature and pressure at intervals during the test.

6.5 At the end of the specified time, turn off the heat and allow the panels to cool to room temperature under pressure before removal and separation.

6.6 After removal from the press, grade the panels separately for both sticking or blocking and mottling in accordance with Table 1. In assessing the results ignore effects due to sharp or uneven edges.

6.7 Maintain panels that are to be reaccessed after a recovery period at $75 \pm 5^\circ\text{F}$ ($24 \pm 3^\circ\text{C}$).

6.8 For both properties assign each coating a grade which is the arithmetic mean of all panels tested.

7. Report

7.1 Report the following data for each coating:

7.1.1 Equipment used,

7.1.2 Number of panels tested,

7.1.3 Pressure,

7.1.4 Temperature and heating process (built-in or oven),

7.1.5 Time,

7.1.6 Grade for sticking or blocking, and

7.1.7 Grade for mottling.

8. Precision

8.1 In an interlaboratory test of this method, two operators in each of seven laboratories tested two different coatings, subjected to two treatments (pressure, temperature, and duration) and rated them for sticking, and pressure mottling. The within-laboratory and between laboratory pooled standard deviations were found to be as follows:

Rating Parameter	Pooled Standard Deviations	
	Within-Laboratory	Between Laboratory
S	0.42	1.10
PM	0.38	1.06
Average	0.40	1.08

Based on these results the following criteria should be used in judging the acceptability of results at the 95 % confidence level.

8.1.1 *Repeatability*—Two results, each the mean of the two sets of panels, obtained by the same operator should be considered suspect if they differ by more than 1 to 2 units.

8.1.2 *Reproducibility*—Two results, each the mean of two sets of panels, obtained by operators in different laboratories should be considered suspect if they differ by more than 3.7 units.

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Standard Practice for Determination of Resistance of Factory-Applied Coatings on Wood Products to Stains and Reagents¹

This standard is issued under the fixed designation D 3023; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

- 1.1 This practice covers evaluation of clear factory-applied coating systems on wood substrates.
- 1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)²
D 333 Test Methods for Clear and Pigmented Lacquers³
D 2571 Guide for Testing Wood Furniture Lacquers³

3. Significance and Use

3.1 When used in conjunction with Methods D 333, this practice will provide a comprehensive evaluation of resistance to stains caused by chemical reagents and household chemicals.

3.2 This practice applies only to coatings applied in sufficient quantity to form a continuous film. It is recommended that the dry film thickness of the coating under test be reported.

3.3 Results from stain tests conducted in accordance with this practice distinguish differences between coatings.

4. Reagents

4.1 *Code for Applicability of Reagents*—V = Vertical, any surface that may be vertical as on a dresser front. H = Horizontal, any surface that may be horizontal as on a table top. L = Laboratory, any surface that may be used for laboratory furniture (Section 9).

4.2 Water:

4.2.1 *Tap Water*, V, H, L

4.2.2 *Boiling Water* (test in accordance with the Boiling Water Resistance section of Guide D 2571): H, L

4.3 Alcohol:

4.3.1 *Ethyl Alcohol* (test in accordance with the Alcohol Resistance section of Guide D 2571): V, H, L

4.4 Aliphatic:

4.4.1 *Mineral Spirits*: L (Specification D 235, Type III)

4.4.2 *Perchloroethylene*: V, H, L

4.5 Ketone:

4.5.1 *Methyl Ethyl Ketone*: L

4.6 Acetate:

4.6.1 *Amyl Acetate*: H, L

4.7 Inorganic Acid:

4.7.1 *Hydrochloric Acid (3 N)*—Dilute 258 mL of 12 N (36 %) HCl to 1 L: L

4.8 Organic Acid:

4.8.1 *Acetic Acid (3 N)*—Dilute 172 mL of 99 % acetic acid to 1 L: V, H, L

4.8.2 *Grape Juice* (unsweetened): V, H

4.8.3 *Lactic Acid (5 % solution)*: V, H

4.9 Bases:

4.9.1 *Unscented Mild Soap* (saturated solution): V, H, L

4.9.2 *Potassium Tripolyphosphate* (saturated solution containing 0.5 % sodium *N*-methyl-*N*-oleyl laurate (Igepon T-73): V, H, L

4.9.3 *Ammonium Hydroxide (3 % solution)*: L

4.9.4 *Trisodium Phosphate* (saturated solution): V, H, L

4.9.5 *Urea (6.6 % solution)*: V, H, L

4.10 Disinfectant:

4.10.1 *Cresols (5 % solution)*: V, H, L

4.11 Ink:

4.11.1 *Washable Ink*: V, H, L

4.11.2 *Permanent Ink*: V, H, L

4.11.3 *Ball-Point Ink*: V, H, L

4.12 Dye:

4.12.1 *Coffee* (test in accordance with the Coffee Stain Resistance section of Guide D 2571): V, H

4.13 Wax:

4.13.1 *Wax Crayons, Red, Blue, and Yellow*: V, H

4.13.2 *Lipstick* (test in accordance with the Cosmetic Stain section of Guide D 2571): V, H

4.14 Shoe Polish:

4.14.1 *Liquid Shoe Polish, tan*: V, H

4.15 Oil:

4.15.1 *Corn Oil or equivalent*: V, H

4.15.2 *Hair Oil*: V, H

4.16 Miscellaneous:

4.16.1 *Mustard*: V, H

4.16.2 *Tincture of Merthiolate (1+1000)*: V, H (see Section 5)

4.16.3 *Sodium Hypochlorite (6 % solution)*: V, H, L

4.16.4 *Tincture of Iodine*—Wet 50 g of potassium iodide (KI) with water, weigh into the wet KI 70 g of iodine, let stand 1 h to dissolve the iodine, and make up to 1 L with alcohol: V, H, L

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved March 25, 1988. Published September 1988. Originally published as D 3023 - 72. Last previous edition D 3023 - 81(1987).

² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 06.01.

4.17 *Staining Solutions*, unless otherwise specified, are water solutions.

5. Selection of Staining Agents for Testing Stain Resistance of Coatings

5.1 The producer and the user shall select staining agents from the reagents in Section 4. Selection of staining agents should depend on end use, such as wall, ceiling, horizontal surface, or floor.

5.2 In the absence of agreement between the producer and the user, the suggested staining reagents for each intended use are indicated in Section 4 by V, H and L.

5.3 It is not necessary that all finishes pass all tests.

6. Test Specimen

6.1 Test panels shall be of regular production finish.

6.2 If regular production finished panels are not available, the producer and the user shall agree upon the following variables: face veneer or wood surface, filler or filler stain, primer coat, topcoat, and dry film thickness for each coating material.

7. Panel Conditioning

7.1 Before testing, panels must be aged for a time and by a method agreed upon between the producer and the user.

8. Procedure

8.1 Conduct the test with panel surfaces horizontal at room temperature unless a different temperature is specified.

8.2 Place 0.5 mL of each staining agent on the finish surface and allow to stand uncovered for 18 h (or a time agreeable to the producer and the user) except for staining agents 4.3 to 4.6. On these put a 1-in. (25-mm) square of double-acid-washed quantitative filter paper. The purpose of the filter paper is to maintain a longer wet contact of volatile reagents with the surface of the finish.

8.3 After 18 h exposure, or other time period agreed to by the producer and the user, wash the surface with water, rinse with ethanol (1+1) and dry with a paper towel. Place the panel in a vertical position with a diffuse light source and view (Note) from a distance of 2 to 3 ft (0.6 to 0.9 m). Examine the surface for graying, spotting, wrinkling, discoloration, or other film defects and report.

NOTE—This method of viewing panels gives the best correlation of staining damage between laboratories. However, the following information should be noted: (1) When viewing panels across the grain at a low angle that is being varied, many more stains will appear visible than would appear visible perpendicular to the surface. These additional stains are superficial stains only. (2) When viewing with and against the grain, there appears to be an even greater number of superficial stains than would appear when viewing perpendicular to the surface or across the grain.

9. Interpretation of Results

9.1 Each product finish differs greatly in stain resistance requirements. The key, in general, is that the resistance to staining must be adequate for that finish's end use when first used in place and throughout the expected life span of the specific product.

9.2 The end use choice of resistance to staining should be agreed upon between the producer and the user.

10. Precision

10.1 Agreement between laboratories by visual comparison varied according to how samples were viewed.

10.1.1 Stains viewed perpendicular with a diffused light source at a distance of 2 to 3 ft (0.6 to 0.9 m) for graying, spotting, wrinkling, discoloration, or other film defects gave complete agreement between laboratories.

10.1.2 The same test stains viewed both with and across the grain at a low angle resulted in many more superficial stains being evident, depending upon the eyesight and judgment of the observer.

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Standard Guide for Testing Exterior Latex House Paints¹

This standard is issued under the fixed designation D 3129; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the selection and use of procedures for testing exterior latex house paints and trim paints in the laboratory and in the field. The test methods included are listed in Tables 1 and 2. All of these may not be required for each paint. Selection of the test methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between purchaser and seller.

1.2 This guide covers the testing of a ready-mixed latex house or trim paint for application by brush, roller, or spray on exterior surfaces.

1.3 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 215 Methods of Chemical Analysis of White Linseed Oil Paints³
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts³
- D 358 Specification for Wood to Be Used as Panels in Weathering Tests of Coatings³
- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings³
- D 523 Test Method for Specular Gloss²
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer³
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints³
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints³
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints³
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints³

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood³

D 1038 Definitions of Terms Relating to Veneer and Plywood⁴

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems³

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products³

D 1554 Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials⁴

D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature³

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³

D 1736 Test Method for Efflorescence of Interior Wall Paints³

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer³

D 2243 Test Method for Freeze-Thaw Resistance of Water-Borne Paints³

D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates³

D 2369 Test Method for Volatile Content of Coatings³

D 2574 Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms³

D 2805 Test Method for Hiding Power of Paints by Reflectometry³

D 3456 Practice for Determining by Exterior Exposure Tests the Susceptibility of Paint Films to Microbiological Attack³

D 3719 Test Method for Quantifying Dirt Collection on Coated Exterior Panels³

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings³

D 3928 Test Method for Evaluation of Gloss or Sheen Uniformity³

D 4214 Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films³

D 4287 Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer³

D 4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation³

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.42 on Architectural Finishes.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 3129 - 72. Last previous edition D 3129 - 85 (1990) ϵ 1.

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 04.09.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Method	Federal Test Method Std. No.
Sampling	5	D 3926	141
<i>Liquid Paint Properties:</i>			
Condition in container	6.1		3011
Coarse particles and foreign matter	6.2	D 185	
Density or weight per gallon	6.3	D 1475	
Nonvolatile	6.4	D 2369	
Fineness of dispersion	6.5	D 1210	
pH	6.6	E 70	
Consistency	6.7	D 562	
Rheological properties of non-Newtonian liquids	6.8	D 2196	
Freeze-thaw stability	6.9	D 2243, D 4287	
Microorganism resistance	6.10	D 2574	
<i>Paint Application and Film Formation:</i>			
Drying properties	7.1	D 1640	
Producing films of uniform thickness	7.2	D 823	
Application properties	7.3		4541
Application by brush	7.3.1		2141
Application by spray	7.3.2		2131
Touch-up uniformity	7.4	D 3926	
<i>Appearance of Dry Paint Film:</i>			
Color differences by visual evaluation	8.1	D 1729	
Color differences using instrumental measurements	8.2	D 2244	
Daylight directional reflectance	8.3	E 97	
Gloss 60°	8.4	D 523	
Hiding power	8.5	D 2805, D 344	
<i>Properties of Dry Paint Film:</i>			
Flexibility	9.1	D 522	
Moisture blister resistance	9.2	D 4585	
Fume resistance	9.3		
Exterior exposure resistance	9.4	D 1006	
Wood panel description	9.4.1	D 358	
Chalk resistance	9.4.2	D 659, D 4214	
Checking resistance	9.4.3	D 660	
Crack resistance	9.4.4	D 661	
Erosion resistance	9.4.5	D 662	
Flaking resistance	9.4.6	D 772	
Mildew resistance	9.4.7	D 3456	
Stain resistance	9.4.8		
Efflorescence resistance	9.4.9	D 1736	
Fade resistance	9.4.10		
Dirt pickup	9.4.11	D 3719	
Adhesion to chalky substrates	9.4.12		6301
Wet adhesion	9.4.13		6301
<i>Analysis of Paint:</i>			
Chemical analysis	10.1		
Volatile content	10.2	D 2369	7261
Pigment analysis	10.3	D 215	

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁵

E 97 Test Method for Directional Reflectance Factor 45-deg, 0-deg of Opaque Specimens by Broad-Band Filter Reflectometry⁶

⁵ Annual Book of ASTM Standards, Vol 15.05.

⁶ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

TABLE 2 Alphabetical List of Test Methods

Test Method	Section	ASTM Test Method	Federal Test Method Std. No.
Adhesion to chalky substrates	9.4.12		6301
Adhesion, wet	9.4.13		6301
Application by brush	7.3.1		2141
Application by spray	7.3.2		2131
Application properties	7.3		4541
Chalk resistance	9.4.2	D 4214	
Checking resistance	9.4.3	D 660	
Chemical analysis	10.1		
Coarse particles and foreign matter	6.2	D 185	4091
Color differences by visual evaluation	8.1	D 1729	4249
Color differences using instrumental measurements	8.2	D 2244	
Condition in container	6.2		3011
Consistency	6.3	D 562	
Crack resistance	9.4.4	D 661	
Daylight directional reflectance	8.3	E 97	
Density or weight per gallon	6.3	D 1475	
Dirt pickup	9.4.11	D 3719	
Drying properties	7.1	D 1640	
Efflorescence	9.4.9	D 1736	
Erosion resistance	9.4.5	D 662	
Exterior exposure resistance	9.4	D 1006	
Fade resistance	9.4.10		
Fineness of dispersion	6.5	D 1210	
Flaking resistance	9.4.6	D 772	
Flexibility	9.1	D 522	
Freeze-thaw stability	6.9	D 2243	
Fume resistance	9.3		
Gloss 60°	8.4	D 523	
Hiding power	8.5	D 2805, D 344	
Microorganism resistance	6.10	D 2574	
Mildew resistance	9.4.7		
Moisture blister resistance	9.2	D 4585	
Nonvolatile	6.4	D 2369	
pH	6.6	E 70	
Pigment analysis	10.3	D 215	
Producing films of uniform thickness	7.2	D 823	
Rheological properties of non-Newtonian liquids	6.8	D 2196, D 4287	
Sampling	5	D 3926	
Stain resistance	9.4.8		
Touch-up uniformity	7.4	D 3926	
Wood panel description	9.4.1	D 358	

E 105 Practice for Probability Sampling of Materials⁷

2.2 U.S. Federal Test Methods Standard No. 141:⁸

2131 Application of Sprayed Films

2141 Application of Brushed Films

3011 Condition in Container

4541 Working Properties and Appearance of Dried Film

6301 Wet Adhesion (Tape Test)

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this practice, refer to Definitions D 16, D 1038, and D 1554.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

4. Condition

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4. Conditions Affecting House Paint Topcoats

4.1 Practical requirements and performance of latex house paint may vary with:

4.1.1 Substrate type, such as type and quality of wood or hardboard, grain of wood, knots, pitch, etc., type and quality of masonry, ratio of cement to aggregate, alkalinity, etc.

4.1.2 Substrate weathering. Weathering of wood before painting will probably adversely affect the performance. Some weathering of masonry surfaces before painting may have beneficial effects on the performance.

4.1.3 The type, quality, and suitability of primer, undercoat, conditioner, etc., used under the topcoat and the time between coats.

4.1.4 Environmental conditions such as temperature at the time of application and during drying. As these paints contain water as a thinner, surfaces do not have to be completely dry before application. However, low temperature during drying may cause poor film formation.

4.1.5 Contaminants on the surface of the substrates.

4.1.6 Condition of previously painted substrates such as degree of chalk, adhesion of film, dirt, mold, general condition of substrate paint.

5. Sampling

5.1 Prior to sampling, the condition of the container should be established since damage to it may cause evaporation, skinning, or other undesirable effects in the coating. Determine the condition of the paint in accordance with 6.1 and 6.2.

5.2 Sample in accordance with Practice D 3925. Determine the weight per gallon in accordance with Test Method D 1475. Repeat this procedure until successive values agree within 0.1 lb (45 g) or as agreed upon between the purchaser and the seller. Samples for testing may then be taken.

5.3 Specify the amount of sample, the package sizes, and identification codes to assure a representative sample. A 1-gal (4-L) sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling plan, consult Practice E 105.

6. Liquid Paint Properties

6.1 *Condition in Container*—Thickening, settling, and separation are undesirable and objectionable if a paint cannot be reconditioned and made suitable for application with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties of paints after storage. Determine the condition in the container in accordance with Method 3011 of Federal Test Method Standard No. 141.

6.2 *Coarse Particles and Foreign Matter*—Paints must be free of coarse particles to form uniform films of good appearance, a typical maximum being 1 weight % of total paint. The specified test with a No. 325 (45- μ m) sieve gives the percent of these particles in a paint. Determine coarse particles and foreign matter in accordance with Test Method D 185.

6.3 *Density or Weight per Gallon*—The density as measured by weight in pounds per gallon or kilograms per litre is used to assure product uniformity from batch to batch. In the referenced method the density is expressed as the weight in pounds of 1 U.S. gal (kg/L) of the paint at a specified

temperature. Determine the density in accordance with Test Method D 1475.

6.4 *Nonvolatile*—The amount of nonvolatile in a coating is not necessarily an indication of performance. It is, however, useful for determining similarity of two samples. Determine nonvolatile solids in accordance with Test Method D 2369.

6.5 *Fineness of Dispersion*—The more finely a pigment is dispersed, the more efficiently it is being used. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the material down a calibrated, tapered groove in a hardened steel block with the groove varying in depth from 4 to 0 mils (100 to 0 μ m) and calibrated Hegman 0–8. The point at which continuous groupings of particles or agglomerates, or both, are visible through the surface of the liquid is taken as the fineness reading. Lower readings in mils or micrometres or higher readings in Hegman units indicate better fineness of dispersion. Determine fineness of dispersion in accordance with Test Method D 1210.

NOTE—The fast drying of latex paints makes it difficult to make accurate measurements of this type. Low-gloss paints do not generally require dispersion finer than a 1 to 2 Hegman reading.

6.6 *pH*—Latex paints with low (acidic) pH may corrode the container. Changes in pH during storage may indicate poor stability of a latex paint. Determine pH in accordance with Test Method E 70.

6.7 *Consistency*—Paints of a given type should fall within a stated consistency range for satisfactory reproduction of a specific formula. In the referenced method, consistency is defined as the load in grams required to produce a specified rate of shear. Although the consistency of most latex paints is about 150 to 300 g/100 revolutions, a much wider range is possible because of the wide variations in rheological properties of these paints. Also two paints of the same consistency may have quite different rheological properties. Determine the consistency of the product in accordance with Test Method D 562.

6.8 *Rheological Properties of Non-Newtonian Liquids*—Rheological properties are related to application and leveling properties of the liquid paint. The referenced methods cover the determination of rheological properties and are particularly suited for use with paints that display thixotropic characteristics. They actually measure viscosity under different shear rates. Determine rheological properties in accordance with Test Methods D 2196 or D 4287, or both.

6.9 *Freeze-Thaw Stability*—Water-based paints may be subjected to freezing conditions during shipping and storage. Suitably stabilized paints will resist several cycles of freezing and thawing without showing deleterious changes such as coagulation, graininess, or excessive viscosity increase. Many latex paints will increase in viscosity but can still be considered satisfactory if other properties which may be affected by a higher viscosity such as leveling and brushability are satisfactory. Determine the extent of change in several properties in accordance with Test Method D 2243.

6.10 *Microorganism Resistance*—Bacteria in a latex paint can cause gassing, putrefactive or fermentative odors, and loss of viscosity. Determine if the paint contains living

bacteria and if it is resistant to attack by bacteria in accordance with Test Method D 2574.

7. Paint Application and Film Formation

7.1 Drying Properties—Under average conditions most flat latex paints are dry-to-touch in 1 to 2 h when the water has evaporated from the film. Slow drying of the film may result in dust pickup, poor appearance and, when used on an exterior surface, rain or dew may cause a nonuniform appearance. They can usually be recoated from within a few hours to 18 h. Curing to obtain the ultimate properties may take only a few days for some latex paints, while others may require 1 to 2 weeks depending on composition. Determine drying time in accordance with Test Methods D 1640.

7.2 Producing Films of Uniform Thickness—Methods D 823 covers the preparation of coating films of uniform thickness essential in conducting tests.

7.3 Application Properties—Application properties of a paint are generally compared to a standard or described by requirements in the product specification. Determine application properties in accordance with Method 4541 of U.S. Federal Standard No. 141.

7.3.1 Brush Application—For proper method of application of a brushed film for test purposes refer to Method 2141 of U.S. Federal Test Method Standard No. 141.

7.3.2 Spray Application—House and trim paints are sometimes applied by spray. Determine the spray application properties in accordance with Method 2131 of U.S. Federal Test Method Standard No. 141.

7.4 Touch-Up Uniformity—After a paint has dried, areas where less material was applied sometimes become noticeable. If the paint has suitable touch-up properties, additional material can be applied to these areas only, instead of refinishing the complete wall. The color, gloss, and leveling of the touched-up areas and the previously coated area should be uniform. Differences in these properties are often caused by short wet-edge time, poor leveling on recoat, and pigment orientation or flotation during and after application. Determine touch-up properties in accordance with Test Method D 3928.

8. Appearance of Dry Paint Film

8.1 Color Differences by Visual Comparison—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, sizes of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color difference in accordance with Practice D 1729.

8.2 Color Differences Using Instrumental Measurements—The differences in color between a product and its standard can be measured by instrument. Generally, the tolerance is agreed upon by the purchaser and the seller and may also be required if a product specification is involved. Color instruments provide numerical values that can be compared to subsequent measurements. The referenced method covers the instrumental determination of small color differences, observable in daylight illumination, between nonfluorescent, nonmetameric, opaque surfaces such as paint specimens. If metamerism is suspected, visual evalua-

tion (8.1) should be used to verify the results. Calculate in accordance with Test Method D 2244 the color differences that have been measured instrumentally.

8.3 Directional Reflectance—This property is a measure of the appearance of lightness of a coating. It is usually assigned a value in specifications for white and pastel shades, a typical minimum reflectance being 86 % for white wall paint. In the referenced method, the direction of illumination and viewing is specified so as to eliminate the effect of gloss. Determine daylight directional reflectance in accordance with Test Method E 97.

8.4 Gloss 60°—Gloss level can affect dirt pickup and washability. Exterior latex house paints and trim paints may vary in gloss. A typical range would be from 5 to 60 units. Determine the specular gloss in accordance with Test Method D 523.

8.5 Hiding Power (Dry Opacity)—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness which is influenced by flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, opacity is evaluated visually as compared to a standard paint, and results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test that does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Determine hiding power in accordance with Test Method D 344 or D 2805.

9. Properties of Dry Paint Film

9.1 Flexibility—Elongation is a measure of flexibility of a paint film. Generally house paints will have no problem in passing a mandrel bend test. Most latex paints can be bent over a 1/4-in. (6.4-mm) mandrel without affecting the film. Determine elongation in accordance with Test Method D 522.

9.2 Moisture Blister Resistance—Moisture blister resistance is a necessary property of a house and trim paint. It can be evaluated by an accelerated laboratory test. Determine resistance to moisture blistering in accordance with Practice D 4585.

9.3 Fume Resistance—Fume resistance is the ability of a dry paint film to resist discoloration in a moist hydrogen sulfide atmosphere. This type of atmosphere may be present near industrial areas and can cause paint to yellow or darken in as little time as overnight exposure. There are no applicable ASTM or Federal test methods for fume resistance, but one method used in industry is as follows:

9.3.1 Apply a sufficient number of coats of the paint to two glass plates to hide the surface completely, allow to dry for 6 h, and expose one of the plates in a moist atmosphere of hydrogen sulfide for 18 h. Compare the color with the unexposed plate. The exposed plate should be no darker than the unexposed one.

9.4 Exterior Exposure Resistance—In conducting exterior exposures, refer to Practice D 1006.

9.4.1 Wood Panel Description—In establishing exposure

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performance, use the panels described in Specification D 358.

9.4.2 *Degree of Chalking*—Determine the rating using Test Method D 4214.

9.4.3 *Degree of Checking*—Determine the rating using Test Method D 660.

9.4.4 *Degree of Cracking*—Determine the rating using Test Method D 661.

9.4.5 *Erosion Resistance*—Determine the rating using Test Method D 662.

9.4.6 *Flaking Resistance*—Determine the rating using Test Method D 772.

9.4.7 *Mildew Resistance*—All exterior latex paints are subject to microbiological discoloration that will occur on the surface of the coating over a period of time. This is especially true in warm, moist climates. Determine mildew resistance using Practice D 3456.

9.4.8 *Stain Resistance*—There is neither an ASTM nor a Federal test method available for evaluating stain resistance of exterior latex paints. There are, of course, different kinds of stain such as the water-soluble extractives in wood substrates, the formation of lead or mercury sulfides, and run-down from metal surfaces of oxides that stain. Different kinds of test methods are required for the various stains.

9.4.9 *Efflorescence Resistance*—Exterior latex paints can contain sufficient solid water-soluble material to cause a noticeable deposit on the film through leaching by water and subsequent evaporation. Determine the tendency of the paint to effloresce in accordance with Test Method D 1736.

9.4.10 *Fade Resistance*—Exterior latex paints usually have good color retention because of their good chalk resistance. However, the use of improper latex, pigment volume concentration, or pigments can lead to fading. There are no applicable ASTM or Federal test methods for fade resistance. One method on the evaluation of chalk-fade has been published in the literature.⁹

9.4.11 *Dirt Pickup*—Low-gloss exterior latex paints generally have good resistance to dirt pickup. Gloss or semigloss latex paints may be more subject to this type of disfigurement. Exterior exposure particularly under an overhang will

⁹ Daiger, W. H., and Madson, W. H., "Chalk-Fade Evaluation of Pigmented Finishes by Use of Instrumentation and Computer Analysis," *Journal of Paint Technology*, Vol 39, No. 510, 1967, p. 399.

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indicate a paint's tendency for this property in a relatively short time (about 1 year). Determine degree of dirt collection in accordance with Test Method D 3719.

9.4.12 *Adhesion to Chalky Surfaces*—Latex paints generally have little ability to penetrate porous substrates. Therefore they do not adhere well to chalky substrates unless they are modified with some liquid that does penetrate and can bind a layer of chalk. (However certain latices do exist that are designed to adhere well to chalky surfaces. These could be considered without the need for a modifying liquid.) There are no directly applicable ASTM or Federal test methods for adhesion to chalky substrates. The industry generally uses a pressure-sensitive tape for testing this property. The tape is pressed firmly onto the dried latex film (fresh dry films do not adhere as well as aged dry films) and then removed rapidly by pulling back upon itself. Method 6301 of U.S. Federal Test Method Standard No. 141 describes a similar method but includes water exposure.

9.4.13 *Wet Adhesion*—Determine the wet adhesion of exterior latex paints in accordance with Method 6301 of U.S. Federal Standard Test Method No. 141.

10. Analysis of Paint

10.1 *Chemical Analysis*—If a specification requires certain raw materials or certain components in a given amount, then chemical analysis is required to determine whether the specified materials are present, and in what amounts. Analysis does not necessarily establish paint quality that can be also greatly affected by manufacturing techniques. Most ASTM analytical methods apply to solvent-reducible coatings. However, some of these can be adapted for analysis of water-reducible paints.

10.2 *Volatile Content*—The percent of volatile matter indicates the thinner loss from the film as it dries. This quantity subtracted from 100 % gives the nonvolatile content. Determine the volatile content in accordance with Test Method D 2369.

10.3 *Pigment Analysis*—The analysis of pigment may be required if the product is covered by a specification, or if it is agreed between the purchaser and the seller. Analyze the pigment in accordance with selected test procedures from Methods D 215.

11. Keywords

11.1 Exterior paints; house paints; latex paints



Standard Test Method for Quantitative Determination of Cellulose Nitrate in Alkyd Lacquers by Infrared Spectrophotometry¹

This standard is issued under the fixed designation D 3133; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout in March 1989.

1. Scope

1.1 This test method covers the quantitative determination of the content of cellulose nitrate (also known as nitrocellulose) in lacquers containing alkyd resins.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see Note 1.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1644 Test Methods for Nonvolatile Content of Varnishes²
- D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints²
- E 168 Practices for General Techniques of Infrared Quantitative Analysis³
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers³

3. Summary of Test Method

3.1 The method of standard additions is employed. Increments of cellulose nitrate, in solution, are added to aliquots of the sample. Absorbance measurements are made of the band at 848 cm^{-1} ($11.8\text{ }\mu\text{m}$) for each addition. The original content is then calculated from absorbance versus concentration.

4. Significance and Use

4.1 Coating compositions based on a mixture of synthetic resins and cellulose nitrate dissolved in organic solvents are quantitatively analyzed for the cellulosic derivative without isolating it. The test method is applicable to lacquers for which the grade of nitrocellulose is known and available. Other cellulose, alkyd resins, many vinyl resins, and solvents do not interfere. Components, such as acrylic resins

and some vinyl polymers, that absorb infrared near 848 cm^{-1} ($11.8\text{ }\mu\text{m}$) interfere with the determination. High boiling ester solvents, in particular methyl cellosolve acetate, may also interfere with the determination if not removed in the evaporation procedure (see 8.3).

5. Apparatus

5.1 *Infrared Spectrophotometer*, automatic recording, double-beam. Most infrared spectrophotometers operate from 4000 to 650 cm^{-1} (2.5 to $15\text{ }\mu\text{m}$), but in this test method only the range between 1000 and 750 cm^{-1} (10 to $14\text{ }\mu\text{m}$) is used. See Practices E 168.

5.2 *Absorption Cells*, sealed, with sodium chloride (NaCl) windows, 0.1-mm path length, one pair approximately matched.

5.3 *Film Vacuum Evaporator*, rotary thin or equivalent apparatus, to obtain redissolvable lacquer solids without decomposition of the cellulose nitrate.

5.4 *Oven*, vacuum drying, thermostatically controlled to operate at $65 \pm 2^\circ\text{C}$.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Cellulose Nitrate*, of the same grade as in the sample.

6.3 *N,N-Dimethylacetamide (DMA)*, spectrograde quality.

6.4 *Nitrogen Gas*, dry.

7. Standard Solution

7.1 Thoroughly dry a quantity of cellulose nitrate at 65°C , in the vacuum oven (Note 1). Transfer 4000 g , weighed to 0.1 mg , to a 200-mL volumetric flask. Add DMA, shake to dissolve the cellulose nitrate, and dilute to volume with DMA. One millilitre of solution is equivalent to 20 mg of

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Oct. 27, 1972. Published January 1973.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 14.01.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY., and the "United States Pharmacopeia."

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NOTE 1: Warning—Every precaution must be exercised in the handling and drying of cellulose nitrate because of its explosive nature. No more than 15 g should be dried at any one time.

8. Procedure

8.1 If the material is pigmented, remove the pigment quantitatively in accordance with Method D 2372, and proceed with the determination on the vehicle.

8.2 Determine the nonvolatile content of the clear lacquer, on the vehicle, in accordance with Test Methods D 1644.

8.3 Transfer, by any convenient means, 10.000 g of unpigmented material to a 50-mL volumetric flask. Attach the flask to the rotary evaporator and evaporate off the bulk of the solvents. Detach the flask, dissolve the lacquer solids in DMA, and dilute to volume with DMA. If the presence of methyl cellosolve acetate is suspected in the solvents, care must be taken to evaporate off the solvents as completely as possible.

8.4 Pipet 10.0 mL aliquots of this solution into each of three 25-mL volumetric flasks. To each, in turn, add 0, 5.0, and 10.0 mL of the standard solution. Dilute to volume with DMA.

8.5 Infrared Analysis:

8.5.1 Schedule the operating parameters of the infrared spectrometer to attain highly accurate absorbance values (refer to the directions of the manufacturer of the instrument). To accomplish this, set for lengthier scanning time and high signal-to-noise ratio, in accordance with Practice E 275.

8.5.2 Place one cell, containing DMA, in the reference beam. Place the other cell, filled in turn, with each of the solutions (Note 2), in the sample beam. Record the spectrum between 1000 and 750 cm^{-1} (10 and 14 μm) three times for each solution. Rinse the cell with DMA, and purge with dry nitrogen gas after each solution has been examined.

NOTE 2—To facilitate the transfer of the solutions to the cell, a small glass syringe fitted with a 3-in. (75-mm) hypodermic needle may be used.

8.5.3 Measure the difference in absorbance, ΔA , between

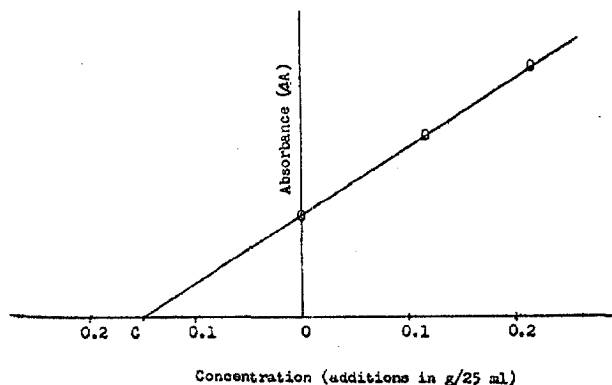


FIG. 1 Plot of ΔA versus Cellulose Nitrate Additions

the maximum absorbance at 848 cm^{-1} (11.8 μm) and the minimum at 813 cm^{-1} (12.3 μm). Determine the mean ΔA for the three runs on each solution.

9. Calculation

9.1 Plot the absorbance difference, ΔA , versus the cellulose nitrate additions and extrapolate the curve to zero absorbance. The intersection of the curve with the abscissa (concentration axis) gives the original cellulose nitrate content in the diluted sample. See Fig. 1.

9.2 *Alternative Method*—Solve for C in the three simultaneous equations derived from the expression for the Beer-Bourguier law, $A = abc$

$$\Delta A_0/(C + 0.0) = \Delta A_{0.1}/(C + 0.1) = \Delta A_{0.2}/(C + 0.2)$$

where:

C = original concentration of cellulose nitrate, in grams per 25 mL and the subscripts for ΔA correspond to the amounts of cellulose nitrate added in grams per 25 mL. Calculate the mean of C .

9.3 Calculate the weight percent of cellulose nitrate E in the original lacquer as follows:

$$E = (C \times D \times 100)/S$$

where:

D = dilution factor,

S = weight of specimen, and

C = grams of cellulose nitrate in 25 mL.

9.4 Calculate weight percent cellulose nitrate E in nonvolatile portion of original lacquer as follows:

$$E = (C \times 5 \times 100 \times 100)/(S \times N)$$

where:

N = nonvolatile content (see 8.2).

10. Precision

10.1 On the basis of a study in which four laboratories analyzed two different samples for percent of cellulose nitrate relative to the total samples, T , and relative to the nonvolatile matter, NVM , the within and between laboratory standard deviations were found to be:

Percent Relative to	Standard Deviations	
	Within	Between
T	0.14	0.12
NVM	0.39	0.64

Based on these standard deviations the following criteria should be used in judging the acceptability of results at the 95 % confidence level.

10.1.1 *Repeatability*—Two results obtained by one operator on one instrument should be considered suspect if they differ by more than the following:

Relative to	Percent
T	0.48
NVM	1.34

10.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than the following:

Relative to	Percent
T	0.71
NVM	2.51

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Standard Practice for Qualitative Identification of Polymers in Emulsion Paints¹

This standard is issued under the fixed designation D 3168; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Sections 6 to 10 were renumbered and Section 11 was added editorially in May 1990.

1. Scope

1.1 This practice describes a procedure for the qualitative identification in emulsion paints of most types of polymers present as major components of the paint vehicle. Limitations are discussed in Sections 5 and 10.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²

D1193 Specification for Reagent Water³

D2621 Test Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints²

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers⁴

3. Terminology

3.1 *Definitions*—For definitions of terms, refer to Definitions D 16.

4. Summary of Practice

4.1 The vehicle is extracted from the dried paint and after filtering is cast on a salt plate. The infrared spectrum is obtained and compared with reference spectra for identification of major components.

4.2 The paint is applied to a filament and pyrolyzed to depolymerize the vehicle. An internal standard is added and the pyrolyzate is separated into its components by gas-liquid chromatography. Monomers are identified by comparison of relative retention times.

5. Significance and Use

5.1 Identification of specific acrylic polymers in emulsion paints is often difficult or impossible by infrared alone. This

is particularly true when the acrylic is present in a small amount as a comonomer with vinyl acetate, or when blended with alkyds or other ester systems. If identification of an acrylic component is required in such a system, it may often be accomplished by gas-liquid chromatographic analysis of the pyrolyzed paint film. The presence of a number of other polymers may often also be confirmed by pyrolysis since they produce characteristic and reproducible pyrograms.

5.2 The pyrograms obtained from unknown samples vary in complexity according to the sample composition. It is necessary to establish the presence or absence of as many components as possible from a study of the infrared spectra obtained in the first part of this practice. The gas-liquid chromatography results may then be used to help identify any unknown components present and to confirm identifications made by infrared.

6. Interferences

6.1 Dibutyl maleate and dibutyl fumarate monomers are not successfully recovered by this procedure. If their presence is suspected as comonomer in a vinyl acetate copolymer system, *n*-butyl alcohol should be found in the pyrolyzate. This evidence, together with the absence of butyl acrylate or butyl methacrylate monomer peaks, is an indirect indication of the presence of one or both of these monomers.

6.2 The presence of relatively low quantities of copolymerized acids may not be successfully established by this procedure, due to some unavoidable decomposition of acrylate and methacrylate esters to acrylic or methacrylic acid and the corresponding alcohols during the pyrolysis. The infrared spectrum and an acid number determination, both run on the polymer solids, may usually be relied upon to establish the presence or absence of acid groups in the polymer.

6.3 In the case where the polymer being analyzed is simple (such as a vinyl acetate-alkyl ester copolymer, or a single acrylate-methacrylate copolymer), it might be advantageous to examine the total pyrolyzate directly by infrared (as a solution in carbon disulfide).

7. Apparatus

7.1 *Spectrophotometer*, recording, double-beam, infrared, with a range from at least 2.5 μm to 15 μm and a spectral resolution of at least 0.04 μm over that range. For checking the performance of the infrared spectrophotometer, see Practice E 275.

7.2 *Cell Mount*, demountable.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Oct. 25, 1985. Published December 1985. Originally published as D 3168 - 73. Last previous edition D 3168 - 73 (1979)^{ε1}.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁴ Annual Book of ASTM Standards, Vol 14.01.

7.3 *Halide Salt Crystals*, for use with demountable cell mount.

7.4 *Oven*, gravity or forced-draft, maintained at $105 \pm 2^\circ\text{C}$.

7.5 *Linear Programmed Temperature Gas Chromatograph*, equipped with a thermal conductivity detector.

7.6 *Pyrolysis Accessory*—Any suitable apparatus for achieving pyrolysis external to the chromatograph, that results in the recovery of sufficient pyrolyzate for identification purposes. The apparatus described in the Annex has been found to meet these requirements.

7.7 *Gas Chromatographic Column*, 10 ft (3 m) in length, $\frac{1}{4}$ in. (6.4 mm) in outside diameter copper tubing packed with 10 % silicone resin⁵ on 80 to 100-mesh acid-washed, dimethyl-dichlorosilane treated calcined diatomaceous earth.⁶

7.8 *Steam Bath or Low-Temperature Hot Plate*.

7.9 *Flask*, small Dewar.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

8.3 *Extraction Solvent Mixture*—Equal volumes of *o*-dichlorobenzene, dimethylformamide, and tetrahydrofuran.

8.4 *Liquid Nitrogen*.

8.5 *Petroleum Ether* (30 to 60°C boiling range) or heptane.

9. Procedure—Infrared Analysis

9.1 Transfer 1 to 2 g of emulsion paint to a 100-mL borosilicate glass beaker. Add approximately 2 mL of water and swirl or use a clean glass rod to spread the paint uniformly over the bottom of the beaker.

9.2 Place the beaker in an oven at $105 \pm 2^\circ\text{C}$ for at least 2 h, breaking up any skins that form so that complete drying is ensured.

9.3 Add 50 mL of water, cover with a watchglass, and place on a steam bath or low-temperature hotplate for 1 h. This process removes emulsifiers, protective colloids, and other water-soluble components that might interfere with interpretation of the infrared spectrum. Decant off the clear water layer and discard. If the addition of the distilled water

to the dried paint film produces a milky dispersion, the drying step in 9.2 was not complete and should be repeated.

NOTE 1—If there is an interest in characterization of the emulsifier system used, the water should be filtered through a fine-texture filter paper and taken to dryness. The solids may then be examined by infrared.

9.4 Dry for 15 to 20 min in an oven at $105 \pm 2^\circ\text{C}$. Add 50 mL of petroleum ether or heptane, cover with a watchglass, and bring to a slow boil for approximately 5 min. This process removes most emulsified plasticizers, oils, and other water insoluble, nonpolymeric organic materials, which might also interfere with the interpretation of the infrared spectrum. Decant off the solvent and discard.

NOTE 2—If there is an interest in characterizing the water-insoluble petroleum ether-soluble fraction, it should also be filtered through fine-texture paper, taken to dryness, and examined by infrared.

9.5 Add 10 mL of the extraction solvent mixture (8.3) and place on a steam bath or low-temperature hotplate for approximately 1 h. Filter, while still hot, through a fine-texture filter paper. If the polymer solution is viscous, it may be diluted further with extraction solvent mixture and warmed prior to filtration. Alternatively, the pigment may be removed from the polymer solution by centrifuging. Evaporate the solution on the steam bath or hotplate to a volume of 1 mL or less.

9.6 Place the concentrated polymer solution on a halide salt crystal and spread to form a uniform film. The thickness of the film should be such that when the infrared spectrum is recorded, the transmittance of the strongest band falls between 5 and 15 %. Dry the film in an oven at $105 \pm 2^\circ\text{C}$ for 1 h and cool in a desiccator.

9.7 Record the infrared spectrum from 2.5 to $15 \mu\text{m}$ so that a spectral resolution of $0.04 \mu\text{m}$ is maintained throughout that range. (Methods for achieving this resolution will vary according to the directions of the manufacturer of the instrument used.)

9.8 Compare the spectrum obtained with reference spectra prepared in a similar manner from emulsion paints of known composition or with published collections of polymer spectra such as Test Method D 2621. Other sources are listed in the references (1 to 7).⁸

10. Procedure—Pyrolysis-Gas Liquid Chromatography

10.1 Coat the resistance wire filament of the pyrolysis unit by dipping it into the original paint sample to be tested. Remove the volatiles either by drying the filament in an oven for about 30 min at $105 \pm 2^\circ\text{C}$ or by heating it electrically with the power supply set at 1 or 2 A (100 to 200°C) for approximately 10 min. If a larger volume of pyrolyzate is required than would be produced by the initial amount of sample deposited, additional layers of coating may be added by repeating the dipping and drying steps.

10.2 Place the coated filament-vial cap assembly in a small beaker. Extract with water, followed by petroleum ether or heptane, as outlined in 9.3 and 9.4.

10.3 Add a few drops of liquid nitrogen to the vial or flush with nitrogen gas to prevent oxidation reactions from

⁵ Silicone resin SE-30 produced by General Electric Co., has been found satisfactory for this purpose.

⁶ Chromosorb W, manufactured by Manville Sales Corp., Filtration and Minerals, P.O. Box 5108, Denver, CO 80217-5108, has been found satisfactory for this purpose.

⁷ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

⁸ The boldface numbers in parentheses refer to the list of references at the end of this practice.

occurring during the pyrolysis.

NOTE 3—If the infrared spectrum obtained in 9.8 shows poly(vinyl acetate) or poly(vinyl chloride) to be present, the pyrolysis step should be modified to prevent the acetic or hydrochloric acid formed from degrading other monomers that may be present in the pyrolyzate. Addition of a drop of concentrated ammonium hydroxide to the vial just prior to pyrolysis, neutralizes the acid as it is formed. The pyrolyzate may then be recovered by dissolving in diethyl ether just before sampling.

10.4 Tightly screw the coated filament-cap assembly onto the glass vial and connect the filament leads to the power supply.

10.5 Submerge the entire vial in liquid nitrogen contained in a small Dewar flask. Allow to equilibrate for several minutes, then apply a predetermined current for approximately 10 to 15 min.

NOTE 4—The optimum temperature which varies for different polymer types may be determined experimentally from known polymer systems. Six amperes (approximately 600°C) has been found to be close to optimum for the types tested. A study of the pyrolysis of acrylic polymers showed that heating times varying from 10 to 30 min produced no significant differences in the composition of the pyrolyzate.

10.6 Turn off the power supply and allow the filament to cool for several minutes. Remove the vial from the liquid nitrogen and substitute a standard screw cap for the filament-cap.

10.7 Using a syringe, add *n*-butyl acetate to the pyrolyzate equal to approximately 20% of the pyrolyzate volume.

10.8 Install the silicone resin column in the chromatograph and allow it to condition to 200°C. Set up the following conditions on the chromatograph:

Temperatures, °C	
Injection port	270
Detector block	270
Column (programmed):	
Initial	70
Final	200
ΔT, °C/min	6
Carrier gas:	
Flow rate, mL/min	helium
Detector current, mA	60
	150

10.9 Inject 5 μL of pyrolyzate into the chromatograph, engage the chart drive, and start the temperature programmer. Attenuate as necessary during the run.

NOTE 5—The use of the method described minimizes the tendency of pyrolysis products to undergo secondary reactions while still in the vapor state. This simplifies the chromatograms obtained, making interpretation easier.

TABLE 1 Relative Retention Times^a

Monomer or Alcohol	Relative Retention
Methanol	0.13
Ethanol	0.16
Acrylonitrile	0.25
Vinyl acetate	0.37
Methyl acrylate	0.45
Isobutanol	0.48
<i>n</i> -Butanol	0.57
Ethyl acrylate	0.66
Methyl methacrylate	0.72
Ethyl methacrylate	0.93
<i>n</i> -Butyl acetate	1.00
Isobutyl acrylate	1.15
<i>n</i> -Butyl acrylate	1.29
Styrene	1.33
Hydroxyethyl acrylate	1.38
Isobutyl methacrylate	1.43
Butyl methacrylate	1.55
Vinyl toluene	1.64
2-Hydroxyethyl methacrylate	1.64
2-Ethylhexanol	1.72
2-Ethylhexyl acrylate	2.31
2-Ethylhexyl methacrylate	2.56

^a Relative retention times listed are meant to serve as a guide for identification purposes. It should be understood, however, that the numbers listed are not absolute, and will vary somewhat from laboratory to laboratory and from instrument to instrument. For maximum accuracy in making identifications from retention time data, each analyst should prepare his own table by running the monomers of interest under the same conditions used for the analysis of the pyrolyzate.

10.10 Calculate retention times relative to the *n*-butyl acetate internal standard peak for each major peak obtained. Compare relative retention times obtained with known values for monomers of interest (see Table 1).

10.11 If any doubt remains concerning an identification based on the pyrogram obtained, it is recommended that the peak of interest be trapped as it is eluted from the chromatograph for subsequent positive identification by infrared spectroscopy or mass spectrometry.

10.12 Burn off any remaining organic residue by applying 8 or 9 A for a few seconds. Disconnect the filament from the power supply; then clean off any adhering pigment by gently brushing with a test tube brush while holding the filament under running water. The same filament may be reused many times.

11. Keywords

11.1 emulsion paints; gas chromatography; infrared analysis; paint vehicle; polymers; pyrolysis

ANNEX

(Mandatory Information)

A1. PYROLYSIS ACCESSORY

A1.1 Fig. A1.1 shows 12.5 in. (320 mm) of 20 gage Nichrome wire wound in a helix configuration. The characteristics of the filament have been chosen so that the current in amperes $\times 100$ closely approximates the temperature of the filament in degrees Celsius. Two 0.6-in. (15-mm) lengths of 0.05 in. (1.3 mm) outside diameter. Number 18 stainless steel hypodermic tubing is crimped onto the filament ends and inserted through snugly fitting holes drilled through the cap of a 1-dram (15 by 45-mm) vial (see drawing for detail). The hypodermic tubing, which functions as a heat sink to prevent pyrolysis of the plastic cap, may be firmly fastened to the cap by means of an epoxy cement.

A1.2 The tubing ends are connected by means of alligator clips to the leads of a suitable power supply.

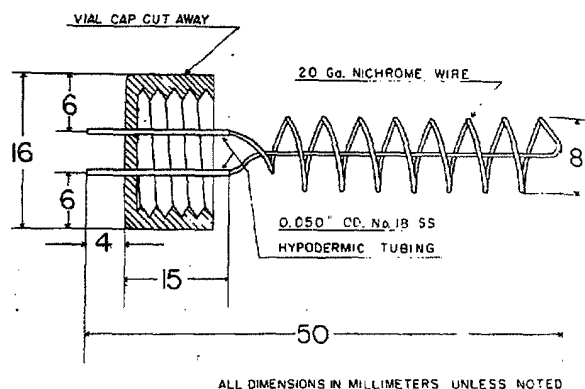


FIG. A1.1 Pyrolysis Accessory

REFERENCES

- (1) Chicago Society for Paint Technology, Infrared Spectroscopy Committee, *An Infrared Spectroscopy Atlas for the Coatings Industry*, Federation of Societies for Paint Technology, Philadelphia, PA, 1969.
- (2) Sadtler Commercial Infrared Spectra, Sadtler Research Laboratories, Inc., Philadelphia, PA.
- (3) Nyquist, R. A., *Infrared Spectra of Plastics and Resins*, 2nd Ed., Dow Chemical Co., Midland, MI, May 1961.
- (4) Haslam, J., and Willis, H. A., *Identification and Analysis of Plastics*, D. Van Nostrand Co., Inc., New York, NY, 1965.
- (5) Weinberger, L. A., and Kagarise, R. E., "Infrared Spectra of Plastics and Resins," *OTS Bulletin* No. PB-111438, U. S. Dept. of Commerce, May 1954.
- (6) Clark, G. L., Ed., *The Encyclopedia of Spectroscopy*, Reinhold Publishing Corp., New York, NY, 1960, p. 506-15.
- (7) Hummel, D. O., *Infrared Analysis of Polymers, Resins, and Additives. An Atlas Vol 1 Plastics, Elastomers, Fibers, and Resins*, Wiley Interscience, New York, NY, 1969.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Chipping Resistance of Coatings¹

This standard is issued under the fixed designation D 3170; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Keywords were added editorially in March 1991.

1. Scope

1.1 This test method covers the determination of the resistance of coatings to chipping damage by stones or other flying objects.

NOTE 1—This test method is similar to SAE J-400.

1.2 This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

D823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²

D1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

D1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

D1733 Method of Preparation of Aluminum-Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products³

D2201 Test Method for Preparation of Hot-Dipped Nonpassivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²

2.3 Other Documents:

Test for Chip Resistance of Surface Coatings (J-400)⁴
New Pictorial Standards Coating Defects⁵

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 3170 - 73. Last previous edition D 3170 - 74 (1980)^{e1}.

² Annual Book of ASTM Standards, Vol 06.01.

³ Discontinued, see 1980 Annual Book of ASTM Standards, Part 27.

⁴ Available from the Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096.

⁵ Available from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422-2350.

3. Summary of Test Method

3.1 Standardized road gravel is projected by means of a controlled air blast at the coated specimens. Generally the test conditions are made more severe by performing the test at low ambient temperatures. The resultant chipping effect is evaluated by comparison with a set of photographic standards that appear in the Pictorial Standards Coating Defects.

4. Significance and Use

4.1 Chipping of coatings, particularly on the leading faces and edges of automobile surfaces, is considered unacceptable by owners. In formulating a coating or coating system to meet service requirements, the resistance to chipping damage by flying objects such as gravel is one of the properties of importance since it can vary considerably as other properties are adjusted. Since resistance to chipping decreases at lower temperatures partly as the result of decreased flexibility, the test may be more directly related to service conditions by performing it at a low temperature. This test method is designed to produce a controlled amount of impact by the media on the coated panel in order to enhance reproducibility.

5. Apparatus

5.1 *Gravel-Projecting Machine*, constructed according to the design specifications shown in Fig. 1.⁶

5.2 *Gravel*—Water-eroded alluvial road gravel⁷ passing through a 3/8-in. (16-mm) space screen but retained on a 3/16-in. (9.5-mm) space screen. Note that mesh screen is not a substitute for a space-screen. It is important to remove the small pieces of gravel before reusing the gravel.

5.3 *Masking Tape*, 4 in. (100 mm) wide.⁸

5.4 *Temperature-Conditioning Equipment (alternatives):*

5.4.1 A cold room or freezer of sufficient size in which the gravel-projecting machine and test specimens can be maintained at the specified temperature of testing.

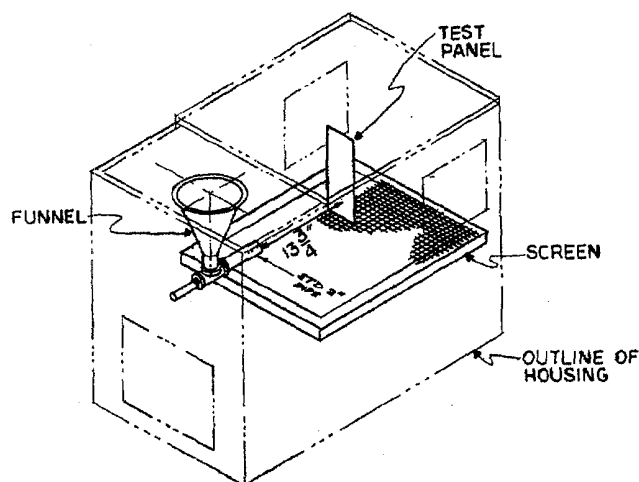
5.4.2 A freezer or cooler in which the test panels can be cooled 10°F (5°C) below the specified test temperature.

5.5 *Chipping Rating Standards*—A set of photographic standards depicting size and number of chips in each

⁶ A suitable apparatus meeting these specifications can be obtained from Q-Panel Co., 26200 First St., Westlake, OH 44135.

⁷ Gravel meeting these specifications can be obtained from Q-Panel Co., 26200 First St., Westlake, OH 44135.

⁸ No. 202-2 masking tape manufactured by the 3M Co., St. Paul MN 55101, has been found satisfactory for this purpose.



ISOMETRIC - COMPONENTS
SCALE 1"=1'-0"

FIG. 1 Gravel-Projecting Machine

category (see 2.3). The photographs in Fig. 2 are for illustration purposes only and should not be used for evaluation(s).

6. Test Specimens

6.1 The composition, surface preparation, and size of specimens shall be agreed upon between the purchaser and the seller. Test panels of 4 by 12 in. (102 by 305 mm) are most suitable for this test.

6.2 The number, type, method of application, and aging of coatings shall be agreed upon between the purchaser and the seller.

NOTE 2—Application, metal preparation, and film thickness measurement methods are given in the following ASTM methods: D 609, D 823, D 1005, D 1186, D 1400, D 1733, and D 2201.

7. Procedure

7.1 Condition the specimens for a minimum of 1 h at the specified test temperature in the equipment specified in 5.4. Make certain the test specimens are separated and have free access to the conditioning environment so that optimum heat transfer occurs.

7.2 Adjust the air pressure on the gravel apparatus to 70 ± 3 psi (480 ± 20 kPa) with the air valve open. Keep the lid to the gravel chamber on the apparatus closed during this operation as a safety precaution.

7.3 After adjusting the air pressure, shut off the air valve, open the lid to the gravel chamber and collect 1 pt (550 mL) of graded gravel (approx 250 to 300 stones) in a suitable container. Collect the gravel by scraping across the screen to allow fines to fall through.

7.4 Place the panel in the apparatus.

NOTE 3—the apparatus should be closed after the test begins.

7.5 After the apparatus is placed, place the panel in the apparatus.

7.6 After the gravel is collected, return the gravel to the chamber.

7.7 Shut the chamber, return to moisture level.

7.8 Cover the masking tape and pull off the tape.

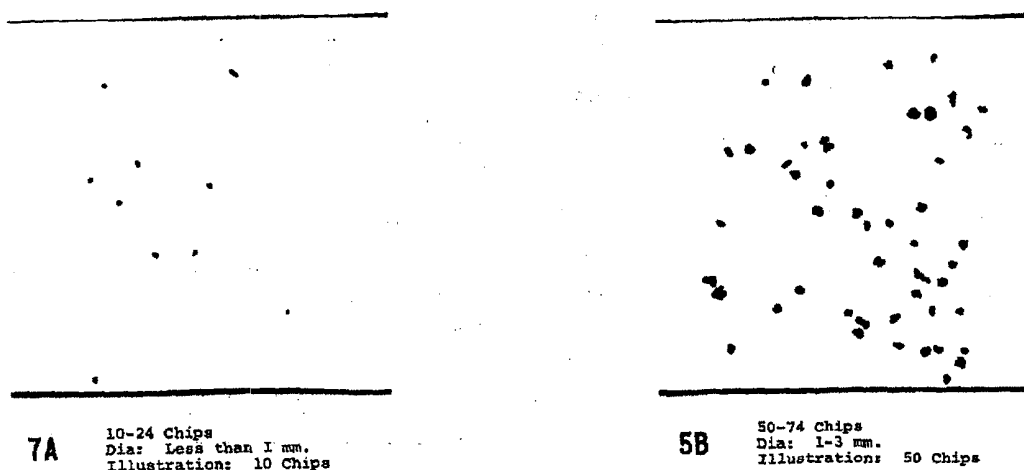


FIG. 2 Reference Photographs (Illustration Purpose Only)

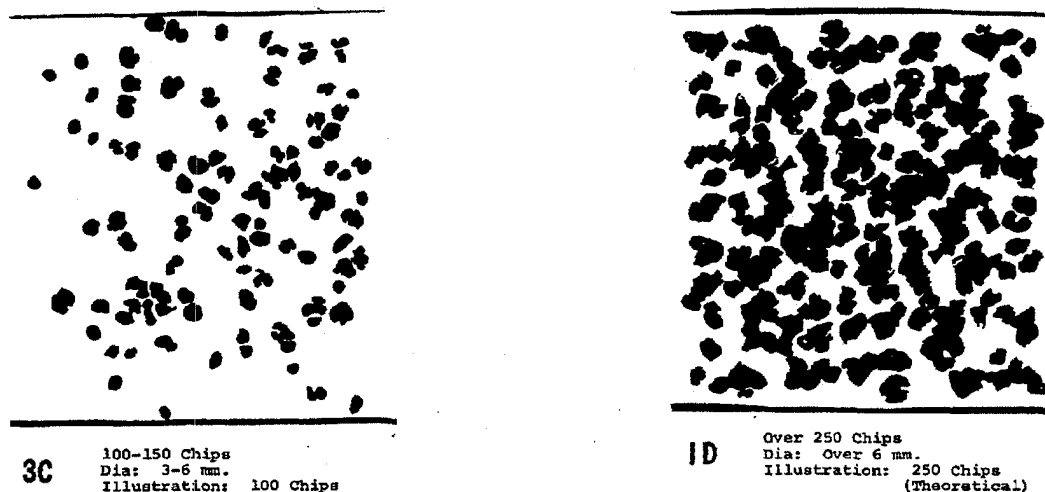


FIG. 2 Reference Photographs (Contd)

7.4 Place one test specimen at the desired test temperature in the panel holder with the coated side facing the front of the apparatus and close the lid to the gravel chamber.

NOTE 3—Due to space limitations, it may not be possible to operate the apparatus at the desired test temperature. In this event, the panels should be cooled below the desired test temperature by 10°F (5°C) and the test begun within 10 s of removal from the cooler.

7.5 *Automatic Gravel Feed*, with automatic feed apparatus, place one pt of the gravel in the vibrating hopper. Opening the air valve automatically feeds the gravel into the air stream over approximately a 10-s interval.

7.6 *Manual Gravel Feed*, with a manual feed, slowly pour the gravel into the funnel over approximately a 10-s interval.

7.7 Shut off the air valve, open the lid to the gravel chamber, and remove the tested panel. Allow the panel to return to room temperature and remove any condensed moisture by wiping dry with a cloth.

7.8 Cover the test panel with a 4-in. (100 mm) strip of masking tape. Press the tape down firmly by rubbing; then pull off slowly to remove any loose chips of paint.

7.9 Determine the degree of chipping by comparison with the photographic standards.

8. Report

8.1 Report the substrate composition, type and age of coating, test temperature, and chipping rating.

9. Precision

9.1 Since the rating scale consists of a combination letter and number, no standard deviation value is obtainable from the supporting test results. It is the judgment of those familiar with this test method that the following precision statements are representative:

9.1.1 *Repeatability*—Results of tests within a laboratory differing by more than one letter or number unit should be considered suspect.

9.1.2 *Reproducibility*—Results of tests between laboratories differing by more than two letters or number units should be considered suspect.

10. Keyword

10.1 chip resistance

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Standard Test Method for Porosity of Paint Films¹

This standard is issued under the fixed designation D 3258; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1}NOTE—Editorial changes were made throughout in May 1987.

1. Scope

1.1 This test method covers the determination of the porosity of a white or near white mineral spirits insoluble paint film to indicate the degree to which a subsequent coat will penetrate.

1.2 The texture of the film can affect cleanup that will influence the results of the test. A stain applied to a high-hiding paint will not lower the reflectance as much as the same stain applied to a low-hiding paint of equal porosity. These points must be considered in comparing different paints.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry²

3. Summary of Test Method

3.1 The test paint is applied to a nonporous surface, air dried, then measured for reflectance. A special colored penetrating medium is applied, the excess removed in a specified manner, and reflectance measured again. The difference between the two readings indicates the degree of porosity; the smaller the difference, the lower the porosity of the film and the greater its ability to resist penetration.

4. Apparatus and Materials

4.1 *Film Applicator*, 6 in. (150 mm) wide with a clearance of 12 mils (300 μ m).³

4.2 *Film Applicator*, 5½ in. (140 mm) wide with a

clearance of 10 mils (250 μ m).⁴

4.3 *Reflectometer*, meeting the requirements of Test Method E 97.

4.4 *Vacuum Drawdown Plate*.

4.5 *Camelhair Brush* (approximately ½ in. (13 mm) wide).

4.6 *Plastic Wash Bottle*, containing odorless mineral spirits (about 16 oz (500 mL)).

4.7 *Test Panel*—Smooth nonporous panel such as sealed white, cardboard chart, or white plastic sheet.

4.8 *Penetrating Medium*—A composition of pigment and dark-colored dye dispersed in organic liquid vehicle.⁵

4.9 *Filter Paper*.

5. Procedure

5.1 Place the test panel on the vacuum plate and apply the paint under test with the 12-mil (30- μ m) clearance drawdown blade. Remove the test panel from the vacuum plate and air dry 48 h under standard conditions of 73 $\pm$ 3.5°F (23 $\pm$ 2°C) and 50 $\pm$ 5 % relative humidity.

5.2 Determine the percent reflectance of the dry film using the green filter on the reflectometer. When making this reading, back up the panel with a flat rigid opaque object and mark the area on which the reading was made.

5.3 Place the panel containing the dry paint film on the vacuum plate. Using the 10-mil (25- μ m) clearance blade, apply the penetrating medium over the test paint, covering the area marked as above and drawing the applicator blade in the same direction as before.

5.4 After 5 min $\pm$ 15 s wash off excess penetrating medium. To do this, hold the panel vertically and remove the medium using the camelhair brush wet with odorless mineral spirits. Repeat the process until most of the excess is removed. Then continue to remove more of the excess by applying mineral spirits directly from the wash bottle to the area above the stain so that it flows across the stained area. Pause to observe the bead that forms at the bottom of the panel. If the bead is not clear, continue washing with mineral spirits until the bead is essentially clear. Test with filter paper to be sure no dye remains in the bead.

5.5 Hang the panel in a vertical position and air dry for 3 h. As before, read the percent reflectance over the same area

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 14, 1980. Published December 1980. Originally published as D 3258 - 73. Last previous edition D 3258 - 79.

² *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

³ Bird film applicators or their equivalent have been found satisfactory for this purpose. Available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

⁴ The Dow latex film caster has been found satisfactory for this purpose. Available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

⁵ Available from: K & N Laboratories, Inc., 5331 Dansher Rd., Countryside, IL 60525.

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6. Report

6.1 Report the difference between the percent reflectance of the untested film and that of the penetrated film.

7. Precision

7.1 On the basis of an interlaboratory study of this test method in which one operator in each of ten laboratories tested paint films of two nominally different levels of porosity, the within-laboratory and between-laboratories standard deviations were found to be:

Nominal Difference in Reflectance, %	Standard Deviations	
	Within-Laboratory	Between-Laboratories
7	0.27	1.26
35	0.60	4.22

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Based on these standard deviations the following criteria should be used to judge the precision or results at the 95 % confidence level:

7.1.1 *Repeatability*—Duplicate results, obtained by a single operator, should be considered suspect if they differ by more than 0.8 % relative for difference in reflectance levels of about 7 % and more than 1.8 % relative for difference in reflectance levels of about 35 %.

7.1.2 *Reproducibility*—Two results, each the mean of duplicate measurements obtained in different laboratories should be considered suspect if they differ by more than 4.0 % relative for difference in reflectance levels of about 7 % and 14 % relative for difference in reflectance levels of about 35 %.

8. Index Terms

8.1 This test method is indexed under the following terms: porosity; resistance—penetration (into paint films).

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Standard Practice for Infrared Determination of the Temperature of Applied Coatings on Wood Products During the Curing Cycle¹

This standard is issued under the fixed designation D 3259; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Section 5 was editorially changed in May 1990.

1. Scope

1.1 This practice is intended to serve as a guide in measuring with infrared instruments the temperature during the curing process of coatings applied to wood products.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 The forest products finishing industry has encountered difficulties in measuring the temperature of painted surfaces during the curing process. The use of thermocouples is not entirely satisfactory because the thermocouple wires tend to conduct heat away too rapidly from the area where the temperature is being measured. Infrared radiation thermometers that are simple to operate can circumvent this difficulty. After calibration they are aimed at the surface, switched on, and the temperature read directly from an indicating gage.

NOTE 1—Temperature-sensitive crayons, papers, and pellets may be successfully used to measure only the highest temperature reached by painted surfaces during the curing cycle.

2.2 There are several different types of infrared radiation thermometers, including those based on lead sulfide or thermistor sensors and those that are simple thermal voltaic transducers. As such they respond to different wavelengths of infrared radiation and have different areas of applicability. Only instruments that have been evaluated are included in this practice.

3. Apparatus

3.1 *Lead Salt Types²* (Note 2)—Two lead salts are commonly used. Lead sulfide that responds to wavelengths between 1.0 and 3.0 μm and lead selenide that responds to wavelengths between 15 and 4.5 μm .

3.2 *Thermistor Type*—Thermistors are sensitive to longer wavelength infrared of 3 to 20 μm . Instruments may be

designed with a narrow or broad band pass and may compensate for changes in ambient temperatures. One instrument³ with the latter feature has a band pass of 8 to 14 μm . Another instrument⁴ that must be rezeroed to compensate for changes in ambient temperatures has a band pass of 3 to 20 μm . However, this instrument can be completely portable and requires essentially no warm up time.

3.3 *Thermalvoltaic Type⁵*—These instruments are equipped with collection chambers for radiated energy that then heat a thermocouple to the temperature of the surface measured. As such, they require a minimum target area of 1 in. (25 mm) in diameter and have a delay time that averages about 1.5 s.

3.4 Instrumentation is available for on-line process monitoring control of product temperatures. These units have long term stability that permits operation in varying ambients encountered in plants or mills without periodic calibration. The instruments also have output signals to operate recorders and controllers.

4. Procedure

4.1 *Calibration*—Calibrate each instrument according to the instructions of the manufacturer. A standard blackbody capable of being controlled at various temperatures is almost essential for calibration. One such blackbody⁶ is a modified hot plate with a 1/2-in. (13-mm) thick aluminum plate and a ventilated cowling to minimize the effects of ambient drafts. A dial thermometer with its stem within the aluminum plate serves to monitor the temperature of the blackbody.

4.2 Operation:

4.2.1 Detailed instructions on the operation of each instrument are not included in this practice. Unless otherwise agreed follow the manufacturer's instructions in operating the equipment.

4.2.2 One general precaution is that the instrument must observe only the surface being measured and not stray radiation from infrared heaters, sunlight, electric lights, or other sources. The permissible distance from the surface to the sensor depends upon the area of surface, the viewing

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of D01.52 on Factory Coated Wood Products.

Current edition approved Aug. 31, 1984. Published January 1985. Originally published as D 3259 - 73. Last previous edition D 3259 - 73.

² The Williamson 1200 manufactured by the Williamson Development Co., Inc., West Concord, MA 01781, is an example of this type.

³ Optitherm II Model 8014 manufactured by Barnes Engineering Co., Stamford, CO 06904, is an example of this type of instrument.

⁴ Raynger R2LT manufactured by Raytek, Inc., Santa Cruz, CA 95060, is an example of this type of instrument.

⁵ Kustotherm B distributed by Epic Inc., New York, NY 10038, is an example of this type of instrument.

⁶ Field Calibration Source, Model 11-101 manufactured by Barnes Engineering Co., Stamford, CO 06904.

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angle of the instrument and the wavelength that the sensor responds to. Instruments operating only in the long wavelength region of the infrared spectrum are affected much less by stray radiation and by color variations.

5. Keywords

5.1 cure temperature; infrared; paint surface temperature; temperature measurement; wood coating

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Standard Test Method for Acid and Mortar Resistance of Factory-Applied Clear Coatings on Extruded Aluminum Products¹

This standard is issued under the fixed designation D 3260; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the evaluation of the mortar and acid resistance of clear protective coating factors applied to extended aluminum substrates.

2. Referenced Documents

2.1 ASTM Standards:

C 207 Specification for Hydrated Lime for Masonry Purposes²

D 823 Test Method for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

3. Summary of Test Method

3.1 Panels that have been buffed, cleaned, neutralized, and coated with the material being evaluated are exposed to freshly prepared mortar and to a hydrochloric acid solution.

4. Significance and Use

4.1 This test method will provide differentiation between types of coatings for acid and motor resistance providing the coating is applied in a uniform continuous film with a specified film thickness.

5. Reagents

5.1 *Acid*—Ten volume percent solution of commercial grade hydrochloric acid (31.4 to 31.8 % HCl, 20°Bé) in distilled water.

5.2 *Cleaning Solution*—An alkaline cleaner⁴ in the concentration recommended by the manufacturer, usually 6 to 8 oz/gal (45 to 60 g/L) of water. The pH of the solution should be between 11.4 and 12.2.

5.3 *Lime*—Building lime complying with Specification C 207.

5.4 *Neutralizing Solution*—Prepare from a stable free-flowing, granular material having a chromate base⁵ as recommended by the supplier, usually at a concentration of

12 to 16 oz/gal (90 to 120 g/L) of water.

5.5 *Sand*—Dry sand capable of passing through a 10-mesh wire screen.

6. Test Panels

6.1 Panels of extruded aluminum alloy No. 6063-T5 with dimensions 6 by 3 by ¼ in. (150 by 75 by 6.3 mm) shall be used for the test.

7. Procedure

7.1 Preparation of Test Panels:

7.1.1 Buff the aluminum panels and remove the buffing compound with an organic solvent.

7.1.2 Immerse the panels in cleaning solution (5.2) for 1 h at a temperature of 200°F (93°C).

7.1.3 Rinse the panels in tap water having a pH of 6.5 to 7.5 until the surface is free of water break. Use reagent water for rinsing if the pH of the tap water is outside the specified limits. The panels shall be free from evidence of corrosion or surface attack.

7.1.4 Immerse the panels in neutralizing solution (5.4) at 75 ± 2°F (24 ± 1°C) for 30 s to neutralize any retained alkalinity and to promote adhesion of the applied coatings.

7.1.5 Rinse the panels in reagent water and allow them to dry a minimum of 1 h before coating.

7.2 Coating of Test Panels:

7.2.1 Apply the coatings being evaluated to the test panels in a uniform continuous film by automatic spray equipment in accordance with Method A of Test Method D 823.

7.2.2 For the mortar test, spray two panels on one side with two or more full wet coats to produce a minimum dry film thickness of 0.6 mils (15 µm) and two panels with one or more full wet coats to produce a minimum dry film thickness of 0.3 mils (7.5 µm).

7.2.3 For the acid test, spray two panels with two or more full wet coats, on both sides, to produce a minimum dry film thickness of 0.6 mils (15 µm) and two panels with one or more full wet coats on both sides to a minimum dry film thickness of 0.3 mils (7.5 µm).

7.2.4 Allow the panels to dry for 7 days at 73 ± 3.5°F (23 ± 2°C) and 50 ± 5 % relative humidity.

7.3 Mortar Test:

7.3.1 Prepare freshly mixed mortar by dry mixing 75 g of building lime (5.3) and 225 g of sand (5.5) and adding sufficient reagent water while stirring to make a soft paste that can be formed into a firm mortar patty approximately 1½ in. (40 mm) in diameter and ¼ in. (6 mm) thick.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Oct. 29, 1982. Published January 1983. Originally published as D 3260 - 73. Last previous edition D 3260 - 73 (1978).

² Annual Book of ASTM Standards, Vol 04.01.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Diversey No. 808 has been found satisfactory for this purpose.

⁵ Diversey No. 814 has been found satisfactory for this purpose.

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7.3.2 Apply the mortar to the top and bottom areas of the test panels and, without drying, immediately expose them for 7 days at 95 to 98 % relative humidity at a temperature of $70 \pm 3.5^\circ\text{F}$ ($21 \pm 2^\circ\text{C}$) in an automatically controlled cabinet.

7.3.3 At the end of 7 days take the panels from the test cabinet and immediately dislodge the mortar patties from the panels using a damp cloth to remove any residue.

7.3.4 View the surface of the panels perpendicular to the surface under a diffused light source providing a minimum light intensity at the surface of 200 ft-candles (2150 lx). Examine for blistering, peeling, lifting, crazing, flaking, or discoloration of the coating, and for corrosion of the coated part of the aluminum panel. Three observers shall evaluate each panel and the majority assessment shall govern.

7.4 Acid Test:

7.4.1 Apply a mixture of 50 parts beeswax and 50 parts

paraffin to protect the raw edges to a distance of $\frac{3}{8}$ in. (10 mm).

7.4.2 Immerse the panels in the acid bath (5.1) at $70 \pm 3.5^\circ\text{F}$ ($21 \pm 2^\circ\text{C}$) for 6 h. Remove the panels, rinse in water, and allow to dry.

7.4.3 Examine and evaluate as in 7.3.4.

8. Precision

8.1 The effect of acid and mortar on the coatings is expressed in terms of appearance and numerical values are not generally assigned.

8.2 In a cooperative test program, consistent results were obtained by all cooperators when all of the test variables were controlled closely. The success of the procedure depends particularly on having a continuous film of the coating on the panels, the proper pH value when rinsing the panels, and proper protection of the edges of the panels in the acid test.

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Standard Practice for Direct Injection of Solvent-Reducible Paints Into a Gas Chromatograph for Solvent Analysis¹

This standard is issued under the fixed designation D 3271; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes the techniques used to inject whole paint samples directly into a gas chromatograph to obtain a chromatogram from which the solvent composition may be established.^{2,3}

1.2 This practice is not designed to be quantitative.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific hazard statement is given in 6.1.*

2. Referenced Document

2.1 *ASTM Standard:*

E 260 Practice for Packed Column Gas Chromatography⁴

3. Summary of Practice

3.1 A suitable aliquot of whole paint is injected, by means of a syringe, into a gas chromatographic column in order to separate the solvents.

4. Significance and Use

4.1 Gas chromatographic separation of solvents present in whole paints is the preferred first step for identifying and quantitating solvent compositions, using auxiliary procedures and techniques.

5. Apparatus

5.1 *Gas Chromatograph*—Any instrument with temperature programming capability may be used. It should be equipped with either a thermal conductivity or flame ionization detector (see Practice E 260).

5.2 *Recorder*—A 1 to 10-mV recorder with a full-scale response time of 2 s or less and a maximum noise of $\pm 0.03\%$ of full scale.

5.3 *Column*—The resolution of the column must be such that under the operating conditions selected the distance from the base line to the depression between two adjacent peaks must be not more than 50 % of the smaller peak. Columns may be either packed or capillary.

6. Reagents and Materials

6.1 *Carrier Gas*—Helium or hydrogen for use with thermal conductivity detector units; and nitrogen, helium or argon for use with flame ionization detector units.

NOTE 1: Precaution—When hydrogen is used, special precautions should be taken to prevent gas leakage from causing a possible explosion.⁵

6.2 *Column Packing Material*, meeting requirements in 5.3. The following materials have been used satisfactorily:

6.2.1 Polyethylene glycol, molecular weight 20 000,⁶ and diisodecyl phthalate as liquid phases on a solid support of 60 to 80 mesh (250 to 175 μm) diatomaceous earth.

6.2.2 Porous beads of ethylvinylbenzene and divinylbenzene copolymer,⁷ 60 to 80-mesh (250 to 177- μm) have also been successfully used as column material.

6.3 *Liquid Charging Devices*, such as micro syringes.⁸ Disposable type is preferred.

6.4 *Septum Sample Vials*, PTFE-fluorocarbon-faced.⁹

7. Procedure

7.1 Protect the injection port from the nonvolatile portion of the paint by using a borosilicate glass injection port sleeve (Note 2), glass wool plug, or by any other suitable means. If the whole paint sample is of sufficiently low viscosity, draw an aliquot directly into the syringe. When the whole paint sample is too viscous, dilute with a suitable solvent (Note 3) that will not interfere with the analysis. In order to minimize solvent losses due to evaporation, the whole paint sample

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved June 26, 1987. Published August 1987. Originally published as D 3271 - 73 T. Last previous edition D 3271 - 76(1981)⁶¹.

² New York Society for Paint Technology, Technical Committee 76, "The Application of Gas Chromatography to the Analysis of Coating Solvents," *Journal of Paint Technology*, Vol 40, No. 516, January 1968, pp. 33-48.

³ Esposito, G. G., and Swann, M. H., "Direct Analysis of Solvents in Lacquer by Programmed Temperature Gas Chromatography," *Official Digest Federation Society Paint Technology*, Vol 33, No. 440, September 1961, pp. 1122-1131.

⁴ *Annual Book of ASTM Standards*, Vol 14.01.

⁵ Consult Pamphlet G-5, available from the Compressed Gas Association, Inc., 500 5th Ave., New York, NY 10036.

⁶ Carbowax 20M, available from Union Carbide Corp., 270 Park Ave., New York, NY 10017, has been found suitable for this purpose.

⁷ Porapak Q, available from Waters Associates, Inc., 34 Maple St., Milford, MA 01757, has been found suitable for this purpose.

⁸ Disposable syringes, available from Precision Sampling Corp., P.O. Box 15886, Baton Rouge, LA 70895, have been found suitable for this purpose.

⁹ Vials, available from Pierce Chemical Co., P.O. Box 117, Rockford, IL 61105, have been found suitable for this purpose.

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should be placed in a PTFE-fluorocarbon-faced septum sample bottle prior to being drawn into the syringe. Proceed with normal techniques to obtain a chromatogram of the solvent or solvents present in the sample.

NOTE 2—Consult manufacturer of particular instrument used.
NOTE 3—Ethyl ether and methylene chloride have been found satisfactory for most sample types.

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Standard Practice for Vacuum Distillation of Solvents From Solvent-Reducible Paints For Analysis¹

This standard is issued under the fixed designation D 3272; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} NOTE—Editorial changes were made throughout, including the title, in February 1988.

1. Scope

1.1 This practice covers a vacuum distillation procedure that separates the solvents from the nonvolatile portion of paints so they may be analyzed.

1.2 An alternative procedure for the analysis of solvents in paints is described in Practice D 3271.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 3271 Practice for Direct Injection of Solvent-Reducible Paints into a Gas Chromatograph for Solvent Analysis²

3. Summary of Practice

3.1 The sample of whole paint with added tricresyl phosphate is heated by means of a silicone oil bath. The distillation is allowed to proceed for a period of time and then vacuum is applied. Finally dry air is passed through the system to facilitate the removal of any high-boiling solvents present.

4. Apparatus

4.1 Apparatus³ is shown in Fig. 1. It is essential in assembling this apparatus, that the volume of the portion preceding the flask be kept to a minimum by using diameters of connections and distances as small as possible. Include a gage in the assembly for measuring pressure.

4.2 *Vacuum Pump*, capable of pulling 2 mm Hg or less of vacuum pressure.

4.3 *Septum Sample Vial*, PTFE-fluorocarbon-faced.⁴

4.4 Safety Shield.

5. Reagents

5.1 *Methylene Chloride or Acetone.*

5.2 *Silicone Oil.*⁵

5.3 *Tricresyl Phosphate.*

6. Procedure

6.1 Immerse the receiver in a bath containing dry ice with methylene chloride or acetone of sufficient depth that the liquid reaches to the bottom level of the rubber stopper.

6.2 Pour approximately 15 g of the whole paint sample into the 50-mL distillation flask. Immediately follow with 10 mL of tricresyl phosphate and without delay mix the contents of the flask.

6.3 Assemble the apparatus and close the needle valve and stopcock attached to the capillary tubing. Immerse the distillation flask by raising a silicone oil bath preheated to 155 to 160°C (Warning—see Note 1) until it reaches the side arm (Note 2). Allow the distillation to proceed for 15 min without vacuum.

NOTE 1: Warning—Care should be taken to prevent the temperature of the silicone oil exceeding 160°C because of possible formation of explosive mixtures of air and solvents from the sample being analyzed.

NOTE 2—The liquid level in the oil bath should be as high as is practical.

6.4 After 15 min apply vacuum slowly and carefully to avoid bumping or rapid distillation until 2 mm Hg is reached. This step should take approximately 10 to 15 min. Next, carefully open the stopcock. After bubbling subsides, adjust the needle valve so as to regulate the flow of air at a rate of 60 to 80 bubbles per minute (at the bubble counter). Pass air through the system for a total of 30 min while keeping the dry ice bath at a minimum low temperature.

6.5 At the end of the 30-min period release the vacuum very slowly with air flowing to prevent back-up of the paint. Remove the receiver containing the solvents and warm the contents only to the temperature of cold running tap water. Transfer the isolated solvents promptly to an airtight septum faced vial for subsequent analysis.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Material.

Current edition approved Oct. 29, 1976. Published December 1976. Originally published as D 3272 - 73 T. Last previous edition D 3272 - 73 T.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Obtainable from SGA Scientific, Inc., 735 Broad St., Bloomfield, NJ 07005.

⁴ Septum vials available from Precision Sampling Corp., Baton Rouge, LA 70315, have been found satisfactory for this purpose.

⁵ Silicone oil 710 available from Dow Corning Corp., Midland, MI has been found satisfactory for this purpose.

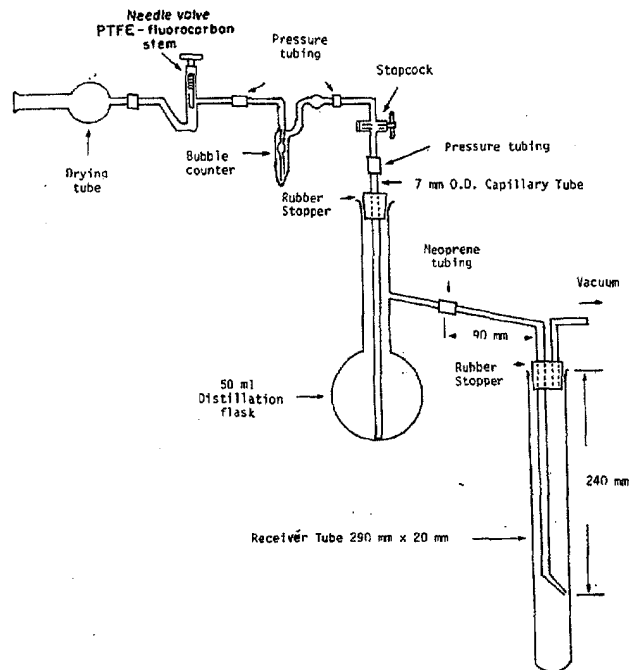


FIG. 1 Distillation Apparatus

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Standard Test Method for Resistance to Growth of Mold on the Surface of Interior Coatings in an Environmental Chamber¹

This standard is issued under the fixed designation D 3273; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{ε1} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This test method describes a small environmental chamber and the conditions of operation to evaluate reproducibly in a 4-week period the resistance of a paint film to surface mold growth in a severe interior mold environment. The apparatus is designed so it can be easily built or obtained by any interested party and will duplicate results obtained in a large tropical chamber.

1.2 Temperature and humidity must be effectively controlled within the relatively narrow limits specified in order for the chamber to function reproducibly during the short test period. Severity and rate of mold growth on a film is a function of the moisture content of both the film and the substrate. A relative humidity of 95 to 98 % at a temperature of $90 \pm 2^\circ\text{F}$ ($32.5 \pm 1^\circ\text{C}$) is necessary for test panels to develop rapidly and maintain an adequate moisture level to support mold growth.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 3274 Test Method for Evaluating Degree of Surface Disfigurement of Paint Films by Microbial (Fungal or Algal) Growth or Soil and Dirt Accumulation²

3. Significance and Use

3.1 An accelerated test for determining the resistance of interior coatings to mold growth is useful in estimating the performance of coatings designed for use in interior environments that promote mold growth and in evaluating compounds that may inhibit such growth and the aggregate levels for their use.

3.2 This test method should preferably be used by persons who have had basic microbiological training.

4. Apparatus

4.1 *Environmental Chamber*, capable of maintaining a relative humidity of 95 to 98 % at a temperature of $90 \pm 2^\circ\text{F}$ ($32.5 \pm 1^\circ\text{C}$) while providing a continuous inoculation of the surface of exposed panels with mold spores. The chamber should be kept in a room controlled to 70 to 75°F (21 to 24°C) so that heat loss from the cabinet is insignificant and that 95 to 98 % relative humidity is readily obtained at the test temperature. Alternatively the cabinet must be insulated with suitable materials to minimize heat loss.

4.2 *Small Cabinet*, suitable for holding about twenty-five 3 by 4-in. (75 by 100-mm) test panels under these conditions can be constructed as follows (Fig. 1):

4.2.1 *Tank*, polypropylene or polyethylene, with an offset shoulder at the top rim is used as the chamber.³ A pitched top with straight sides should be constructed out of acrylic plastic so moisture condensation will run down the sides and be recirculated instead of dripping onto the panels.

4.2.2 *Heating Coil*,⁴ 125-W, regulated by a controller⁵ and installed in the bottom of the chamber by connections through the end wall. It is so placed that it is immersed when there are 2 to 3 in. (50 to 75 mm) of water in the bottom of the chamber. A 90°F (32.5°C) thermostat should be installed in the chamber in the area by the test panels to regulate the heat input through the controller to the heating coil in the water.

4.2.3 *Tray*, stainless steel or plastic, approximately 1 in. (25 mm) smaller than the inside dimensions of the chamber and 1 to 3 in. (25 to 75 mm) deep with a noncorrodible metal⁶ mesh bottom should be supported 1 in. (25 mm) above the water level and centered in the chamber. One layer of fine plastic or fiberglass screen should be placed over the metal mesh for holding soil.

4.2.4 *Small Fan*,⁷ mounted through the end wall perpen-

³ Dynalab Corp., Rochester, NY, and Cole Parmer Instrument Co., Chicago, IL have tanks of this type available in dimensions approximating 27 by 18 and 18 in. (686 by 457 and 457 mm) high.

⁴ The Chromalox® heating coil manufactured by the Wiegand Div., Emerson Electric Co., Pittsburgh, PA has been found satisfactory for this purpose.

⁵ The Lumenite Electronic Level Control has been found satisfactory for this purpose.

⁶ Monel has been found satisfactory for this purpose.

⁷ 1/250-hp fan operating at 300 r/min with a 1.5-in. blade, obtained from Dayton Electric Manufacturing Co., or equivalent, has been found satisfactory for this purpose.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.28 on Biodeterioration.

Current edition approved May 30, 1986. Published July 1986. Originally published as D 3273 - 73 T. Last previous edition D 3273 - 82^{ε1}.

² Annual Book of ASTM Standards, Vol 06.01.

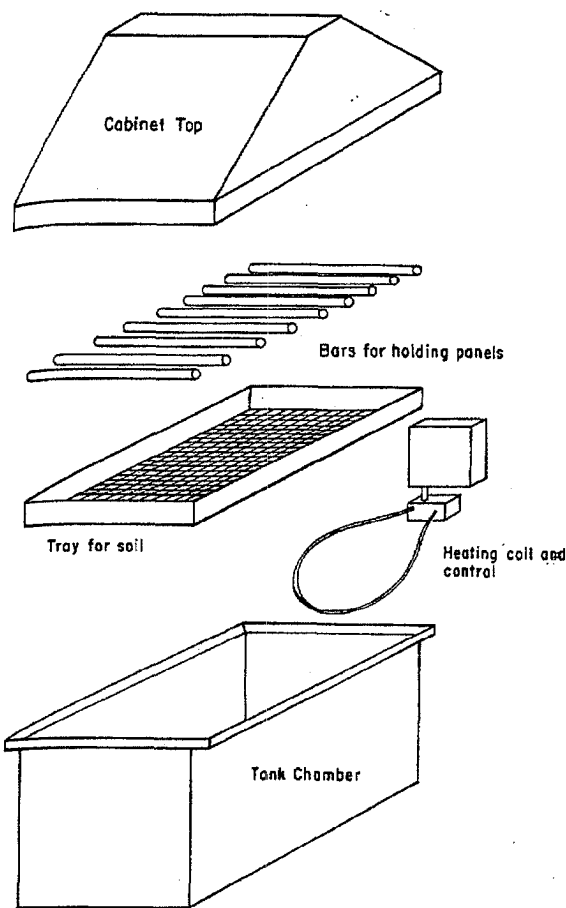


FIG. 1 Environmental Cabinet Assembly

pendicular to and just above the surface of the soil bed in the tray (4.2.3) to provide dispersion and circulation of the mold spores to achieve continuous inoculation of the surfaces of the painted panels.

4.2.5 *Series of Wood Bars*, suspended across the width of the chamber at a height and spacing that allows the use of test panels 3 by 4 in. (75 by 100 mm), hung vertically, with approximately 3-in. (75-mm) clearance above the inoculated soil with a suitable method of fastening. Screw eyes are used with the wooden panels while a wire frame or a large clip is used with the gypsum board panels. Other support systems may be utilized.

NOTE 1—Other angles of exposure may be used but may alter the rate and severity of mold growth.

4.3 *Psychrometer*, for measuring relative humidity in the test area.

5. Reagents and Materials

5.1 *Soil*—A good quality greenhouse-grade potting soil, suitable for plant propagation, containing 25 % peat moss. pH of the soil should fall from 5.5 to 7.6. Do not allow soil to become compacted.

5.2 *Cultures*:

5.2.1 *Aureobasidium pullulans*,⁸ ATCC 9348

5.2.2 *Aspergillus niger*,⁸ ATCC 6275

5.2.3 *Penicillium*,⁸ Sp. 12667 or ATCC 9849

5.3 *Test Panels*:

5.3.1 *Ponderosa Pine* (*Pinus ponderosa* Laws) *Sapwood Panels*, ½ in. (12.7 mm) thick, 3 by 4 in. (75 by 100 mm), free of excessive resins, knots, growth rings or other abnormalities, surfaced smooth on four sides. Wood shall be kiln dried after sawing to avoid infestation of wood-rotting fungi and any wood showing evidence of such infestation shall be eliminated as test material. Wood shall be weighed after conditioning at room temperature in a dry room to 15 % moisture content. Calculated weight shall fall between 6.0 and 7.0 g/in.³ (365 and 425 kg/m³). Panels containing heartwood areas should not be used as they will inhibit mold growth under test conditions.

5.3.2 *Gypsum Board Panels*, ½ in. (12.7 mm) thick, 3 by 4 in. (75 by 100 mm).

NOTE 2—Other substrates may be used but may result in a different rate or severity of mold growth.

6. Preparation of Apparatus

6.1 Place greenhouse soil in the tray in the cabinet and add water to the tank chamber to the desired depth. Allow the cabinet to equilibrate for 24 h before inoculating the soil with the specified mold suspensions.

6.2 Prepare mold slants of all three cultures and age 10 to 14 days or purchase prepared mold slants⁸ of all three cultures. Prepare mold suspensions from each type of mold slant by the following procedure: Add one drop of 25 % nonionic surfactant⁹ solution to 95 to 100 mL sterile deionized or distilled water and shake. Pipet 5 mL of this solution onto the mold slant. Scrub the surface of the slant with a sterile cotton swab to remove as much spore and mycelial growth as possible without digging up the surface of the agar. Pour the water from the scrubbed slant back into the surfactant-sterile water mixture for dilution. Shake gently for 15 to 20 min to break up clumps of mold. Use a pipet to distribute the mold suspensions evenly over the surface of the greenhouse soil in the tray in the cabinet.

6.3 Allow two weeks of continuous operation for the mold to sporulate and equilibrate with the environment before starting a test. It should not be necessary to recontaminate continually the chamber of panels after sufficient microorganism growth has built up in the soil, if the chamber is maintained in continuous operation.

6.4 Viability of the mold growth in the cabinet can be checked by placing several malt agar or potato dextrose agar plates,¹⁰ open and face up, at several locations on the panel support rods. After 1 h, cover plates and place in incubator at 90 ± 2°F (32.5 ± 1°C) for 3 days. If an incubator is not available, leave the covered plates in the cabinet. Mold

⁸ Suitable cultures can be obtained from American Type Culture Collection, 12301 Parklawn Dr., Rockville, MD 20852. Cultures can be maintained on malt agar or potato dextrose agar. Prepared slants can be obtained from Difco Laboratories, Inc., Detroit, MI 48232; Baltimore Biological Laboratories, Baltimore, MD 21218 or from equivalent sources.

⁹ Triton X100, Rohm & Haas, Igepal CA-630, G.A.E., or equivalent.

¹⁰ Prepared agar plates can be obtained from Difco Laboratories, Inc., Detroit, MI 48232; Baltimore Biological Laboratories, Baltimore, MD 21218, or from equivalent sources.

growth should be medium-heavy to heavy and cover the complete surface of the agar plate.

7. Procedure

7.1 Preparation of Test Panels—Wear disposable plastic gloves or utilize other techniques when handling panels to avoid fingerprints. Prepare triplicate panels by applying two coats of the material under test to both faces and to all edges of the panels at a spreading rate of approximately 450 ft²/gal (11 m²/L) per coat or as specified by the coating manufacturer, allowing 1 day between coats unless otherwise specified. Condition the panels at 73.5 ± 3.5°F (23 ± 2°C) and 50 ± 5 % relative humidity for 4 days after application of the last coat before placing in the test chamber for start of environmental exposure.

7.2 Exposure—Hang the panels vertically with the bottom approximately 3 in. (75 mm) above the surface of the inoculated soil and with sufficient spacing to allow free circulation of air and to prevent contact between panels or with wall surfaces. Place triplicate panels randomly in the cabinet. Include uncoated control panels in all tests. If the cabinet is operating properly, unpainted panels should develop a 4 to 6 mold growth rating within 2 to 3 weeks. If this growth is not obtained, the cabinet conditions are not satisfactory or there is some interfering treatment on the panel.

7.3 Rating—Rate the panels for mold growth each week for 4 weeks on a 0 to 10 rating scale using photographic standards (Test Method D 3274). The 50 and 100 magnification photographic standards for types of fungal or dirt disfigurement (Test Method D 3274) should be used to confirm the presence of mold as the cause of discoloration.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

8. Report

8.1 Report the results at the end of the 4-week exposure giving the mean and range of the three panels. The result from any panel that differs by more than 2 rating units from either of the others can be considered manifestly faulty and discarded and the mean of the remaining two panels reported. If all panels in a set differ by more than 2 units in their ratings, discard all results and repeat the test.

9. Precision and Bias

9.1 On the basis of an interlaboratory study of this test method in which one operator, in each of seven laboratories, rated resistance to mold growth of interior paints at seven levels of resistance by visual comparison with photographic standards, the standard deviations within and between laboratories were found to be:

	Standard Deviations
Within laboratories	0.4 units
Between laboratories	0.7 units

Based on these deviations the following criteria should be used to judge the precision of results at a 95 % confidence level:

9.1.1 Repeatability—Two ratings, each the mean of a set, obtained by a single operator should be considered suspect if they differ by more than 1 unit.

9.1.2 Reproducibility—Two ratings, each the mean of a set, obtained in different laboratories should be considered suspect if they differ by more than 2 units.

9.2 Bias—No bias has been determined for this test method.

10. Keywords

10.1 environmental chamber; mold; surface interior coatings



1. Scope

1.1 This test method covers the determination of mold growth on interior paint surfaces.

1.2 This test method is applicable to the determination of mold growth on interior paint surfaces.

2. Significance and Use

2.1 This test method is used to determine the mold growth on interior paint surfaces and to compare the results with the mold growth on control surfaces.

3. Terminology

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1. This standard is related to D01.28. Current edition published.



Standard Test Method for Evaluating Degree of Surface Disfigurement of Paint Films by Microbial (Fungal or Algal) Growth or Soil and Dirt Accumulation¹

This standard is issued under the fixed designation D 3274; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{ε1} NOTE—Editorial changes were made throughout, including the title, in October 1988.

1. Scope

1.1 The photographic reference standards available for use with this test method provide a numerical basis for rating the degree of fungal and algal growth or soil and dirt accumulation on paint films.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 The growth of fungi and algae in and on the surface of paint films represents a major cause of discoloration or disfigurement of painted surfaces. Because of their dark pigmentation, it is frequently difficult to distinguish fungi and algae from soil or dirt particles. The use of magnification of as much as 100 $\times$ affords easy distinguishability among fungal spores, fungal mycelia, algae, and dirt and is recommended in cases where such difficulty is apparent.

3. Types of Fungal and Algal Growth

3.1 Two types of fungal growth may be observed:

3.1.1 *Spores*—Spherical bodies, gray black in color, observed singly or in clusters. May or may not be associated with fungal mycelia. Spores are the reproductive form of fungi. Spores of *Pullularia pullulans* magnified 100 $\times$ are shown in Fig. 1.

3.1.2 *Hyphae*—Thread or filament structures, gray to black in color, that make up the mycelium or vegetative form of fungi. Hyphae may appear alone or with spores attached. Hyphae of *Pullularia pullulans*, magnified 50 $\times$ are shown in Fig. 2.

3.2 Amorphous growth typical of an alga such as *Chlorococcum* Sp. appears in Fig. 3 at a magnification of 39 $\times$. Terrestrial algae can appear green to dark brown in color.



FIG. 1 Spores of *Pullularia pullulans*, Magnified 100 $\times$

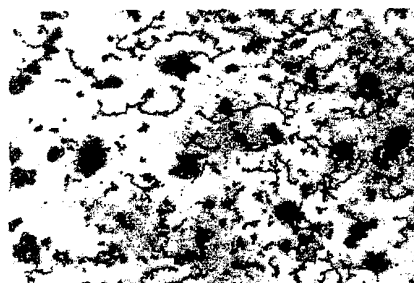


FIG. 2 Hyphae of *Pullularia pullulans*, Magnified 50 $\times$

4. Soil or Dirt Particles

4.1 Soil or dirt particles may range from light brown to black. The particles, viewed at 50 $\times$ to 100 $\times$ magnification, are generally amorphous and are frequently translucent. Soil particles on paint magnified 100 $\times$ are shown in Fig. 4.

5. Use of Photographic Standards²

5.1 The photographic reference standards that are part of

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.28 on Biodeterioration.

Current edition approved Oct. 29, 1982. Published December 1982. Originally published as D 3274 - 73 T. Last previous edition D 3274 - 76.

² Available from ASTM Headquarters (Request PCN 12-432740-00). Copies of the pictorial photographic standards are contained in the publication *Pictorial Standards of Coating Defects*, which may be obtained from the Federation of Societies of Coating Technology, 492 Norristown Rd., Blue Bell, PA 19422.

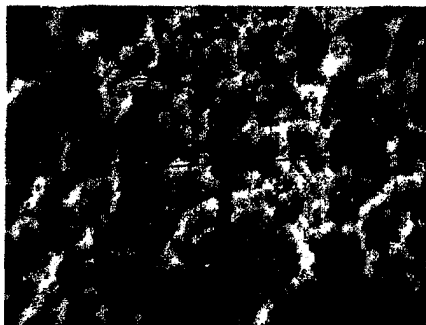


FIG. 3 Amorphous Algal Growth, Magnified 39x

this test method are for illustration purposes only and should not be used for evaluation.

5.2 The photographic standards illustrated in Fig. 5 rate the disfigurement of paint films from 0 to 8. A rating of 10 would indicate a film totally absent of disfigurement by particulate matter.

6. Procedure

6.1 Compare each specimen with the photographic standards associated with this test method to determine which most closely matches the degree of disfigurement on the specimen. When the degree is intermediate between two adjacent standards select the intermediate odd number as the disfigurement rating.

6.2 The density of fungal or algal growth as well as dirt



FIG. 4 Soil Particles on Paint, Magnified 100x

accumulation may vary over the painted surface being evaluated. This variation may be the result of the location of the surface (that is, under an eave of a house *versus* an exposed area), nonuniformity of the substrate, film thickness or other factors. Such variation should be noted when reporting the rating assigned to specimen under test. In examining test specimens for fungal, algal, or dirt disfigurement, the possibility of failure due to the wood substrate should be recognized and reported.

6.3 When microbial disfigurement is detected (or established), distinguish between algal and fungal growth. For the latter, estimate whether spores or mycelia predominate.

7. Report

7.1 Report the numerical disfigurement rating as defined in 5.1 and the predominant type, that is, fungi, algae, or soil.

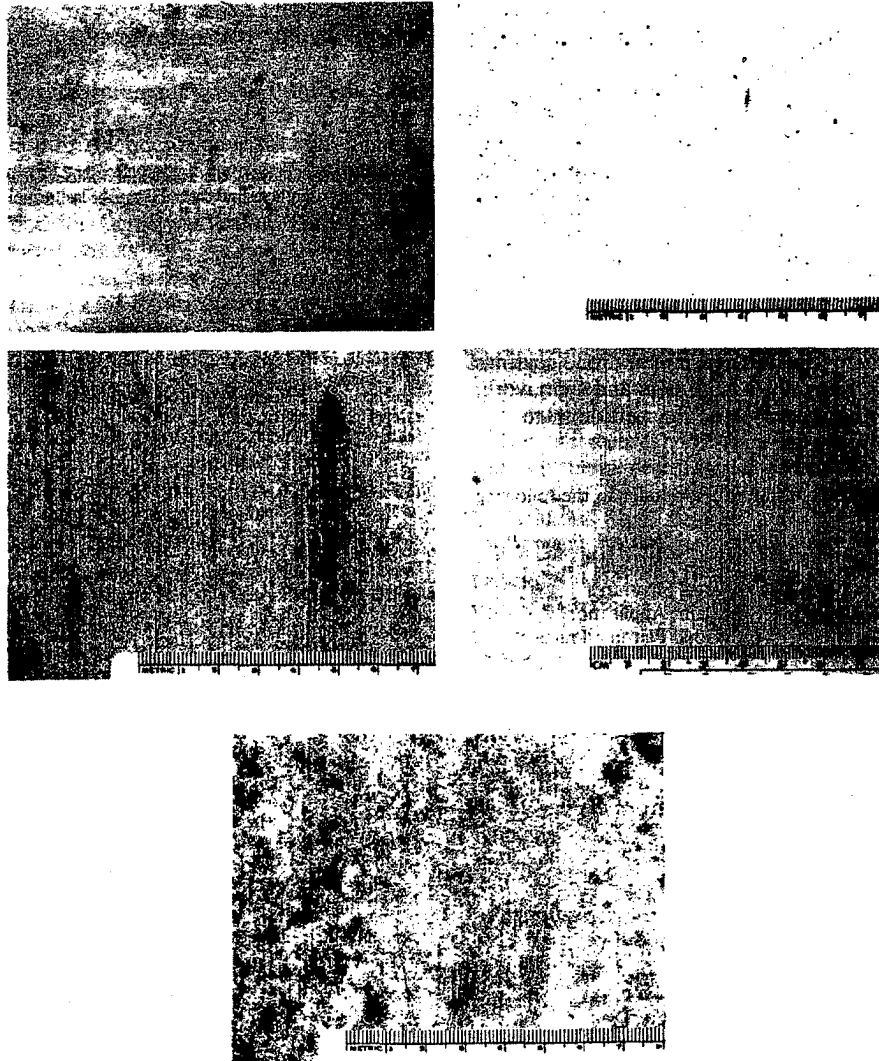


FIG. 5 Disfigurement Ratings

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Standard Guide for Painting Inspectors (Metal Substrates)¹

This standard is issued under the fixed designation D 3276; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide is intended as an information aid to painting inspectors in carrying out their task efficiently. It includes the key elements of surface preparation, coatings application, and final approval for both field and shop work. The items should be selected that are pertinent to the specification of a particular job.

NOTE 1—For additional helpful information, refer to the following documents:

*Manual of Coating Work for Light-Water Nuclear Power Plant Primary Containment and Other Safety-Related Facilities*²
*New Concepts for Coating Protection of Steel Structures*³
 SSPC-PA Guide 3 A Guide to Safety in Paint Application⁴
 Steel Structures Painting Manual Vol 1 Good Painting Practice⁴
 Steel Structures Painting Manual Vol 2 Systems and Specifications⁴
 Manufacturers Specifications and Instructions (made available to the inspector for reference to special requirements for proper application)
 Material Safety Data Sheets (needed to ensure that personnel take necessary precautions in handling hazardous materials).

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.3 This guide is arranged in the following order:

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Steel Structures Painting Council Publications	2.3
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¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

Current edition approved Sept. 29, 1986. Published November 1986. Originally published as D 3276 - 73. Last previous edition D 3276 - 73.

² ASTM, 1979.

³ ASTM STP 841, ASTM, 1984.

⁴ Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

2. Referenced Documents

2.1 ASTM Standards:

D1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁵

⁵ Annual Book of ASTM Standards, Vol 06.01.

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- D 1186 Test Methods for Nondestructive Measurement of Dry-Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁵
- D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings⁵
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base⁵
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁵
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁶
- D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting⁵
- D 2200 Pictorial Surface Preparation Standards for Painting Steel Surfaces⁵
- D 3359 Test Methods for Measuring Adhesion by Tape Test⁵
- D 3843 Practice for Quality Assurance for Protective Coatings Applied to Nuclear Facilities⁵
- D 4212 Test Method for Viscosity by Dip-Type Viscosity Cups⁵
- D 4414 Practice for Measurement of Wet Film Thickness by Notch Gages⁵
- D 4417 Test Methods for Field Measurement of Surface Profile of Blast Cleaned Steel⁵
- D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers⁵
- E 376 Practice for Measuring Coating Thickness by Magnetic-Field or Eddy-Current (Electromagnetic) Test Methods⁷
- 2.2 Occupational Safety and Health Administration (OSHA) Standard:
Hazard Communication⁸
- 2.3 Steel Structures Painting Council Standards:⁴
SSPC-SP 1 Solvent Cleaning
SSPC-SP 2 Hand Tool Cleaning
SSPC-SP 3 Power Tool Cleaning
SSPC-SP 5 White Metal Blast Cleaning
SSPC-SP 6 Commercial Blast Cleaning
SSPC-SP 7 Brush-off Blast Cleaning
SSPC-SP 10 Near-White Blast Cleaning
SSPC-PA 1 Paint Application Specifications
SSPC-PA 2 Measurement of Paint Thickness with Magnetic Gages
SSPC-Guide to Vis 1 Pictorial Surface Preparation Standards for Painting Steel Surfaces
SSPC-Paint 27 Basic Zinc Chromate-Vinyl Butyryl Wash Primer

3. Significance and Use

3.1 This guide is intended as a reference for those concerned with the inspection of industrial coating work. A punch list for use in the field is included as an appendix. Many of the details covered may be in a specification for a particular project. A specification for coating projects should

include the coatings to be used. The various items are explained in detail in the appendix.

4. Preparation for Inspection

4.1 The guide describes the duties of the inspector and discusses inspection methods, both visual and instrumental, that can be used to determine that the specification requirements have been met by the painting contractor.

4.2 Before painting is started the project engineer must provide the inspector with information from the official plans and specifications as to coating type, thinner to be used, mixing ratios to be used, recommended application thickness, recommended primer, tie coat, topcoat, time between coats, surface preparation, method of application, and any special precautions to be followed such as limits on ambient conditions. These details should be recorded in an inspector's record book to eliminate any misunderstanding between the inspector and the contractor.

5. Field and Shop Inspection

5.1 *Surface Preparation* is one of the most important factors affecting the performance of coatings. The specifier should determine the proper level in accordance with the expected service life and type of coating specified.

5.1.1 *Pictorial Standard D 2200 (SSPC-Vis 1)* should be provided to the inspector on a job involving painting of structural steel. The standard is used by the inspector to determine whether the degree of surface preparation specified in a contract has been attained by the contractor. For large jobs it is recommended that before work starts, an actual steel sample of adequate size be blasted to the satisfaction of the project engineer. This blasted surface should be protected by a clear acrylic coating or encased in plastic and used for reference purposes as the work progresses.

5.2 *Factors Affecting Coating Performance*—There are a number of factors that must be considered to ensure a proper painting job.

5.2.1 *Cleanliness*—Many materials if not removed from the surface will affect the life of the coating. These include oil, grease, soil, weld spatter, and slag that make it impossible to obtain proper adhesion to the metal surface. Deposits of salts (such as chlorides and sulfates) must be removed or long-term coating performance will be seriously affected. The Steel Structures Painting Council (SSPC) issues detailed surface preparation specifications that cover methods for solvent cleaning and hand and power tool cleaning as well as the various methods of blast cleaning.

5.2.2 *Mill Scale*, the bluish-black oxide resulting from the hot-rolling process, is a constant source of trouble leading to coating failure. This scale is very hard and can crack or loosen from temperature changes both in fabricating and weathering in the field.

5.2.3 *Surface Profile*—The texture of the metal surface has a significant effect on the performance of coatings since it increases the surface area to which the coating can develop adhesion. In fact, the term "anchor pattern" is sometimes used to describe the depth of profile required. This varies both with the type and size of the abrasive used. Coarser abrasives generally produce a coarser and deeper profile. Deep profiles are advantageous for adhesion but require

Dry-Film
sters⁵

⁶ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁷ Annual Book of ASTM Standards, Vols 01.06 and 03.03.

⁸ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

more coating to fill in the valleys and cover the peaks of the profile; they cannot be used with low-build coatings that do not cover the peaks even when several coats are applied. A general recommendation is that the surface profile should be one quarter to one third of the dry film thickness of the coating system but does not apply in every case if the resulting profile would be too great.

5.3 Cleaning Procedures—Safety precautions are not addressed separately for each of the following cleaning methods. Each has its own safety-related hazards, and U.S. Occupational Health and Safety Administration regulations should be followed. Material Safety Data sheets (MSDS) for the solvents and cleaning compounds provided by the manufacturer should also be consulted for proper worker protection.

5.3.1 Chemical Cleaning—Solvents are used to remove oil, grease, and related materials. The solvent is applied to the surface by wiping or scrubbing with rags or brushes. The contaminants must be removed (not simply spread out) by a thorough wiping of the affected areas with cloths saturated with clean solvent. Contaminated cloths must not be dipped into clean solvent. The cleaning should be repeated with clean rags and fresh clean solvent. Emulsions, cleaning compounds, steam cleaning, or similar methods and materials may also be used. Where emulsion cleaners, soaps, or detergents are used, they must be removed completely by washing with clean hot water. SSPC SP-1 covers cleaning procedures using these materials.

5.3.1.1 Solvent Vapor Cleaning is a procedure that can be adapted to a production line or a piecework operation. Vapor cleaning removes all soluble contaminants but does not disturb the natural oxide film. If this film must be removed, mechanical cleaning will be necessary as well. The part to be cleaned is placed in the saturated vapor above the heated solvent so that the solvent vapor condenses on the metal surface. Vapor degreasing does not remove particulate matter and parts must be wiped to remove any insoluble soils. Vapor degreasing has the advantage over solvent wiping in that hot solvents are used and the solvent condensation removes soils without recontamination.

5.3.2 Hand Tool Cleaning is the method used for the removal of loose mill scale, loose rust, loose or otherwise defective coating, weld flux, slag and spatter from metal surfaces by hand brushing, hand sanding, hand chipping or scraping using wire, fiber, or bristle brushes, sandpaper, steel wool, hand scrapers or chisels, and chipping hammers. The surface is then cleaned to the condition St 2 given in Method D 2200 (SSPC-Vis 1). SSPC also provides a detailed specification, SSPC-SP 2.

5.3.2.1 Hand tool cleaning requires that all tar, oil and grease, weld flux, and other greasy contaminants be removed

first by solvent cleaning (5.3.1).

5.3.2.2 Wire brushes should be rigid enough to clean the surface thoroughly and shaped to penetrate into all corners and joints. Brushes should be kept free of all materials that may clog the wires of the brush. Hand scrapers should be made of tool steel, tempered and ground to a sharp edge and should be of the proper size and shape to enable cleaning to be done as specified. Scrapers should be kept sharp at all times.

5.3.3 Power Tool Cleaning is a method used for the removal of loose mill scale, loose rust, loose or otherwise defective coating, and weld flux from metal surfaces by power wirebrushes, power impact tools, power grinders, power sanders, or by a combination of these methods. The surface is cleaned to the condition St 3 given in Pictorial Surface Preparation Standards D 2200. SSPC-SP 3 is the detailed specification for power tool cleaning.

5.3.3.1 Power Tool Cleaning requires that all oil, grease, weld flux, and other contaminants be removed first by solvent cleaning (SSPC-SP 1). Hand tool cleaning in accordance with 5.3.2 may be used prior to power tool cleaning.

5.3.3.2 All equipment must be suitable for the configuration of the work to be cleaned and maintained free of material that clogs the wire or disks making them ineffective. All impact tools should be kept sharp.

5.3.4 Blast Cleaning is used to remove foreign materials from a metal surface and to provide a roughened surface by striking the surface with a stream of small, hard abrasive particles such as (dry) sand, grit, or shot.

5.3.4.1 One method utilizes compressed air, special blast nozzles, and abrasive. In another method used in a fabricating shop, wheels propel the abrasive centrifugally against the work. The minimum and maximum particle size of the abrasive may be specified as a means of controlling the surface profile. Water may be injected into the air stream to control dust. Occasionally a high-pressure water blast, either with or without an abrasive injected into the stream, is used as an alternative to open blasting since it reduces the release of dust into the atmosphere.

5.3.4.2 Blast cleaning requires that all oil, grease, and weld flux be removed by solvent cleaning. The compressed air used for blast cleaning should be free of condensed water or oil by making certain that separators and traps are in working order.

5.3.4.3 Blast-cleaning operations should be performed so that no damage is done to the completed portion of the work. Blast cleaning is often performed from the top to bottom of the structure and should only be carried on downwind from any recently painted areas. Dry blast cleaning operations should not be conducted on surfaces that will be wet after blasting and before painting. Dew point must be at least 5°F (3°C) above the steel temperature.

5.3.4.4 The degree of blast cleaning required should at least be equal to the appropriate SSPC surface preparation specification and the applicable pictorial standard shown in Pictorial Surface Preparation Standards D 2200 (SSPC-Vis 1) or National Association of Corrosion Engineers (NACE) as shown in Table 1.

5.3.4.5 Blast cleaned surfaces must be examined for any traces of oil, grease, or smudges; where present, the contaminants must be removed by solvent cleaning (see 5.3.1).

TABLE 1 Comparison of Surface Preparation Specifications

Preparation Grade	SSPC	ASTM D 2200	NACE
Blast cleaning to white metal	SSPC-SP 5	Sa 3	1
Blast cleaning to near-white	SSPC-SP 10	Sa 2 ½	2
Commercial blast cleaning	SSPC-SP 6	Sa 2 ^A	3
Brush-off blast cleaning	SSPC-SP 7	Sa 1	4

^A Pictorial standard B Sa 2-½ shows mill scale and conflicts with the SSPC definition of commercial blast (SP6) which does not allow mill scale.

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Surfaces that have been dry blasted should be brushed with clean brushes, blown with compressed air free of oil and moisture, or vacuum cleaned to eliminate any traces of blast products, dust, or dirt from the surface. This also serves to remove abrasive from pockets and corners.

5.3.4.6 Blast cleaned surfaces should be further treated, primed or painted as specified on the same day they are blasted, preferably within 8 h, or in any event before any visible rerusting occurs. Reblasting will be necessary on any surface if rust bloom forms before the coating can be applied.

5.4 *Cleaning and Preparation of Various Surfaces*—Before application of any coating, all surfaces to be coated must be thoroughly cleaned and properly prepared to the requirements of the specification. All dust, dirt, oil, grease, moisture, soot, tars, or other contaminants should be removed from unpainted surfaces. Previously painted surfaces should be similarly cleaned of all foreign matter; all deteriorated coating must be removed as well. Mortar or cement drippings from earlier repairs must be removed by mechanical or chemical means. Tree limbs or other growth obstructing the structure should be cut away to provide ready access.

5.4.1 *Steel Surfaces*—Removal of rust and scale must be done in the manner and to the degree specified, that is, hand, power tool, or blast cleaned.

5.4.1.1 On bridges, all dirt and debris must be cleaned from around bearing plates, shoes, and other components. The entire surface of the beam or truss seat on each unit of the structure should be cleaned. On truss spans particular attention should be paid to removal of dirt and debris from pockets and crevices. Open steel grid-type decking should be cleaned by use of a detergent steam-water-type jet that will clean painted surfaces without softening or removing tightly adhering coating. The machine manufacturer's recommended solution should be used. Surfaces should then be rinsed with a clear water jet.

5.4.2 *Galvanized Surfaces* that are to be painted should be cleaned and treated in accordance with Method A or D of Practices D 2092. Alternatively, the surface may be allowed to weather a minimum of 6 months before cleaning and painting.

5.4.3 *Aluminum Surfaces*:

5.4.3.1 Complete removal of oil and grease and, for unanodized aluminum, treatment is essential. Vapor degreasing or immersion in an alkaline or acid cleaning solution are commonly used in shop work. In the field a water wash followed by solvent cleaning is a good starting point.

5.4.3.2 Vinyl wash primer is one of the metal pretreatments commonly used on unanodized aluminum. The material is described in Practices D 1730, Type B, Method 8 and is covered by SSPC Paint 27. For exterior work an inhibitive primer is required over this pretreatment. Lead pigmented primers should never be used over aluminum surfaces. The minimum treatment for aluminum is Type B, Method 3 of Practices D 1730, which describes the use of an alcoholic phosphoric acid cleaner.

5.4.4 *Precautions in Preparing Unpainted and Previously Painted Surfaces*—Cleaning should proceed by sections, bays, or other readily identifiable parts of the work. The cleaning of each section, bay, or part of the work must be

entirely completed, inspected, and accepted before any coating is applied. The system of alternately cleaning and painting short sections by one workman is not good practice.

5.4.4.1 If, in the opinion of the project engineer, traffic, or any other source produces an objectionable amount of dust, it is customary for the contractor, at his own expense, to control the dust by using tarpaulins, etc., for a sufficient distance around the structure and take any other precaution necessary to prevent dust and dirt from coming into contact with the cleaned or freshly painted surfaces. It may sometimes be necessary between the various coats to clean newly coated surfaces using some of the specified methods.

5.4.4.2 Some areas to be painted or repainted are exposed to chemical fumes and should be washed with water before painting. Washing may also be necessary between coats of paint. If there is reason to suspect the presence of chemicals, the surfaces should be tested before applying subsequent paints.

5.4.4.3 Residual contaminants present on pitted steel can be a problem. Chloride from deicing salts or a marine environment and sulfate contamination from air pollution have recently been recognized as one of the main factors in premature breakdown of coating systems. High-pressure water blasting is often used to remove these contaminants.

5.5 *Inspection of Surfaces Prior to Field Painting*:

5.5.1 *New Construction*—It must be emphasized that the first coat should be applied to the cleaned surfaces before any soiling or deterioration can occur. If painting is done out of doors, the cleaned areas should receive the first protective coat well before nightfall brings lower temperatures and possible moisture condensation on the surfaces. When surface preparation and painting are carried on indoors, overnight delays between cleaning and painting may be permissible except on blast-cleaned surfaces.

5.5.1.1 Shop-coated steel that has been shipped to the erection site should be stored on blocks to prevent contact with the ground and where it is least likely to be marred, scratched, or subjected to harmful contamination by grease, oil, salts, etc. Insofar as practicable, the steel should be stored to avoid the formation of waterholding pockets. If outdoor storage lasts for several months, the inspector should check the integrity of the coating from time to time, and have any deficiencies corrected. Correction of these deficiencies may require complete blast cleaning and repriming in the field if the shop primer has weathered so long as to make touch up too extensive. The length of time between shop priming and erection and subsequent topcoating should be kept to a minimum to avoid the problem of intercoat adhesion.

5.5.1.2 Immediately before applying the first field coat, the shop-coated surfaces should be cleaned of dust. If necessary to remove grime and oil substances, they are wiped with solvents selected so as not to soften the film appreciably. Miscellaneous scratches and breaks in the shop coat, including those occasioned by field welds, bolts, or rivets, should be cleaned and touched-up as specified before the steel receives the first overall field coat.

5.5.1.3 The inspector must ensure that field rivets have been cleaned of slag and weld spatter. It is important that every coat of the system be applied over dry, soil-free surfaces, and that all previous coats be free of mechanical damage. Great care should be exercised to prevent trapping

corrosive salts under or between coats.

5.5.1.4 The inspector should determine whether the specifications are being followed with reference to the painting or prohibition of painting of contact surfaces in bolted or riveted surfaces of construction. He must ensure that surfaces not in contact but that will be made inaccessible by assembly or erection, have received the full number of specified coats before they become inaccessible.

5.5.2 *Maintenance Repainting*—In most cases, maintenance painting will consist of spot-cleaning and priming of small isolated areas of deterioration followed by application of one overall new finish coat to all surfaces of the structure. The inspector of maintenance painting must be alert for several conditions not encountered in the painting of new work.

5.5.2.1 Sound coating not intended to be removed should not be damaged by cleaning operations on adjacent areas. This is particularly important with spot-blast cleaning.

5.5.2.2 The junctions between sound coating and spot-cleaned areas should present a smooth, feathered appearance. The application of coating to spot-cleaned areas should overlap the old, adjacent coating to a slight extent in order to assure full coverage of the cleaned areas. Before the overall finish coat is applied, the inspector must ensure that oil, grime, dust, and other contaminants are cleaned from the old coating surfaces.

5.5.2.3 Before work has progressed too far, adhesion of the newly applied coat to the old coating should be carefully checked. Under the direction of the engineer the inspector may explore beneath the surface of the existing or new coating film for covered-over rust or loosening of the old film, and where he discovers such conditions, require that the surface be cleaned and repainted.

5.5.2.4 The effect of any newly applied coating on the old underlying coating should be noted. Any coating that shows curling, lifting, or excessive wrinkling should be reported to the engineer immediately since it may have to be removed and the area repainted. If the defects are general, rather than existing in a few isolated areas, use of a different type of coating may be necessary.

6. Inspection Equipment

6.1 *General*—Visual observation is the most important part of inspecting coating application. There are, however, instruments and mechanical aids that are of considerable help to the inspector. They make the painter aware that his work can be checked during progress and even after completion. The different instruments that can be used are described in this section.

6.1.1 *Surface Profile Gages*—The inspector can determine the surface profile of blast-cleaned steel substrates using Test Methods D 4417 to assure an adequate "tooth" is obtained for the specified material. Some of the common instruments are:

6.1.1.1 Surface profile comparator⁹ for visual comparisons of the profile against a reference disk for sand, grit, and shot blast-cleaned steel.

6.1.1.2 Depth micrometers with conical points to project into the valleys to determine profile depth.

6.1.1.3 Tape,¹⁰ to create an exact replica of the profile on a special material. The tape is measured in the field using a spring micrometer to determine average maximum profile height.

6.1.2 *Adhesion of Existing Coating*—The inspector should carry a pocket knife that can be used to determine the soundness of existing paint where there might be blistering or underfilm corrosion. This is a subjective test and its value depends upon the inspector's experience. The cross-cut test, Methods D 3359, is more reproducible.

6.1.3 *Portable Pull-off Adhesion Testers* are available. The apparatus measures the force required to remove a metal stud that has been cemented to the coated surface as described in Method D 4541.

6.2 *Field Inspection Equipment* in good working order should be available to the inspector so that he may perform his function properly.

6.2.1 *Drying and Curing Times*—These are both important considerations since drying time and curing time can both be affected. Minimum temperatures are required for reactive and water-borne coatings while too high a temperature can make application difficult. Inorganic zinc-rich primers and moisture-cure urethanes require certain minimum humidity conditions for proper cure. The manufacturer's recommendations must be followed.

6.2.1.1 *Thermometers*—The paint inspector may need several types of thermometers and should have at least an accurate pocket thermometer with a range from about 0 to 150°F (−18 to 65°C) for measuring the ambient air temperature. The same thermometer or a floating dairy thermometer may be used to determine the temperature of liquid coating, solvent, etc. The pocket thermometer may also be used for determining the temperature of metal surfaces by placing it against the metal while shielding the outer (away from the metal) side of the bulb by means of putty or similar material, so that the reading is not affected by the ambient temperature. Flat surface-temperature thermometers are also available for this purpose.

6.2.1.2 *Dew Point*—A psychrometer containing a wet- and dry-bulb thermometer for determining relative humidity and dew point is a useful inspection tool. Hand-held sling or electrical types are available as well as a direct reading digital type.

6.2.2 *Coating Consistency* is an important characteristic since durability is related to film thickness and whether a film of the proper thickness can be applied is partly controlled by consistency.

6.2.2.1 *Consistency Cups*—There are occasions, such as on-site thinning, when it is necessary to check paint consistency during field application. While giving only partial information about the viscosity of a coating, the Zahn cup is a portable device for checking quickly the approximate consistency of coatings and other liquids. It consists of a bullet-shaped, stainless steel cup with an orifice in the bottom. Attached to the cup is a looped handle with a small

⁹ Comparator[®], manufactured by Keane-Tator, 115 Technology Dr. Pittsburgh, PA 15275 has been found satisfactory for this purpose.

¹⁰ Press-O-Film[®] tape, manufactured by Testex, has been found suitable for this purpose.

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ring at the top to align the cup in a vertical position when withdrawing it from the liquid being tested. To operate, the cup is completely immersed in the liquid to fill it and is then withdrawn rapidly and completely. The time in seconds for the liquid to escape through the orifice is an expression of viscosity, that is, Zahn Cup No. ____ () seconds. It should be noted that Zahn cups are not suitable for all coatings and have poor reproducibility (agreement between different cups is poor—see Test Method D 4212).

6.2.3 Weight-per-Gallon Cup—There are times when the inspector may wish to check the weight-per-gallon of the paint in the field. If the value is low compared to the paint specification, it indicates that unauthorized thinning has been done, while differing values from the same container show that the coating has not been thoroughly mixed for application. The weight-per-gallon cup holds a given quantity of water when filled at 77°F (25°C) or other specified temperature. It is made of corrosion-resistant material and has a closely fitted lid with a small hole in it. In use, the cup is filled with liquid slightly below the specified temperature. As the contents warm up, the excess escapes through the hole and is removed. The filled cup is wiped clean on the outside and weighed. A relatively inexpensive balance having a sensitivity of 0.1 g provides sufficient accuracy. The difference between the full and empty weights divided by 10 is the weight in pounds of 1 gal of the paint. Complete instructions for the procedure are given in Test Method D 1475.

6.2.4 Wet-Film Thickness Gages—This type of instrument is designed to measure the thickness of a wet film of paint *immediately* after it is applied to a surface. Note that erroneous readings may result when using the gage on fast-drying paints such as inorganic zinc. If a wet-film gage is used to determine the thickness of coats subsequent to the first, great care must be taken that partially hardened undercoats are not indented by the gage, thus giving high readings. If the coat being measured has an appreciable softening effect on the previous coat, a wet-film gage cannot be used with accuracy. It is very important to record test results and send a copy to the engineer.

6.2.4.1 Interchemical Gage—This instrument is rolled over the newly applied wet film on a *smooth* flat portion of the surface and the thickness read directly in mils (micrometres). Complete details are given in Method A of Methods D 1212.

6.2.4.2 Notched Gage—This device has a series of calibrated steps for measuring thin to heavy coats. The gage with the proper face is placed squarely on the fresh, wet film. It is then withdrawn perpendicularly *without a sliding movement*. The true wet-film thickness lies between the highest step coated and the next highest which was not coated. The procedure is described in Practice D 4414.

6.2.5 Dry-Film Thickness Gages—Dry-film thickness measurements are of great importance because the protection of the substrate is directly related to the thickness of the coating. There are two ways of making the measurements, nondestructively or destructively. The latter involves penetrating or cutting through the film to the substrate with a needle or blade and measuring by some means the distance between the top and bottom of the film. Because the dimensions are so small, some cuts are made at an angle in an attempt to increase the accuracy of the measurement.

This type of gage destroys the film at the inspection points, necessitating touch up with primer and finish to prevent corrosion at these spots. One kind of cutting gage is described in 6.2.5.1.

6.2.5.1 Tooke Gage—The Tooke inspection gage is designed to measure coating film thickness by microscopic observation of a cut into the film. The tungsten carbide cutting tip is specially shaped to slice a precise narrow groove through a film and into the substrate at an angle to the surface. Thickness of coating on any type of stable substrate may be determined and individual coats may be measured separately providing they are distinguishable, for example, by color. The coating cannot be too brittle or soft, otherwise the cutting tip will tear rather than precisely cut through the coating, making accurate readings impossible.

6.2.6 Nondestructive Film Thickness Gages are widely used for field and shop inspection. For ferrous metals they are based on magnetism, and for nonmagnetic metals on inductance and eddy currents. There are no satisfactory nondestructive methods for wood or other nonmetallic substrates. All of the different types require calibration with standards the thickness of which is known and in the same range as the coating to be measured. The calibration must be made on metal of the same kind, temper, thickness, and contour as that beneath the coating. Test Methods D 1186 and 1400 describe procedures. Measurements on relatively rough surfaces, such as a surface blasted to a high profile, may be misleading unless the instrument is calibrated on an identical surface.

6.2.6.1 Magnetic-Type Gages¹¹—Use either an electromagnet requiring a power supply or a permanent magnet. The principle of the method is that a nonmagnetic coating changes the magnetic force between the magnet and the magnetic base or the magnetic flux between the poles of the magnet. As the change is a function of distance from the metal, the gage can be made to read film thickness directly. Magnetic gages may be affected by the mass of the steel, or if electric welding is being carried out on a structure, for example, a ship's hull, at the time of measurement. Test Methods D 1186 describe the procedure for using magnetic gages. The method is also described in SSPC-PA 2 which includes instructions on the number of measurements for different areas and tolerances on the required film thickness.

6.2.6.2 Nondestructive procedures based on inductance and eddy current techniques are described in Test Method D 1400.

7. Coating Storage and Handling

7.1 Storage of Coating and Thinner—All coatings and thinners should be stored in areas or structures that are well-ventilated and not subject to excessive heat, open flames, electrical discharge, or direct rays of the sun. Storage should be in compliance with applicable regulations. Materials susceptible to damage at low temperatures must be stored in heated areas. If a coating is stocked for a consider-

¹¹ Type 1—Pull-off gages (such as Mikrotest, Inspector Thickness Gage, and the Pencil Pull-Off Gage, or Tinsley type gage); and Type 2—Fixed Probe Gages (such as Elcometer Thickness Gage, Minimeter, General Electric Type B Thickness Gage, Verimeter, Permascope, Dermatron, Positector, Certotest, Accuderm and Minitest) have been found suitable for this purpose.

able length of time (several months), it is desirable to invert the containers at monthly intervals. This will prevent hard settling and thus make mixing quicker and easier when the coating is to be used.

7.1.1 Coating containers should remain unopened until needed and the oldest should be used first. The manufacturer's written instructions should be followed regarding shelf life. Coatings that have livered, gelled, or otherwise deteriorated during storage should not be used. If a particular material is in question, do not use it until it has been tested by the manufacturer or independent laboratory and found to be satisfactory.

7.1.2 Where a skin has formed in the container, the skin should be cut loose from the sides of the container, removed and discarded. If it is felt that the skins are thick enough to have a practical effect on the composition, the remaining paint should not be used until it has been tested and found to be satisfactory.

7.2 *Mixing of Coatings*—All coatings must be thoroughly and completely mixed in clean containers before use. Where there is noticeable settling and mixing is done either by power stirrers or by hand, most of the vehicle should be poured off into a clean container. The pigment is then lifted from the bottom of the container with a clean broad, flat paddle, lumps broken up and the pigment thoroughly mixed with the vehicle present. The poured-off vehicle should be returned slowly to the original container with simultaneous stirring. It is also useful at this point to mix or pour repeatedly from one container to another (boxing) until the composition is uniform. The bottom of the original container should be inspected for unmixed pigment. The coating should not be mixed or kept in suspension by means of an air stream bubbling under the coating surface.

7.2.1 Some coatings may require straining after mixing to ensure homogeneity and to remove skins and foreign matter. The strainers should be of a type to remove only skins, etc., but not to remove pigment. For example, a 150-mesh (105- μ m) strainer is normally satisfactory for most coatings unless some specific size is required in the specification. Coatings should not remain in buckets, spray pots, etc., overnight, but be gathered into a container and remixed before use, provided the manufacturer's stated pot life will not be exceeded. Containers must be covered when not in use, to reduce volatile losses and skinning.

7.2.2 Coatings should be agitated enough during application to ensure homogeneity. Some materials may even require constant agitation during use.

7.3 *Thinning*—Some specifications permit field thinning of laboratory-accepted coatings while others do not. This section describes some commonly accepted procedures when thinning is permitted.

7.3.1 *Initial Samples*—When thinning on the job site is permitted and unless other arrangements have been made, (for example using manufacturer-supplied thinner from unopened containers and complying with manufacturer's written thinning instructions), the painting inspector may need to submit to an agreed-upon testing laboratory a 1-qt (1-L) sample from each batch to be thinned, together with a 1-qt sample of the thinner to be used using clean sample containers in both cases. With these samples a request is submitted for advice on the proper thinning rate for the

conditions prevailing and the consistency limits of the thinned coating.

7.3.2 *Thinning of Coating*—All additions of thinner must be made in the presence of the inspector and no other amounts or types of thinner than those permitted by the specification or manufacturer, or both, may be added. Thinning is carried out by pouring about half of the thoroughly mixed coating into an empty, clean container. The required thinner is then added and the two portions are boxed to obtain a homogeneous mixture.

7.3.3 *Sampling of Thinned Coating*—During the work, additional samples need not be submitted for testing unless a deviation is noted in the coating consistency or if it is suspected that there has been a change in the thinner.

7.3.3.1 When an inspector is qualified and has the necessary equipment available at the field office, arrangements may be made for on-site inspection of thinning and of the thinned coating. This speeds acceptance of a coating and lightens the laboratory workload. The inspector must keep a record of all paint modifications, amount of thinning, weight per gallon, and viscosity. Where dry-film thickness is specified, the inspector must calculate the new wet thickness necessary to obtain the desired dried thickness with the thinned coating. He should make frequent checks of wet-film thickness as work progresses, however compliance with the specification should be based on dry-film thickness where possible.

7.3.3.2 To determine the wet-film thickness of the thinned coating required to obtain the specified dried-film thickness, the percent volume of nonvolatile (solids) in the original coating must be known. This figure is readily obtained from the manufacturer. With this information the calculation may be made as follows:

$$W = D (1.0 \pm T)/S$$

where:

W = wet-film thickness,

D = desired dry-film thickness,

S = percent by volume of coating solids, and

T = percent by volume of thinner added.

7.4 *Heating of Coating*—Coatings as delivered in the manufacturer's containers and mixed thoroughly, are ready for use, unless the specification permits on-site thinning of high-viscosity material. Should the coating temperature be low, that is, 50°F (10°C) its consistency may increase to the point where application is difficult. Where thinning is not permitted, the coating may be heated. Should the contractor wish to reduce the viscosity by heating to make application easier, the containers may be warmed in hot water, on steam radiators, or by other acceptable indirect heating processes. In-line heaters are also available for application equipment. Direct application of flame to the containers is forbidden by fire regulations. It should be noted, however, that heating of the coating alone will not compensate for ambient or surface temperatures, or both, that are below the minimum specified for that material.

8. Weather Considerations

8.1 *Drying*—It is well known that most coatings, particularly those for structures, will not dry properly at low temperatures and high relative humidities, nor will they

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protect if applied over wet surfaces.

8.2 Low Temperature—Many specifications indicate temperature limits between which painting may be undertaken either in the shop or in the field. The minimum ambient temperature is usually 40°C (5°C) or for cold-curing epoxy systems 50°F (10°C) with the temperature of the substrate above freezing. The requirements may state further that painting should not be undertaken when the temperature is dropping. However, some authorities believe that many solvent-borne coatings may be applied at (or below) 32°F (0°C) without adverse effects. Within the limitations of the composition of the coating, this may be satisfactory depending upon the type of coating and providing the surface is dry. Painting over ice or frost will result in early adhesion failure of the coating.

8.3 High Temperature—The maximum reasonable surface temperature for application is 125°F (50°C). A surface that is hot may cause the coating solvents to evaporate so fast that application is difficult, blistering takes place, or a porous film results. To keep the temperature down it is desirable, where practical, to paint under cover at a shop or to protect the surfaces from the sun with tarpaulins.

8.4 Moisture—Painting should not be undertaken in rain, snow, fog, or mist, or when the temperature of the metal surface is below the dewpoint. This is especially true in the spring and fall when days are warm and nights are cool. Wet surfaces should not be painted unless the coatings are specifically designed for that condition. Relative humidity is usually a good index of the acceptability of air conditions and specifications often contain an 85 % upper limit. If it is suspected that the temperature and humidity conditions are such that moisture is condensing upon the surface, the inspector may make the following test: Use a psychrometer to determine the actual relative humidity and dew point; compare the dew point with the metal temperature measured with a surface temperature thermometer. It is generally accepted that the surface temperature should be at least 5°F (3°C) higher than the dew point. A less accurate test is to lightly moisten a small area with a damp cloth and observe evaporation and drying; if the dampness decreases and evaporates within 15 min, the surface is considered satisfactory for the application of coating from the standpoint of dampness and condensation at that particular time.

8.4.1 When coatings must be applied in damp or cold weather, the substrate should be painted under cover, or protected from the surrounding air, and the steel heated to a satisfactory temperature. The steel should remain under cover until dry or until weather conditions permit its exposure in the open.

8.4.2 Applied coatings exposed to freezing, excessive humidity, rain, snow, or condensation should be permitted to dry. Damaged areas of coating should then be removed, the surface again prepared and painted with the same number of coats of the same kind as the undamaged area.

8.5 Wind—The wind direction and velocity must be considered when applying coatings in areas where airborne overspray could damage automobiles, boats, and structures nearby. Heavy winds result in considerable loss of coating and in excessive drying of the droplets reaching the surface. This results in an inability of the film to flow together and create a continuous barrier free of "holidays." Such condi-

tions can also create excessive amounts of dry spray that can interfere with adhesion of the applied or subsequent coats. These problems can be avoided by utilizing brush or roller application methods instead of spray, or scheduling the work at the less windy times of day, changing materials to the dry-fog types that do not adhere or damage adjacent property, scheduling the work so that if dry spray becomes attached to adjacent components it will not create a quality problem because it is attached to the final coat.

9. Coating Application

9.1 Residual Contaminants—Visually inspect the surface immediately prior to painting to ensure that spent abrasive, dust, and debris have been completely removed. Dust removal shall be considered satisfactory when the path left by a gloved hand wiped over the surface is barely discernible when viewed from a distance of 3 ft (1 m). During this inspection, also ensure that any oil or grease contamination that may have become deposited on the surface is completely removed. This is accomplished by solvent cleaning in accordance with SSPC-SP1.

9.2 Quality Assurance—The inspector must ensure that: (1) coatings received at the fabricators or in the field meet the description of the products acceptable under the requirements of the specification; (2) they are properly mixed and thinned (where allowed); (3) colors match a visual standard provided that proper precautions have been taken to prevent damage to adjacent areas from cleaning and painting operations; (4) working practices are so scheduled that damage to newly applied coating is minimized; (5) application equipment (brushes, spray) is acceptable for type, cleanliness, and usability; (6) weather conditions are acceptable under the requirements of the specification; (7) field-testing equipment on hand is in satisfactory working order ready for use; and (8) only the methods of application permitted under the specification are used and that their use is in accordance with 9.2.1 and 9.3 through 9.7. SSPC-PA1 is a specification for application of coating.

9.2.1 Film Defects—All coats should have nearly smooth, streamlined surfaces relatively free of dryspray, overspray, orange peel, fish eyes, pinholes, craters, bubbles, or other significant defects. Show through, insufficient hiding skips, and misses are not acceptable. Runs and sags should be brushed out during application. Abrasive, dirt, or other debris that becomes embedded in the paint film should be removed prior to the application of subsequent coats.

9.3 Brush Application—Painting by brush must be done in a neat, workmanlike manner to produce a smooth coat as uniform in thickness as possible. Coating should be worked into all irregularities in the surface, crevices, and corners. Runs, sags, or curtains should be brushed out. Surfaces that are inaccessible for painting with brushes and on which spraying is not permitted should have coating applied by means of sheepskin daubers. To provide adequate film thickness on places prone to premature breakdown, it is recommended that edges and corners of all metal work, rivet heads, bolts and nuts, and all individual members, bars, shapes, and plates should be striped by brush painting in advance of the application of coating to other parts.

9.3.1 Brushes must be of good quality with pliable bristles. They should not exceed 4 in. (100 mm) in width and the

bristle length should be no less than 3½ in. (90 mm). When not in use, the brushes should be kept in a clean, acceptable condition. The inspector may prohibit the use of any brush not in an acceptable condition.

9.4 Spray Application—Spray application may or may not be allowed. Often it is acceptable in fabricating shops but, because of the possibility of damaging surrounding property or coating films from overspray, it may not be permitted in the field. The inspector should be familiar with the different kinds of spraying, which are compressed air spray, airless spray, hot spray, and hot airless spray.

9.4.1 The equipment should be suitable for the intended purpose, capable of properly atomizing the coating to be applied, and be equipped with suitable pressure regulators and gages. The equipment must be kept in a suitably clean condition to permit proper coating application without depositing dirt, dried coating, and other foreign materials in the film. The air supply for conventional and hot spray application should be free of moisture or oil. Airless spray equipment should be properly grounded. Any solvents left in the equipment should be completely removed before applying coating to the surface being painted.

9.4.2 Coating ingredients must be kept properly mixed in spray pots or containers during coating application, either by continuous mechanical agitation or by intermittent agitation. Coating should be applied in a uniform layer, with overlapping at the edge of the spray pattern. The spray pattern should be adjusted so that the coating is deposited uniformly. During application the gun should be held at right angles (perpendicular) to the surface (not arced or fanned) and at a distance that will ensure that a wet layer of coating is deposited on the surface. The trigger of the gun should be released at the end of each stroke. Poor spray technique resulting in excessive overspray (or sand-like finish) should not be tolerated. All runs, sags, or curtains must be brushed out immediately or the coating will have to be removed and these areas repainted.

9.4.3 Usually brush striping of edges and other vulnerable locations as described in 9.3 is required. Brush or sheepskin daubers are used to coat all areas inaccessible to the spray gun and brushes and are used to work coating into cracks, crevices, and blind spots not adequately spray painted.

9.4.4 Particular care should be observed with respect to type and amount of thinner, coating temperature, and operating techniques in order to avoid depositing coating that is too viscous, too dry, or too thin when it reaches the surface.

9.5 Roller Application—Rollers that are clean and of a material not soluble in the coating to be applied should be used. Roller covers are available in a variety of diameters, lengths, type of fabric, and fiber lengths. The nap (length) used on metal surfaces generally varies from ¼ to ¾ in. (25 to 75 mm). The longer fibers hold more coating but do not provide as smooth a finish. Therefore their use is generally restricted to rougher surfaces and faster drying coatings. There are also specialized rollers available for use on pipes, fences, and even pressure rollers that continually feed the coating to the roller cover.

9.5.1 The roller cover should be dipped into the coating until it is saturated and then rolled along the tray ramp until the coating is completely wetted in. The first load of coating

on the roller should be applied to scrap material to force out air bubbles trapped in the nap. Proper roller technique requires application in the form of a V or W depending on the size of the area involved. The coating should then be cross-rolled to fill in the square created by the boundaries of the initial application. Only moderate pressure should be used as heavy pressure can cause foaming and possible cratering by entrapped air. Application should be finished with light perpendicular strokes in one direction (usually vertical) to provide the smoothest, most uniform finish.

9.6 Miscellaneous Methods—Methods such as pads, mitts, and squeegees or trowels are used for specialized products or in situations where the conventional methods are not suitable due to the location or configuration of the work.

9.6.1 Painter's pads generally consist of a roller-type synthetic fabric attached to a foam pad. The size is generally 4 by 7 in. (100 by 175 mm) and the fiber length is ¾ in. (5 mm). Application technique with a pad on large surface areas is similar to that used with a roller.

9.6.2 Painter's mitts are lamb skin gloves that are dipped into the coating and are rubbed across the surface. They are ideal for application of coatings to odd-shaped surfaces such as pipes and railings.

9.6.3 Squeegee or trowel application is generally used for heavy-bodied thick-film coatings that cannot be applied by spray.

9.7 Rate of Application—Properly written specifications require certain minimum and maximum dry-film thicknesses for each coat. The requirements should be augmented with wet-film thickness figures calculated from the composition of the coating so that the proper dry-film thickness is obtained. (Equation shown in 7.3.3.2.) Wet-film thickness figures are useful to the inspector since he can often check as the work progresses to determine reasonably well that the desired amount of coating is being applied. Later, when the films are dry, the inspector should make spot checks with a dry-film measuring gage to ascertain acceptability of the coatings. Film thickness measurements are more informative than visual inspection, which may show only obvious nonuniform application. Instruments for measuring film thickness and the procedures for their use are described in 6.2.5 and 6.2.6. Nondestructive dry-film thickness gages and measurements are applicable to metal substrates only.

9.7.1 Thickness or coverage requirements apply to the whole structure, not some specific part. It is important that the painting inspector check all areas and determine the film thickness for each coat. For instance, if the "shop coat" requires a minimum of 2 mils (50 µm) dry film, the inspector should ensure that it is obtained within the tolerances permitted in SSPC-PA2. The areas tested should be identified and recorded so that the dry thickness of added coating can be also determined. With nondestructive measuring instruments a 50-ft (15-m) long and 6-ft (1.8-m) high I-beam can be tested in 30 min and low areas if any, properly marked and recorded in the inspector's book. Because the shop prime coat inspector may not be the person doing the field coat application inspection, it is very important to record test results and send a copy to the field inspector in order to better determine thicknesses of subsequently applied coats.

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10. Additional Considerations

10.1 *Ventilation*—It is essential when painting in enclosed spaces that adequate ventilation is provided for removal of solvents.

10.2 *Shopcoat Repair*—Normally after erection work, such as riveting, bolting, welding, straightening, etc., has been completed satisfactorily, areas of the shopcoat will be found to have been damaged. Damaged or bare areas must be thoroughly cleaned, prepared, and covered with one new coat of shop or field primer. In addition, crevices and small cracks should be thoroughly cleaned and spot-primed. After drying, the crevices and cracks should be filled with an acceptable caulking compound as required by the specification. The specified field coats should then be applied over the entire structure.

10.3 *Painting Schedule*—As indicated in 5.4.4, painting should proceed by sections, bays, or parts of the work, and each coat on each section should be applied entirely and accepted by the inspector before a succeeding coat is applied. Except for any shop coat touch ups made necessary by welding, etc., none of the metal that will be exposed to view in the completed structure is to be field-painted until all concrete has been placed. Care must be taken by the contractor during painting operations to protect concrete from being stained by the coating being applied. Any stained concrete surface must be restored to its intended color without damage to the concrete.

10.4 *Film Integrity*—Each coat should be applied as a continuous film of uniform thickness, free of holidays and

pores. Any thin spots or areas missed in the application should be repainted and permitted to dry before the next coat is applied.

10.5 *Recoat Time*—Each coat must be dried throughout the full thickness of the film before application of the succeeding coat. Coating is considered dry for recoating when the next coat can be applied without the development of any detrimental film irregularities such as wrinkling, lifting, or loss of adhesion of the previous coat. For most coatings the time to dry for recoat, even under optimum conditions varies with their composition and that of the subsequent coat. Thus, an oil-based coating may take 2 to 4 days to harden sufficiently to be overcoated with a coating of the same type. However, it may take 3 or 4 months to harden to be satisfactorily overcoated with a vinyl coating or other type of coating containing strong solvents. The coating manufacturer's literature should be consulted for recoat times.

10.6 *Coating System Failure*—Failure of completed coating work may be the result of several factors. Most obvious is noncompliance with the specifications indicating insufficient inspection of surface preparation, coating quality, coating application, or conditions during application and drying/curing. On the other hand, failure may be due to improper specification of coatings for the intended use. Defective coatings should be removed in their entirety, the surface recleaned, and the specified coatings, or their alternatives, applied.

APPENDIX

Nonmandatory Information

X1. INSPECTION CHECKLIST

X1.1 The checklist in Fig. X1.1 lists the key elements to be used for inspection of industrial coating work. Many of the details covered may be in a specification for a particular

project. A job specification for painting should include the coatings to be used. The various items are explained in detail in the text of this guide.

TASK

I Surface Condition Examination 1 Condition of edges, weld splatter 2 Grease or oil, or both 3 Chalk, mildew 4 Protective coverings in place 5 Air temperature 6 Surface temperature 7 Wind direction, velocity 8 Dew Point, RH 9 Visible moisture	I Surface Condition: 1 Visual observation and touch 2 Visual observation and clean white rag 3 Visual observation and magnifying glass 4 Visual observation 5 Air thermometer 6 Surface thermometer 7 Current weather report 8 Sling psychrometer 9 Direct observation	I Cleanliness —Many materials if not removed from the surface will affect the life of the coating. These include oil, grease, soil, weld spatter and slag which make it impossible to obtain proper adhesion to the metal surface (5.2.1, 5.4). Careful observation is the key to making certain that a sound surface is available for painting. Factors are listed which will affect job quality (9.1).
II Safety on the Job: Protective clothing Respirator Safety glasses Ear protection	II Safety on the Job: OSHA Hazard Communication Standard Product MSD sheets Municipal and state regulations	II There are a number of hazards associated with any painting operation; safety regulations must be observed and unsafe conditions or practices should be reported promptly to management (1.2, 5.3).
III Blast Cleaning: 1 Type and size abrasive 2 Clean and dry abrasive 3 Recycled abrasive test 4 Compressed air check 5 Nozzle air pressure	III Blast Cleaning: 1 Sieve analysis and visual observation 2 Visual observation 3 Water test 4 White blotter 5 Pressure gage	III Blast cleaning is used to remove foreign materials from a metal surface and to provide a roughened surface by means of a dry sand, grit, or shot blast impinging the surface with hard abrasive particles impelled by air (5.3.4). The minimum and maximum particle size of the abrasive are specified as a means of controlling the surface profile. Precautions must be taken that blasting equipment is operated properly (5.2.3).
IV Surface Preparation: 1 Dust and abrasive removal 2 Degree of cleanliness	IV Surface Preparation: 1 Visual observation 2 <i>Pictorial Standard D 2200</i> should be used by the inspector on a job involving painting of structural steel to determine whether the degree of surface preparation specified in a contract has been attained by the contractor (5.1.1).	IV Surface preparation is one of the most important factors affecting the performance of paint. The specifier will determine the proper level according to the expected service life of the structure and type of paint specified (5.1). <i>Surface Profile</i> —The texture of the metal surface has a significant effect on the performance of coatings since it increases the surface area to which the coating can develop adhesion.
3 Profile measurement	3 Keane-Tator comparator for visual comparisons of the profile against a reference disk for sand, grit, and shot-blast-cleaned steel (6.1.1). Testex Press-O-Film tape is used to create an exact replica of the profile on a special material. The tape is measured in the field using a spring micrometer to determine average maximum profile height.	
4 Magnetic base reading	4 Magnetic thickness gage (6.2.6)	

FIG. X1.1 Inspection Checklist

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TASK

V Coatings Application:

- 1 Time from surface preparation to application
- 2 Coating viscosity
- 3 Compressed air check
- 4 Protective coverings in place
- 5 Time application began
- 6 Surrounding air cleanliness
- 7 Continue to monitor temperature and humidity conditions
- 8 Correct coating and mixing procedure
- 9 Induction time observed
- 10 Proper percent agitation
- 11 Film thickness
 - Wet

—Dry

- 12 Recoat Times Observed
- 13 Intercoat Cleanliness
- 14 Film Defects, Voids, etc.

VI Recordkeeping:

- 1 Record all details
 - Applicator
 - Manufacturer
 - Batch #'s
 - Container Condition
- 2 Record Observations from Sections I, III, IV, and V of Fig. X1.1

V Coatings Application:

- 1 Record
- 2 Dip-type viscosity cup D 4212
- 3 White blotter
- 4 Visual observation
- 5 Record
- 6 Visual observation
- 7 Visual observation
- 8 Record coatings batch numbers mixing procedure
- 9 Visual observation
- 10 Visual observation
- 11 Film thickness gages

Wet (Notched Gage)—This device has a series of calibrated steps for measuring thin to heavy coats. The true wet-film thickness lies between the highest step coated and the next highest that was not coated. This method is not as accurate as the interchemical gage.

Interchemical Gage—This type of instrument is designed to measure the thickness of a wet film of paint.

Nondestructive Film Thickness Gages are widely used for field and shop inspection. For ferrous metals they are based on magnetism, and for nonmagnetic metals, inductance and eddy currents. See guide for discussion of types.

Dry (Tooke Gage)—The Tooke paint inspection gage is designed to measure paint film thickness by microscopic observation of a cut into the paint film.

- 12 Record time application completed
- 13 Tape test D 3359
- 14 Holiday detector

VI Recordkeeping:

Inspector's log book

V Many factors are important in ensuring proper application of coatings. The details are covered in this guide (Section 9). In particular, note: Dry-film thickness measurements are of great importance because the protection of the substrate is directly related to the thickness of the coating. There are two ways of making the measurements, nondestructively or destructively (6.2.5).

VI Maintenance of a record book containing all transactions between the inspector and the contractor is essential to eliminate contract disputes. Details of the many steps involved in proper coatings application must be recorded (Section 4).

FIG. X1.1 Inspection Checklist (continued)

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Formability of Attached Organic Coatings with Impact-Wedge Bend Apparatus¹

This standard is issued under the fixed designation D 3281; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a method of evaluating the formability of organic coatings intended for application to strip metal substrates by coil-coating or other factory application methods.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 This test method is designed to provide a uniform procedure for determining the degree of flexibility and adhesion of factory-applied organic coatings on strip metal.

2.2 It should be recognized that some metal substrates will crack during forming and that the organic coating may crack accordingly.

3. Apparatus

3.1 *Impact-Type Wedge Bend*, assembly illustrated in Fig. 1.² The instrument contains a 4-lb (1.82-kg) impactor rod with one flat end that slides in a vertical slotted guide and serves as a falling weight to hammer the wedge mandrel with the flat end. A knurled cover inserted in the wedge mandrel impact test hole serves as the impact point. When positioned properly the lifting knob on the impactor coincides with the zero mark on the guide tube. The spacer block is removed from the front edge of the wedge mandrel so that the taper is 0 to $\frac{1}{8}$ in. (0 to 3.2 mm) front to back.

3.2 *Cellulose Adhesive Tape*, $\frac{3}{4}$ in. (20 mm) wide.

4. Test Specimens

4.1 Suitable substrates for the test specimen (including thickness) shall be employed and agreed upon between the purchaser and the seller (Note 1). The dimensions of the base metal panel shall be 4 in. (100 mm) in width, with a maximum length of 12 in. (300 mm). The surface preparation of the base metal shall be agreed upon between the

purchaser and the seller. Prior to application of the coating, edges of the specimens that will be bent in the test shall be rounded slightly to remove burrs in order to eliminate effects associated with cracking of a sharp metallic edge when bent.

NOTE 1—When metal thickness exceeds 0.025 in. (0.64 mm), complete compression of the bend may not occur with a single impact.

4.2 The coating under test shall be applied to the clean base metal after the agreed-to surface preparation of the metal substrate has been completed. The coating shall be applied to one or both sides of the metal (as specified) in a manner that will provide a uniform coating thickness. The dry film thickness shall not vary more than 0.1 mil ($\pm 2.5 \mu\text{m}$) from the specified thickness agreed upon between the purchaser and the seller. The method of application, the cure of the film, the time between the application of the coating and the testing of the specimen, as well as the temperature and humidity environment used for aging the film during this period, shall be agreed upon between the purchaser and the seller.

5. Conditioning and Number of Tests

5.1 Test at least three replicate specimens under the conditions of temperature and humidity mutually agreed upon between the purchaser and the seller. Unless otherwise specified by the purchaser and the seller, make measurements at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity after equilibrating at these conditions for at least 24 h.

6. Procedure

6.1 Place the specimen, coated side down, under the $\frac{1}{8}$ -in. (3.2-mm) cylindrical mandrel. Slide the specimen to the rear of the mandrel platform until the edge is flush with the two studs located at the rear of the platform.

NOTE 2—Since the test is usually more severe if the panel is bent parallel to the original rolling direction of the base metal, the direction of the bend with respect to the "grain" of the base metal should be agreed upon between the purchaser and the seller and reported.

6.2 Raise the remainder of the panel at a uniform velocity to bend the specimen 170 to 180° in a time not to exceed 5 s.

6.3 Slide the spacer located under the impact platform to the extreme front of the tester and tighten it in place with the adjustment screws provided. This allows the impact platform to create a wedge that provides stress angles between 170 and 180° .

NOTE 3—The end of the specimen with the 180° stress angle is defined as having a zero T (OT) bend.

6.4 Center the bent specimen under the impact platform. Place the longest segment of the specimen downward.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.53 on Coil Coated Metal.

Current edition approved April 27, 1984. Published December 1984. Originally published as D 3281 - 73. Last previous edition D 3281 - 73 (1979).

² The Gardner Coverall Impact Tester available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910, has been found satisfactory for this purpose. Use of other equipment should be agreed upon by the purchaser and seller.

6.5 Predetermine the load required to result in a OT bend (180°) and run all tests using the same substrate at this load. The wedge bend required shall be obtained with a single impact representing the minimum force to obtain a OT bend.

6.6 After impact, remove the specimen and visually inspect without magnification the bent portion for cracking and adhesion loss, starting at the most severe bend and progressing outward.

6.7 Measure the distance fractured (cracking) from the edge of the most severe bend outward to the edge with the least severe bend in inches or millimetres.

NOTE 4—Disregard heavy beads of coating on the edges.

6.8 Determine film adhesion loss by firmly applying the adhesive tape to the entire bend area and then removing the tape in a uniform rapid motion. The time between the first bending of the specimen and removal of tape shall not be less than 1 nor more than 5 min. Start removal from the edge of most extreme bending (OT edge) and remove the tape rapidly in a motion parallel to the surface of the longitudinal edge of the bend, such that the tape is pulled back upon itself.

6.9 Examine the taped area and measure the amount of film removed. It should be noted that the primary concern of this rating is the maximum distance of adhesion failure.

7. Precision

7.1 On the basis of an interlaboratory study of this test method in which two operators in each of five laboratories measured two replicates on two days for cracking and adhesion, the standard deviations within and between laboratories were found to be as follows:

	Standard Deviation	
	Cracking, mm	Adhesion, mm
Within laboratory	2.0	3.9
Between laboratory	4.7	10.0

Based on these standard deviations, the following criteria should be used to judge the precision of results at the 95 % confidence level:

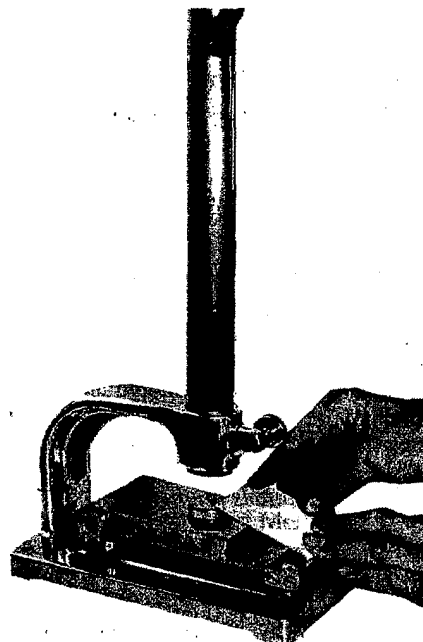


FIG. 1 Coverall Impact Tester

7.1.1 *Repeatability*—Two measurements obtained by the same operator should be considered suspect if they differ by more than

Cracking, mm	5.8
Adhesion, mm	11.4

7.1.2 *Reproducibility*—Two measurements, each the mean of duplicate measurements, obtained in different laboratories should be considered suspect if they differ by more than

Cracking, mm	16.8
Adhesion, mm	35.7

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Standard Practice for Testing Primers and Primer Surfacer Over Preformed Metal¹

This standard is issued under the fixed designation D 3322; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the selection and use of procedures for testing primers and primer surfacers. The test methods included are listed in Table 1.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- B 117 Method of Salt Spray (Fog) Testing²
- C 540 Test Method for Image Gloss of Porcelain Enamel Surfaces³
- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products⁴
- D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings⁵
- D 523 Test Method for Specular Gloss⁵
- D 609 Practice for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁵
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces⁵
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive⁵
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints⁵
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁵
- D 714 Test Method for Evaluating Degree of Blistering of Paints⁵
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁵
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion⁵
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁵
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁵

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved Nov. 26, 1982. Published January 1983. Originally published as D 3322 - 74. Last previous edition D 3322 - 74a.

² Annual Book of ASTM Standards, Vols 03.02 and 06.01.

³ Annual Book of ASTM Standards, Vol 02.05.

⁴ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

⁵ Annual Book of ASTM Standards, Vol 06.01.

- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁵
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁵
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base⁵
- D 1474 Test Methods for Indentation Hardness of Organic Coatings⁵
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁵
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁶
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁷
- D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting⁷
- D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting⁷
- D 1733 Method of Preparation of Aluminum Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products⁸
- D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus⁵
- D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁹
- D 2091 Test Method for Print Resistance of Lacquers⁵
- D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting⁵
- D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion⁵
- D 2201 Test Method for Preparation of Hot-Dipped Nonpassivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁵
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁵
- D 2246 Test Method for Finishes on Primed Metallic Substrates for Humidity-Thermal Cycle Cracking⁵
- D 2248 Practice for Detergent Resistance of Organic Finishes⁵
- D 2454 Practice for Determining the Effect of Overbaking on Organic Coatings⁵
- D 2803 Test Method for Filiform Corrosion Resistance of Organic Coatings on Metal⁵
- D 3170 Test Method for Chipping Resistance of Coatings⁵

⁶ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

⁷ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁸ Discontinued; see 1980 Annual Book of ASTM Standards, Part 27.

⁹ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

TABLE 1 Test Methods

Property	Section	ASTM Method	Fed. Test Method Spec. No. 141B
Abrasion resistance:			
Air blast abrasion tester	5.2	D 658	...
Falling sand method	5.2	D 968	6191
Adhesion:			
Scrape adhesion	5.3.2	D 2197	6303.1
Parallel-groove adhesion	5.3.2	D 2197	6302.1
Tape adhesion	5.3.2	D 3359	...
Chemical resistance:			
Household chemical resistance	5.4.2	D 1308	...
Detergent resistance	5.4.3	D 2248	...
Hydrocarbon resistance	5.4.4	...	6011
Chip resistance	5.5	D 3170	...
Color difference:			
Visual evaluation	5.6	D 1729	4249.1
Instrumental evaluation	5.6	D 2244	6123
Cracking resistance	5.7	D 2246	...
Elongation:			
Conical mandrel	5.8	D 522	...
Cylindrical mandrel	5.8	D 1737	...
Film corrosion	5.9	D 2803	...
Gloss	5.10	D 523	6101
Hardness	5.11	D 1474	...
Holdout	5.12	C 540	...
Mildew resistance	5.13	...	6271.1
Outdoor exposure:			
Blistering	5.14.2	D 714	6461
Cracking	5.14.2	D 661	6471
Rusting	5.14.2	D 610	6451
Checking	5.14.2	D 660	6421
Print resistance	5.15	D 2091	...
Salt spray resistance	5.16	B 117	6061
Sanding properties	5.17	...	6321
Water resistance:			
High humidity	5.18.2	D 1735	...
Water immersion	5.18.3	D 670	...
Weldability	5.19	...	A

^a U.S. Military Specification MIL-P-46105 (MR).

D3359 Test Methods for Measuring Adhesion by Tape Test⁵

D3456 Practice for Determining by Exterior Exposure Tests the Susceptibility of Paint Films to Microbiological Attack⁵

2.2 Federal Test Methods:¹⁰

141B/6011 Immersion Resistance

141B/6271.1 Mildew Resistance

141B/6321 Sanding Characteristics

2.3 U.S. Military Specification:

MIL-P-46105¹⁰

3. Terminology

3.1 Definitions:

3.1.1 *primer*—the first of two or more coats of paint, varnish, or lacquer system (same as in Terminology D 16).

3.1.2 *primer surfacer*—a pigmented coating for filling minor irregularities which is sanded to obtain a smooth uniform surface preparatory to applying finish coats. A primer surfacer is not usually applied over a primer.

4. Significance and Use

4.1 Primers and primer surfacers may be used over many

different surfaces top coated with one or more of a variety of coatings and subjected to many kinds of wear and exposure.

4.2 The selection of the tests to be used for any given product or system must be governed by experience and by the requirement agreed upon between producer and user.

5. Panel Preparation

5.1 *Treatment of Substrate*—Preparation of test panels should include any cleaning treatment agreed upon between purchaser and seller or one of the following ASTM Practices: D 609, D 1730, D 1731, D 1732, D 1733 and D 2092, and Test Method D 2201.

5.2 *Substrate, Film Thickness and Application Means*—Conduct performance tests on the specified substrate on coatings having a film thickness agreed upon between the purchaser and seller. Primers are generally applied to a dry film thickness of 0.3 to 1.5 mils (8 to 38 μ m) and primer surfacers to film thickness of 0.7 to 2.0 mils (17 to 50 μ m). Unless otherwise agreed upon, apply primers and primer surfacers in accordance with Test Method D 823.

5.3 *Measurement of Film Thickness*—Since the properties of the primer or primer surfacer can vary considerably with the thickness of the coating, it is important to know the film thickness. Measure the film thickness in accordance with Test Methods D 1400, D 1005, or D 1186.

5.4 Drying of Primer or Primer Surfacers:

5.4.1 Before tests are run, air dry or bake the primer or primer surfacer according to the schedule and temperature and age as agreed upon between the purchaser and seller.

5.4.2 Overbake the primer or primer surfacer to determine the time/temperature effect on the physical and chemical properties. Do this in accordance with Practice D 2454.

5.4.3 It may be desirable for some reason (handling, stacking, etc) to determine the various stages and rates of film formation in the drying or curing of primers and primer surfacers at room temperatures. Do this as described in Test Method D 1640.

6. Physical Properties of The Dry Film

6.1 Primers and primer surfacers are usually (but not always) topcoated. Therefore, many of the following tests should be run on the complete system (substrate/primer or primer surfacer/topcoat). Some of the tests however are for the untopcoated primer or primer surfacer. The properties required of a primer or primer surfacer depend on the intended end use and the tests to be used should be selected on the basis of experience and agreed upon between the purchaser and seller.

6.2 *Abrasion Resistance*—Determine the abrasion resistance as described in either Test Method D 658 or D 968.

6.3 Adhesion:

6.3.1 The primer or primer surfacer of a specified substrate as agreed upon by the purchaser and seller is subjected to an adhesion test to determine the degree of attachment the coating has to the substrate.

6.3.2 Determine the adhesion of the primer or primer surfacer as described either in Test Methods D 2197 or D 3359.

6.3.3 The above methods, in addition to measuring the adhesion of the coating to the substrate, can also be used to determine the intercoat adhesion between the topcoat and

¹⁰ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

the primer or primer surfacer.

6.4 Chemical Resistance:

6.4.1 Coating systems frequently come into contact with various chemicals that may have an effect on the properties of the system. Failure when it occurs is usually in the form of discoloration, change in gloss, blistering, softening, swelling, dissolving, or loss of adhesion. Unless a primer is to be left untopcoated in actual service, primers and primer surfacers should be topcoated with the appropriate product before undergoing chemical resistance tests.

6.4.2 *Household Chemical Resistance*—Determine the effect of chemicals in accordance with Test Method D 1308.

6.4.3 *Detergent Resistance*—Determine the resistance to failure under conditions of immersion in a detergent solution in accordance with Practice D 2248.

6.4.4 *Hydrocarbon Resistance*—Test hydrocarbon resistance in accordance with Method 6011 of U.S. Federal Test Method Standard No. 141B.

6.5 *Chip Resistance*—The chip resistance of a primer or primer surfacer is the ability of a film to withstand sudden impact from stones, gravel, etc, without being loosened from the substrate. Determine chip resistance by Test Method D 3170.

6.6 *Color Difference-Pigmented Dry Film*—The color differences between two similarly homogeneously colored, opaque film such as those formed by primers or primer surfacers may be determined using visual evaluating techniques or by instrumental means. Determine color differences visually using Practice D 1729. Determine color differences instrumentally using Test Method D 2244.

6.7 *Cracking Resistance*—A test for resistance to temperature and humidity changes, or a cold cracking test as it is sometimes called, is designed to give an indication of the resistance of a coating system to cracking or checking caused by temperature and humidity changes and also by aging. The degree of correlation between accelerated crack results and long term room temperature aging varies with the types of coating. The industry uses the test widely and it is felt that a system showing good cold crack resistance will perform satisfactorily in service. Some factors that can affect results are type of substrate, substrate thickness, primer, primer surfacer, topcoat, and film thickness of the different coatings. Determine cracking resistance in accordance with Test Method D 2246.

6.8 *Elongation*—An elongation test may be used as an indication of the flexibility of an attached primer or primer surfacer. It can also show whether there is any change during aging. Determine elongation by Test Methods D 522 or D 1737.

6.9 *Filiform Corrosion Resistance*—Filiform corrosion is a type of corrosion that occurs under coatings on metal substrates and is characterized by a definite thread-like structure and directional growth. Determine the susceptibility of organic films over metal substrates to this type of corrosion by Test Method D 2803.

6.10 *Gloss*—Determine the gloss of primers and primer surfacers in accordance with Test Method D 523.

6.11 *Hardness*—Determine the film hardness of primers and primer surfacers in accordance with Test Methods D 1474, using either Test Method A (Knoop indentation hardness) or Method B (Pfund indentation hardness) as

agreed upon by the purchaser and seller. Other methods of determining hardness may be used as agreed between purchaser and seller.

6.12 *Holdout*—Holdout is the ability of a primer or primer surfacer to give a smooth (nonporous), uniform appearance when topcoated. This property can be evaluated visually or by instrumental means. One method of measuring for holdout is described in Test Method C 540.

6.13 *Mildew Resistance*—Test mildew resistance in accordance with Method 6271 of U.S. Federal Test Method Standard No. 141B or Practice D 3456.

6.14 Outdoor Exposure:

6.14.1 Primers and primer surfacers can have an important effect on the durability of any paint system destined for exterior use. While the accelerated tests given in other sections of this recommended practice are intended to enable one to predict performance, actual outdoor exposure should be made. Usage of paint systems is so varied that no one set of conditions (length of exposure or place of exposure) can be given in this practice to cover all situations. These conditions as well as the type of substrate, substrate preparation, etc should be agreed upon between the purchaser and the seller. However, it is suggested that, unless otherwise agreed upon, prepare panels for outdoor exposure in accordance with Section 4 of this practice.

6.14.2 Many properties of organic coating systems should be evaluated periodically throughout the outdoor exposure period. Where failures occur on a topcoated system, experience is required to determine whether or not the primer or primer surfacer is involved. Properties most likely to involve the primer or primer surfacer may be evaluated as follows: Blistering, Test Method D 714; Cracking, Test Method D 661; Rusting, Test Method D 610; Checking, Test Method D 660.

6.15 *Print Resistance*—A print test can be used to determine the degree of thermoplasticity or solvent retention of a film and hence whether the product can be safely stacked or packaged and, in the case of a thermoplastic film, at what temperature the film prints or mars. A print test can also be used to determine the degree of marring due to pressure. Determine the imprinting and thermoplasticity of primer or primer surfacer films as described in Test Method D 2091.

6.16 *Salt Spray Resistance*—Salt spray testing of coatings is helpful in determining their resistance to failure in service under conditions of high humidity and salt concentrations. Under accelerated conditions of laboratory testing the temperature, the pH, the concentration of the salt solution and other physical properties can be controlled. The selection of the substrate, the application technique, the choice of the topcoat, the manner in which the coating is scribed, the location or position of the panels within the cabinet, the length of the test, the inspection of panels and the method of reporting results must be agreed upon between the purchaser and seller. Test for salt spray resistance in accordance with Method B 117.

6.17 Sanding Properties:

6.17.1 Sanding properties are normally expected of primer surfacers only. Method 6321 of U.S. Federal Test Method Standard No. 141B covers this property.

6.17.2 Prepare and dry a film of the material to be tested as specified in the product specification. Scuff the surface of

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the dried film manually with 400 softback sandpaper. Examine the film for gouging and deep scratches and determine whether there has been any clogging of the sandpaper. Depending on the end use of the primer surfacer involved, other methods of sanding may be used as agreed upon by the purchaser and seller.

6.18 Water Resistance:

6.18.1 Testing of coating systems with water is helpful in determining their resistance to failure under conditions of high humidity or water immersion. Failure in water tests is usually evidenced by blistering, dulling, softening or loss of

adhesion which does not disappear or recover upon evaporation of the absorbed water.

6.18.2 Determine the resistance to failure under conditions of high humidity in accordance with Practice D 1735.

6.18.3 Determine the resistance to failure under conditions of water immersion in accordance with Practice D 870. This test is best suited for coating systems that will actually be soaked in water during service.

6.19 Weldability—In some instances it is necessary to weld metal that has been primed. The welding characteristics of a primer or primer surfacer may be tested in accordance with U.S. Military Specification MIL-P-46105 (MR), Section 4.4.11.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Guide for Testing Interior Solvent-Reducible Flat Wall Paints¹

This standard is issued under the fixed designation D 3323; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{ε1} NOTE—Editorial changes were made throughout, including the title, in October 1988.

1. Scope

1.1 This guide covers the selection and use of procedures for testing interior solvent-reducible flat wall paints, such as alkyd or other solvent-thinned finishes. The test methods included are listed in Tables 1 and 2. All of these test methods may not be required for each paint. Selection of the test methods must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and seller.

1.2 This guide covers the testing of ready-mixed finish coats for application by brushing, roller coating, spraying, or other methods, on wood, plaster, wallboard, masonry, previously painted surfaces, and other interior architectural surfaces.

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester³
- D 154 Guide for Testing Varnishes⁴
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints⁵
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁴
- D 522 Test Methods for Mandrel Bend Test of Attached Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁴
- D 1208 Test Methods for Common Properties of Certain Pigments⁶

- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁴
- D 1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus⁷
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁴
- D 1554 Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials⁸
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁴
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁹
- D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus¹⁰
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁴
- D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion⁴
- D 2244 Test Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates⁴
- D 2369 Test Method for Volatile Content of Coatings⁴
- D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁴
- D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints⁴
- D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints⁴
- D 2621 Test Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints⁴
- D 2801 Test Method for Leveling Characteristics of Paints by Draw-Down Method¹¹
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁴
- D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus⁷
- E 105 Recommended Practice for Probability Sampling of Materials⁹
- E 313 Test Method for Indexes of Whiteness and Yellowness of Near-White Opaque Materials⁹

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 31, 1980. Published December 1980. Originally published as D 3323 - 74. Last previous edition D 3323 - 79.

² Annual Book of ASTM Standards, Vols 06.01, 06.02 and 06.03.

³ Annual Book of ASTM Standards, Vols 05.01, 06.01, and 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁶ Annual Book of ASTM Standards, Vol 06.02.

⁷ Annual Book of ASTM Standards, Vol 06.03.

⁸ Annual Book of ASTM Standards, Vol 04.09.

⁹ Annual Book of ASTM Standards, Vol 14.02.

¹⁰ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

¹¹ Discontinued; see 1989 Annual Book of ASTM Standards, Vol 06.01.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Method	Federal Test Method Std. No. 141
Sampling	5	E 105	1022
Liquid paint properties:			
Absorption	6.10	...	4421
Coarse particles and foreign matter	6.3	D 185	4091†
Color acceptance	6.8	...	...
Condition in container	6.2	...	3011
Consistency	6.6	D 562	4281†
Fineness of dispersion	6.4	D 1210	4411†
Flash point	6.7	D 1310, D 93, D 3278	...
Rheological properties of non-Newtonian materials	6.9	D 2196	...
Skinning	6.1	D 154	3021
Weight per gallon	6.5	D 1475	4184†
Paint application and film formation:			
Application of brushed films	7.1	...	2141, 4321
Application of sprayed films	7.2	...	2131, 4331
Drying time	7.3	D 1640	4061
Leveling properties	7.4	D 2801	...
Producing films of uniform thickness	7.5	D 823	2162
Roller coating properties	7.6	...	4335
Working properties	7.7	...	4541
Appearance of dry paint film:			
Color difference by visual comparison	8.1	D 1729	4249†
Color difference by instrument evaluation	8.2	D 2244	6123†
Specular gloss (sheen)	8.3	D 523	6103†
Properties of dry paint film:			
Adhesion	9.1	D 2197	6303†
Cleanability	9.2	D 2486	...
Scrub resistance	9.2.1	D 2486	...
Washability	9.2.2	...	6141
Elongation	9.3	D 1737, D 522	6221
Fume resistance	9.4	...	...
Yellowness index	9.5	E 313	6131
Hiding power (dry opacity)	8.4	D 2805, D 344	...
Analysis of paint:	10.1	...	...
Identification of vehicle solids	10.6	D 2621	...
Pigment content	10.4	D 2371	4021, 4022†
Separation of vehicle	10.5	D 2372	4032†
Volatile content	10.2	D 2369	4041†, 4042
Water content	10.3	D 1208	4081

† Cancelled U.S. Federal Methods.

2.2 U.S. Federal Test Method Standard 141.¹²

- 1022 Sampling (General)
- 2131 Application of Sprayed Films
- 2141 Application of Brushed Films
- 3011 Condition in Container
- 3021 Skinning (Partially Filled Container)
- 4042 Volatile and Nonvolatile Content (Vacuum Oven)
- 4321 Brushing Properties
- 4331 Spraying Properties
- 4335 Roller Coating Properties
- 4421 Absorption Test
- 4541 Working Properties and Appearance of Dried Film

3. Definitions

- 3.1 For definitions of terms in these practices, refer to Definitions D 16 and D 1554.

4. Conditions Affecting Solvent-Thinned Flat Wall Paint Topcoats

- 4.1 Practical requirements and performance of solvent-thinned flat wall paints may vary with:
 - 4.1.1 *Substrate Type*, such as type and quality of wood or

hardboard, wallboard and joint cement systems, alkalinity, type and quality of plaster, and type and quality of old paint.

4.1.2 *Substrate Conditions*—Smoothness of substrate and quality of topcoat will affect spreading rate, final appearance, and texture.

4.1.3 Type and quality of primer and the time before topcoating.

4.1.4 Environmental conditions, such as temperature and humidity, at the time of paint application and during drying.

5. Sampling

5.1 Prior to sampling, the condition of the container should be established since damage to it may cause evaporation, skinning, or other undesirable effects in the coating. Determine the condition of the paint in accordance with 6.1 and 6.2.

5.2 Sample in accordance with Section 4.2.1 of Method 1021 of Federal Test Method No. 141. Determine the weight per gallon in accordance with Test Method D 1475. Repeat this procedure until successive values agree within 0.1 lb (45 g) or as agreed upon between the purchaser and the seller. Then take samples for testing.

5.3 Specify the amount of sample, the package sizes, and identification codes to assure a representative sample. A

¹² Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

TABLE 2 Alphabetical List of Test Methods

Test Method	Section	ASTM Test Method	Federal Test Method Std. No. 141
Absorption test	6.10	...	4421
Adhesion	9.1	D 2197	6303
Application of brushed films	7.1	...	2141, 4321
Application of sprayed films	7.2	...	2131, 4331
Coarse particles and foreign matter	6.3	D 185	4091
Color acceptance	6.8	...	...
Color difference by visual comparison	8.1	D 1729	4249
Color difference by instrument evaluation	8.2	D 2244	6123
Condition in container	6.2	...	3011
Consistency	6.6	D 562	4281
Drying time	7.3	D 1640	4061
Elongation	9.3	D 1737, D 522	6221
Fineness of dispersion	6.4	D 1210	4411
Flash point	6.7	D 1310, D 93, D 3278	...
Fume resistance	9.4	...	...
Hiding power (dry opacity)	8.4	D 2805, D 344	...
Identification of vehicle solids	10.6	D 2621	...
Leveling properties	7.4	D 2801	...
Pigment content	10.4	D 2371	4021, 4022
Producing films of uniform thickness	7.5	D 823	2162
Rheological properties of non-Newtonian materials	6.9	D 2196	...
Roller coating properties	7.6	...	4335
Sampling	5	E 105	1022
Scrub resistance	9.2.1	D 2486	...
Separation of vehicle	10.5	D 2372	4032
Skinning	6.1	D 154	3021
Specular (gloss sheen)	8.3	D 523	6103
Volatile content	10.2	D 2369	4041, 4042
Washability	9.2.2	...	6141
Water content	10.3	D 1208	4081
Weight per gallon	6.5	D 1475	4184
Working properties	7.7	...	4541
Yellowness index	9.5	E 313	6131

1-gal (4-L) sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling plan, consult Recommended Practice E 105.

6. Liquid Paint Properties

6.1 Skinning—Paints containing a binder which dries by oxidation are subject to skin formation in a partially filled can. Since skins are insoluble in the paint, they must be removed before use. This test in a partially filled container indicates the tendency of a paint to skin. Examine the original sample for skins, both on the surface and in its mass. On a well-mixed, skin-free portion of the sample, perform a skinning test in accordance with Guide D 154.

6.2 Condition in Container—Thickening, settling, and separation are undesirable and objectionable if the paint cannot be reconditioned with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties of paints after storage. Determine the condition in the container in accordance with Method 3011 of U.S. Federal Test Method Standard No. 141.

6.3 Coarse Particles and Foreign Matter—Paints must be free of coarse particles and foreign matter to form a uniform film of good appearance, a typical maximum being 1 weight % of the total paint. Determine the percent of coarse particles and foreign matter in accordance with Test Method D 185.

6.4 Fineness of Dispersion—The more finely a pigment is dispersed the more efficiently it is being utilized. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the product

down a tapered groove varying in depth from 4 to 0 mils (100 to 0 μ m). The point at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the wet film is taken as the dispersion reading. Higher readings in Hegman units or lower readings in mils or micrometres indicate better dispersion. Determine the fineness of dispersion in accordance with Test Method D 1210.

6.5 Weight per Gallon—The density of a paint as measured by weight per gallon or kilograms per litre is used to assure product uniformity, batch to batch, and provides a check against the theoretical weight calculated from the formula. The density is expressed as the weight in pounds avoirdupois of 1 U.S. gal of the paint at a specified temperature. Determine weight per gallon or kilograms per litre in accordance with Test Method D 1475, using a calibrated weight per gallon cup.

6.6 Consistency—Paints of a given type should fall within a stated consistency range for satisfactory reproduction of a specific formula. In the referenced test method, consistency is defined as the load in grams required to produce the specified rate of shear. Determine consistency in accordance with Test Method D 562.

6.7 Flash Point—When the flash point of a material is required for shipping information, use Test Methods D 1310, D 3278, or D 93.

6.8 Color Acceptance—This method gives an indication of the ability of the paint to be tinted with tinting systems in common use. Determine color compatibility as agreed upon between the purchaser and seller.

6.9 Rheological Properties of Non-Newtonian Materials—Rheological properties are related to application and flow

properties of the liquid paint. The referenced test method covers the determination of the rheological properties and is particularly suited for use with paints that display thixotropic characteristics. Determine the rheological properties in accordance with Test Method D 2196.

6.10 *Absorption*—This method provides a rapid determination of the relative penetration of the binder into the surface. It provides a rough measure of the wetting and penetrating properties of liquid materials. Loss of binder and the resultant change in pigment volume, as the film dries, are indicated by this test. Determine the absorption in accordance with Method 4421 of U.S. Federal Test Method Standard No. 141.

7. Paint Application and Film Formation

7.1 *Application of Brushed Films*—Refer to Method 2131 or 4321 of U.S. Federal Test Method Standard No. 141 to determine the brushing properties of a coating. Method 4321.1 is quite subjective although someone experienced in the art can obtain consistent results, particularly in the determination of the "drag" properties.

7.2 *Applications of Sprayed Films*—Refer to Method 2131 or 4331 of U.S. Federal Test Method Standard No. 141 to determine the spraying properties of a coating. Method 4331 is very subjective and should be performed by an individual skilled in the art of using spray equipment.

7.3 *Drying Time*—The drying time of an interior finish is important in determining when a freshly painted room can be put back to use. The test can also be used to determine whether or not the drying properties of a paint have changed during storage. Any one or several of the test methods that cover the determination of the various stages and rates of film formation in the drying or curing of organic coatings may be used. For example, if two coats are specified, the determination of "dry-to-recoat" times will be important. Determine the appropriate drying time in accordance with Test Method D 1640.

7.4 *Leveling Properties*—Leveling is an important factor when smooth surfaces are to be produced. The referenced test method covers the laboratory determination of the relative leveling characteristics of liquid coatings. Determine leveling characteristics in accordance with Test Method D 2801.

7.5 *Producing Films of Uniform Thickness*—Method D 823 covers the preparation of coating films of uniform thickness essential in conducting tests.

7.6 *Roller Coating Properties*—The referenced method outlines a procedure for making an evaluation of a material's characteristics when applied by a roller. Determine roller coating properties in accordance with Method 4335 of U.S. Federal Test Method Standard No. 141.

7.7 *Working Properties*—Working properties of a paint are generally compared to a standard or described by requirements in the product specification. Determine working properties in accordance with Method 4541 of U.S. Federal Test Method Standard No. 141.

8. Appearance of Dry Paint Film

8.1 *Color Difference of Opaque Materials by Visual Comparison*—Visual comparison of color is fast and often acceptable. The referenced practice covers the spectral,

photometric, and geometric characteristics of light source, illuminating and viewing conditions, sizes of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color difference in accordance with Practice D 1729.

8.2 *Color Differences of Opaque Materials by Instrument Evaluation*—Color difference between a product and the standard can be measured by instrument. The referenced method covers the instrumental determination of small color differences, observable in daylight illumination, between nonfluorescent, nonmetameric, opaque surfaces such as paint specimens. If metamerism is suspected, visual evaluation (8.1) should be used to verify instrumental results. Determine the color difference in accordance with Method D 2244.

8.3 *Specular Gloss (Sheen)*—The test method given, using the 85° geometry, is useful in characterizing the low angle appearance of flat paints. Determine 85° gloss in accordance with Test Method D 523.

8.4 *Hiding Power*—Hiding power is a measure of the ability of a coating to hide the substrate. It is, however, dependent on uniform film thickness which is influenced by the flow, leveling, and application properties of the enamel. Test Method D 344 uses brush application and compares the enamel to a standard. Test Method D 2805 uses an applicator bar and is more reproducible and measures smaller differences than D 344. Determine hiding power in accordance with Test Methods D 344 or D 2805.

9. Properties of Dry Paint Film

9.1 *Adhesion*—Measure the adhesion of the paint by testing in accordance with Test Method D 2197.

9.2 *Cleanability*—The ability to remove marks satisfactorily without damaging the film is important with interior finishes.

9.2.1 *Scrub Resistance*—The test for scrub resistance of interior latex flat wall paint can be applied to the solvent-thinned type. Use Test Method D 2486.

9.2.2 *Washability*—The referenced method determines the washability of a paint by subjecting a soiled film of the dried paint to the cleaning action of a wet sponge and cake grit soap. Determine the washability in accordance with Method 6141 of U.S. Federal Test Method Standard No. 141.

9.3 *Elongation*—Elongation is a measure of flexibility of a paint film. Determine elongation in accordance with Test Methods D 1737 or D 522.

9.4 *Fume Resistance*—Some paints exhibit a change in appearance (usually color) when subjected to foul atmospheres, for example, air containing certain sulfur compounds. There is no standard test method for determining the ability of a paint to resist this change.

9.5 *Yellowness Index*—The referenced test method is used for white or near white specimens to determine color departure from white toward yellow or color change towards yellow on exposure or treatment. Determine the yellowness index in accordance with Test Method E 313.

10. Analysis of Paint

10.1 If a specification requires certain raw materials or certain components in a given amount, then chemical

analysis is required. Chemical analysis determines whether the specified components are present and if they are, in what amounts. It does not necessarily establish paint quality which can be greatly affected by manufacturing techniques. No single schematic analysis is comprehensive enough to cover the wide variety of materials used in paint compositions.

10.2 Volatile Content—The percent of volatile matter indicates the thinner loss from the film as it dries. The referenced test method covers the determination of the volatile content of solvent-type paints. The quantity determined subtracted from 100 % gives the nonvolatile content of the coating. Determine the volatile content in accordance with Test Method D 2369.

10.3 Water Content—The referenced test method covers the determination of water in paint and related materials by distilling the sample with a volatile solvent. Determine water content in accordance with Test Method D 1208.

10.4 Pigment Content—Pigment provides the hiding and color and influences many other properties of a coating. The referenced test method describes the procedure for the quantitative separation of the vehicle from the pigment in solvent-type coatings. It is used to determine the weight percent pigment in the paint. Determine the pigment content in accordance with Test Method D 2371.

10.5 Separation of Vehicle—The recommended procedure describes the separation of vehicle from certain solvent-type paints. The separated vehicle is subjected to further analysis. Separate the vehicle in accordance with Method D 2372.

10.6 Identification of Vehicle Solids—The suggested method covers the qualitative characterization or identification of separated paint vehicle solids by infrared spectroscopy. It is useful in detecting uniformity, batch to batch, and the presence of adulterants. Characterize vehicle solids in accordance with Method D 2621.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

1. Scope

1.1 This standard covers the determination of volatile matter, water, pigment, and vehicle content in liquid coatings by gravimetric methods. It is not applicable to solid coatings. It is not applicable to coatings that require the use of reagents.

1.2 This standard covers the determination of volatile matter, water, and lacquer content in liquid coatings that are applied to a surface.

1.3 This standard covers the determination of volatile matter, water, and lacquer content in liquid coatings that are applied to a surface.

1.4 This standard covers the determination of volatile matter, water, and lacquer content in liquid coatings that are applied to a surface.

1.5 This standard covers the determination of volatile matter, water, and lacquer content in liquid coatings that are applied to a surface.

2. Referenced Documents

2.1 D 1208

2.2 D 2369

2.3 D 2371

2.4 D 2372

2.5 D 2621

2.6 D 2621

2.7 D 2621

2.8 D 2621

2.9 D 2621

2.10 D 2621

2.11 D 2621

2.12 D 2621

2.13 D 2621

2.14 D 2621

2.15 D 2621

2.16 D 2621

2.17 D 2621

2.18 D 2621

2.19 D 2621

2.20 D 2621



Designation: D 3335 - 85a (Reapproved 1991)^{e1}

Standard Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 3335; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

^{e1} Note—Keywords were added editorially in June 1991.

1. Scope

1.1 This test method covers the determination of lead² contents between 0.01 and 5 %, cadmium contents between 50 and 150 ppm (mg/kg), and cobalt contents between 50 and 2000 ppm (mg/kg) present in the nonvolatile portion of liquid coatings or contained in dried films. There is no reason to believe that higher levels of all three elements could not be determined by this test method, provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

1.2 Only pigmented coatings were used for evaluating this test method, but there is no reason to believe that varnishes and lacquers could not be analyzed successfully, provided that appropriate precautions are taken.

1.3 This test method is not applicable to the determination of lead in samples containing antimony pigments (low recoveries are obtained).

1.4 If lead is present in the sample to be analyzed in the form of an *organic* lead compound at a concentration greater than 0.1 %, small losses of lead may occur, resulting in slightly poorer precision than shown in Section 12.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water³

D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings⁴

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved July 16 and Nov. 29, 1985. Published January 1986. Originally published as D 3335 - 74. Last previous edition D 3335 - 84.

² Vandenberg, J. T., Swafford, H. D., and Scott, R. W., "Determination of Low Concentrations of Lead in Paint," *Journal of Paint Technology*, Vol 47, No. 604, May 1975.

³ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

3. Summary of Test Method

3.1 The specimen of liquid coating or dried film is prepared for analysis by dry ashing. The content of lead, cadmium, or cobalt of an acid extract of the ash is determined by atomic absorption spectroscopy.

4. Significance and Use

4.1 The permissible level of heavy metals in certain coatings is specified by governmental regulatory agencies. This test method provides a fully documented procedure for determining low concentrations of lead, cadmium, and cobalt present in both water and solvent-reducible coatings to determine compliance.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of an atomizer and either a single- or three-slot burner; gas pressure regulating and metering devices for air and acetylene; lead, cadmium, and cobalt source lamps⁵ with a regulated constant-current supply; a monochromator and associated optics; a photosensitive detector connected to an electronic amplifier; and a readout device.

5.2 *Muffle Furnace*, capable of maintaining $500 \pm 10^\circ\text{C}$.

5.3 *Crucibles*, wide-form, porcelain, glazed inside and outside except for the outside bottom surface, approximately 30-mL capacity, 50-mm rim diameter and 31-mm height.⁶

5.4 *Hot Plate*, with variable surface temperature control over the range from 70 to 200°C .

5.5 *High-Silica Glass Beakers*,⁷ 100 and 250-mL.

5.6 *Volumetric Flasks*, 50, 100, and 1000-mL.

5.7 *Dropping Bottles*, $\frac{1}{4}$ or $\frac{1}{2}$ -oz (7 or 15-mL) capacity.

5.8 *Glass or Disposable Syringes*, 5 or 10-mL capacity.

5.9 *Pipets*, 1, 2, 5, and 10-mL capacity.

5.10 *Paint Shaker*.

5.11 *Paint Draw-Down Bar*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be

⁵ Both hollow cathode lamps and electrodeless discharge lamps have been found satisfactory for this purpose.

⁶ Coors No. 25007 crucibles, or equivalent, have been found satisfactory for this purpose.

⁷ Vycor beakers manufactured by the Corning Glass Co. have been found satisfactory for this purpose.

used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁸ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water conforming to Type II of Specification D 1193.

6.3 *Ammonium Acetate Solution* (50 % weight/volume)—Dissolve 500 g of ammonium acetate ($\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$) in water and dilute to 1 L.

6.4 *Ammonium Acetate Diluting Solution*—Add 50 mL of HNO_3 (sp gr 1.42) to 150 mL of 50 % weight/volume ammonium acetate solution and dilute to 1 L.

6.5 *Cadmium Standard Stock Solution* (1 mg/mL)—Dissolve 2.1032 g of cadmium nitrate ($\text{Cd}(\text{NO}_3)_2$) in 10 mL of water, add 10 mL of HNO_3 (sp gr 1.42), and dilute to 1 L.

6.6 *Cobalt Standard Stock Solution* (1 mg/mL)—Dissolve 4.9387 g of cobalt nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) in 10 mL of water, add 10 mL of HNO_3 (sp gr 1.42), and dilute to 1 L.

6.7 *Lead Standard Stock Solution* (1 mg/mL)—Dissolve 1.5980 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 10 mL of water, add 10 mL of HNO_3 (sp gr 1.42), and dilute to 1 L.

NOTE 1—10.00 mg/mL concentrations of cadmium, lead, silver and zinc are available as SRM 2121; and the same concentration of cobalt, copper, iron and nickel as SRM 2124 from: Office of Standard Reference Materials, Room B-311, Chemistry Building, NIST, Washington, DC 20234.

6.8 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

6.9 *Nitric Acid* (1+1)—Add 1 volume of HNO_3 (sp gr 1.42) to 1 volume of water.

7. Hazards

7.1 Concentrated nitric acid is corrosive and may cause severe burns of the skin or eyes; the vapor is irritating to mucous membranes. Use care in handling this acidic substance. Refer to suppliers' Material Safety Data Sheet.

7.2 Use only a rubber bulb aspirator for pipetting liquids.

8. Calibration and Standardization

8.1 Prepare 100-mL quantities of at least four standard solutions bracketing the expected lead, cadmium, or cobalt concentration in the sample to be tested. To suitable aliquots add 5 mL of HNO_3 (sp gr 1.42) and 15 mL of 50 % ammonium acetate solution. Dilute to 100 mL with water.

8.2 Operational instructions for atomic absorption spectrophotometers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

8.3 Turn the instrument on and set the wavelength to the

⁸ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopoeia."

NOTE 4—Ashing at temperatures in excess of 500°C may result in the loss of some lead by volatilization.

9.6 When the ashing appears to be complete (do not exceed 1 to 2 h), remove the crucible or beaker from the

preheated muffle furnace and ash at 475 to 500°C.

9.5 After charring is complete, place the container in a hot plate until the material chars.

9.4 When the specimen appears to be dry or when starting with a dried film, gradually increase the temperature of the

9.3 Place the crucible (or beaker) containing the liquid coating on a hot plate and slowly increase the temperature until the material is dried. With some types of coatings, an initial oven-drying at 105°C may be necessary to remove solvents without incurring losses due to spattering.

NOTE 3—Recover dried paint films from previously coated substrates (being careful not to remove any underlying material from the substrate) or prepare in the laboratory from liquid samples. For the laboratory preparation, flow some of the well-mixed sample onto a clean glass plate. The use of a paint draw-down bar is recommended to obtain uniform wet film thickness not exceeding 2 mils (50 µm). Allow the film to dry in an oven at 105°C for a minimum of 1 h. Scrape the dried film off the glass plate, preferably with a single-edge razor blade.

NOTE 2—The specimen size called for will give a concentration of approximately 10 to 20 µg/mL lead in the final diluted solution for paints containing approximately 500 ppm (mg/kg) lead, based on the solids. If the material is estimated to contain more or less than 500 ppm lead, the quantity taken should be adjusted accordingly. In the event that significantly less than 500 ppm lead is expected, a 250-mL high-silica beaker may be used in place of the crucible to accommodate specimen weights up to 10 g. The size specified will give a concentration of approximately 2 to 4 µg/mL cadmium in the final diluted solution for paints containing approximately 100 ppm cadmium, based on the solids. The size specified will give a concentration of approximately 10 to 20 µg/mL cobalt in the final diluted solution for paints containing approximately 500 ppm cobalt, based on the solids.

NOTE 2—The specimen size called for will give a concentration of approximately 10 to 20 µg/mL lead in the final diluted solution for paints containing approximately 500 ppm (mg/kg) lead, based on the solids. If the material is estimated to contain more or less than 500 ppm lead, the quantity taken should be adjusted accordingly. In the event that significantly less than 500 ppm lead is expected, a 250-mL high-silica beaker may be used in place of the crucible to accommodate specimen weights up to 10 g. The size specified will give a concentration of approximately 2 to 4 µg/mL cadmium in the final diluted solution for paints containing approximately 100 ppm cadmium, based on the solids. The size specified will give a concentration of approximately 10 to 20 µg/mL cobalt in the final diluted solution for paints containing approximately 500 ppm cobalt, based on the solids.

9.2 Prepare at least two replicate specimens by weighing by difference from a dropping bottle or syringe approximately 2 to 3 g of the mixed liquid coating, or by directly weighing approximately 1 to 2 g of dried paint film, into a 30-mL porcelain crucible. Weigh to 0.1 mg.

NOTE 2—The specimen size called for will give a concentration of approximately 10 to 20 µg/mL lead in the final diluted solution for paints containing approximately 500 ppm (mg/kg) lead, based on the solids. If the material is estimated to contain more or less than 500 ppm lead, the quantity taken should be adjusted accordingly. In the event that significantly less than 500 ppm lead is expected, a 250-mL high-silica beaker may be used in place of the crucible to accommodate specimen weights up to 10 g. The size specified will give a concentration of approximately 2 to 4 µg/mL cadmium in the final diluted solution for paints containing approximately 100 ppm cadmium, based on the solids. The size specified will give a concentration of approximately 10 to 20 µg/mL cobalt in the final diluted solution for paints containing approximately 500 ppm cobalt, based on the solids.

9. Procedure

9.1 If the sample is a liquid coating, mix it until homogeneous, preferably on a mechanical shaker. Determine the nonvolatile content in accordance with Guide D 2832.

9.2 Prepare at least two replicate specimens by weighing by difference from a dropping bottle or syringe approximately 2 to 3 g of the mixed liquid coating, or by directly weighing approximately 1 to 2 g of dried paint film, into a 30-mL porcelain crucible. Weigh to 0.1 mg.

NOTE 2—The specimen size called for will give a concentration of approximately 10 to 20 µg/mL lead in the final diluted solution for paints containing approximately 500 ppm (mg/kg) lead, based on the solids. If the material is estimated to contain more or less than 500 ppm lead, the quantity taken should be adjusted accordingly. In the event that significantly less than 500 ppm lead is expected, a 250-mL high-silica beaker may be used in place of the crucible to accommodate specimen weights up to 10 g. The size specified will give a concentration of approximately 2 to 4 µg/mL cadmium in the final diluted solution for paints containing approximately 100 ppm cadmium, based on the solids. The size specified will give a concentration of approximately 10 to 20 µg/mL cobalt in the final diluted solution for paints containing approximately 500 ppm cobalt, based on the solids.

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muffle furnace and allow it to cool to room temperature. Break up the ash into fine particles with a glass stirring rod, leaving the rod with the container through the filtering step.

9.7 Add 10 mL of HNO_3 (1+1), taking care to avoid losses due to spattering in case the ash reacts vigorously with the acid. Heat carefully on a hot plate until 2 to 3 mL of solution remain. Add an additional 10 mL of HNO_3 (1+1) and continue heating on the hot plate until less than 5 mL of the solution remains.

9.8 Filter the solution through medium-porosity filter paper into a 50-mL volumetric flask. If the filtrate is not clear, refilter through fine-porosity filter paper. Wash the container three times with 2.5 mL of hot ammonium acetate solution (6.3), each time transferring the washings to the filter paper. Wash the filter paper several times with water. Adjust the volume to 50 mL with water and mix.

9.9 Aspirate the test solution and determine the absorbance in the same manner in which the instrument was calibrated. Determine the concentration of lead, cadmium, or cobalt in micrograms per millilitre from the calibration curve. If the absorbance is above the range covered by the calibration curve, dilute an aliquot of the sample solution to a suitable volume with ammonium acetate diluting solution.

NOTE 5—For maximum accuracy, calibration and standardization (Section 8) should be completed just prior to aspirating the sample solution.

NOTE 6—The method of standard additions may be used to improve the accuracy of the analysis. This method is particularly recommended for use with unknown samples where matrix effects may be potentially significant. For a detailed description of the procedure and calculations used in the method of standard additions, consult a standard text on atomic absorption spectroscopy or the instruction manual provided by the instrument manufacturer.

10. Calculation

10.1 Calculate the mean concentration of lead, cadmium, or cobalt in the nonvolatile portion of the sample as follows:

$$\text{lead, cadmium or cobalt, ppm (mg/kg) in nonvolatile} = (C \times F \times 5000)/(NV \times S)$$

where:

C = concentration of lead, cadmium, or cobalt in the aspirated specimen solution, ($\mu\text{g/mL}$),

F = dilution factor from 9.9 (volume diluted to/volume of aliquot),

5000 = factor derived from multiplying the 50-mL volume obtained in procedure in 9.8 by 100 (to convert NV used to a whole number) and 10^6 (to obtain ppm), then dividing by 10^6 (to convert grams of sample to μg),

NV = percent nonvolatile of paint sample (use 100 if sample was a dried film), and

S = sample, g.

11. Report

11.1 Report the lead, cadmium, or cobalt content of the nonvolatile content of the sample and whether the analysis was conducted on a liquid coating or a dried film.

12. Precision and Bias⁹

12.1 The precision estimates are based on an inter-laboratory study in which seven different laboratories analyzed in duplicate, on two different days, four samples of water-reducible paints and four samples of solvent-reducible paints containing from 0.01 to 5 % lead, 50 to 150 ppm (mg/kg) cadmium, and 50 to 2000 ppm cobalt. The within-laboratory coefficient of variation was found to be 3.4 % relative at 24 degrees of freedom for lead, 3.7 % relative at 15 degrees of freedom for cadmium, and 3.3 % relative at 21 degrees of freedom for cobalt. The between-laboratory coefficient of variation was 9.3 % relative at 20 df for lead, 8.9 % relative at 11 df for cadmium, and 5.5 % relative at 17 df for cobalt. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 11 % relative for all three metals.

12.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 28 % relative for lead or cadmium or by more than 17 % relative for cobalt.

12.2 Bias:

12.2.1 The true value for the amount of lead present in the solids of a coating should be between 97 and 111 % of the experimental value.

12.2.2 The true value for the amount of cadmium present in the solids of a coating should be between 77 and 143 % of the experimental value.

12.2.3 The true value for the amount of cobalt present in the solids of a coating should be between 96 and 120 % of the experimental value.

13. Keywords

13.1 AAS trace metals in paints; trace cobalt paints; trace lead cadmium paints; trace lead in paints

⁹ Supporting data are available from ASTM Headquarters. Request RR: D01-1005.

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Standard Guide for Testing Water-Borne Floor Paints¹

This standard is issued under the fixed designation D 3358; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been approved for use by the Department of Defense.

1. Scope

1.1 This guide covers the selection and use of procedures for testing water-borne floor paints. The test methods included are listed in Tables 1 and 2.

NOTE—Where no ASTM test method exists or there is a comparable (but not necessarily identical) method in Federal Test Method Standard, No. 141, the Federal Method number is listed. However, the Federal Method shall not be considered approved by ASTM.

1.2 The guide covers the testing of ready-mixed finish coats applied by brush or roller on surfaces subjected to traffic, such as floors, decks, platforms, stairs, and adjacent surfaces such as stair risers and railings.

1.3 Floor paints may be intended for interior use only or may also be suitable for use on exterior surfaces exposed to the weather, such as decks, porches, porch steps, and carport floors. Paints intended for both exterior and interior use require certain test procedures not applicable to paints intended for interior use only. Selection of test methods must be governed by the requirements in each individual case, together with agreement between the purchaser and the seller.

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 215 Methods of Chemical Analysis of White Linseed Oil Paints⁴
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁴
- D 358 Specification for Wood to Be Used as Panels in Weathering Tests of Coatings⁴
- D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive⁴

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.42 on Architectural Finishes.

Current edition approved March 25, 1988. Published May 1988. Originally published as D 3358 - 74. Last previous edition D 3358 - 79.

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

- D 659 Test Methods for Evaluating Degree of Chalking of Exterior Paints⁴
- D 660 Test Methods for Evaluating Degree of Checking of Exterior Paints⁴
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁴
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints⁴
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints⁴
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁴
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁴
- D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood⁴
- D 1210 Test Method for Fineness of Dispersion of Pigment Vehicle Systems⁴
- D 1308 Test Method for Effect of Household Chemical on Clear and Pigmented Organic Finishes⁴
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁴
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁴
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Material⁵
- D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁶
- D 1848 Classification for Reporting Paint Film Failures Characteristic of Exterior Latex Paints⁴
- D 1849 Test Method for Package Stability of Paint⁴
- D 2197 Test Method for Adhesion of Organic Coatings by Scrape Adhesion⁴
- D 2243 Test Method for Freeze-Thaw Resistance of Latex and Emulsion⁴
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
- D 2369 Test Method for Volatile Content of Coatings⁴
- D 2574 Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms⁴
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁴
- D 4062 Test Method for Leveling of Paints by Draw-Down Method⁴

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Discontinued; see 1988 Book of ASTM Standards, Vol 06.01. Replaced by Test Method D 522.

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁷

E 105 Practice for Probability Sampling of Materials⁵

2.2 U.S. Federal Test Method Standard 141.⁸

1021 Sampling (General)

2121 Application of Dipped Films

2141 Application of Brushed Films

3011 Condition in Container

4321 Brushing Properties

4335 Roller Coating Properties

3. Conditions Affecting Performance of Water-Borne Floor Paints

3.1 Practical requirements affecting and performance of water-thinned floor paints may vary with:

3.1.1 Substrate type, such as type and quality of wood, concrete, metal, or composition flooring. The use of water-borne floor paints is generally restricted to previously painted surfaces or unpainted concrete.

3.1.2 Substrate conditions such as porosity, hardness, smoothness, or in the case of unpainted concrete, alkalinity. Cleanliness is very important as traces of oily contaminants can cause water-borne floor paints to peel.

3.1.3 Environmental conditions such as temperature and humidity at the time of application and during drying. As these paints contain water as a thinner, the floor does not have to be completely dry before application. However, low temperature during the drying period may cause poor film formation.

3.1.4 *Substrate Aspects*—If construction defects are such that excessive moisture makes its way through the substrate, or if the substrate is in direct contact with damp ground, blistering, flaking, or peeling may result.

4. Definitions

4.1 For definitions of terms used in this guide, refer to Definitions D 16.

5. Sampling

5.1 Prior to sampling, the condition of the container should be established since damage to it may cause evaporation, skinning, or other undesirable effects in the coating. Determine the condition of the paint in accordance with 6.1 and 6.2.

5.2 Sample in accordance with Section 4.2.1 of Method 1021 of Federal Test Method No. 141. Determine the weight per gallon (or kilograms per litre) in accordance with Test Method D 1475. Repeat this procedure until successive values agree within 0.1 lb (45 g) or as agreed upon between the purchaser and the seller. Samples for testing may then be taken.

5.3 Specify the amount of sample, the package sizes, and identification codes to assure a representative sample. A 1-gal sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling plan, consult Practice E 105.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Method	Federal Test Method Standard No. 141
Liquid Paint Properties:			
Condition in container	6.1	...	3011
Coarse particles and foreign matter	6.2	D 185	...
Density or weight per gallon	6.3	D 1475	...
Fineness of dispersion	6.4	D 1210	...
Consistency	6.5	D 562	...
pH measurement	6.6	E 70	...
Freeze-thaw resistance	6.7	D 2243	...
Package stability	6.8	D 1849	...
Resistance to microorganisms	6.9	D 2574	...
Paint Application and Film Formation:			
Application of brushed film	7.1	...	2141
Brushing properties	7.2	...	4321
Roller coating properties	7.3	...	4335
Drying properties	7.4	D 1640	...
Leveling properties	7.5	D 4062	...
Producing films of uniform thickness	7.6	D 823	...
Appearance of the Dry Film:			
Color difference of opaque materials, visual evaluation of	8.1	D 1729	...
Color difference of opaque materials, instrumental evaluation of	8.2	D 2244	...
Gloss	8.3	D 523	...
Hiding power	8.4	D 2805, D 344	...
Properties of the Dry Film:			
Abrasion resistance	9.1	D 658, D 968	...
Elongation (flexibility)	9.2	D 522, D 1737	6221
Adhesion	9.3	D 2197	...
Resistance to chemicals	9.4	D 1308	...
Exterior exposure	9.5	...	...
Conducting exterior exposures and	9.5.1	D 1006	...
Reporting film features characteristic of exterior latex paints	9.5.1	D 1848	...
Wood panel description	9.5.2	D 358	...
Chalk resistance	9.5.3	D 659	...
Checking resistance	9.5.4	D 660	...
Cracking resistance	9.5.5	D 661	...
Erosion resistance	9.5.6	D 662	...
Flanking resistance	9.5.7	D 772	...
Analysis of Paint:			
Chemical analysis	10.1	...	...
Volatile content	10.2	D 2369	...
Pigment analysis	10.3	D 215	...

6. Liquid Paint Properties

6.1 *Condition in Container*—Thickening, settling, and separation are undesirable and objectionable if a paint cannot be reconditioned and made suitable for application with a reasonable amount of stirring. The referenced methods covers procedures for determining changes in properties of paints after storage. Determine the condition in the container in accordance with Method 3011 of Federal Test Method Standard No. 141.

6.2 *Coarse Particles and Foreign Matter*—Paints must be free of coarse particles to form uniform films of good appearance, a typical maximum being 1 weight % of total paint. The specified test with a No. 325 (45- μ m) sieve gives the percent of these particles in a paint. Determine coarse particles and foreign matter in accordance with Test Methods D 185.

⁷ Annual Book of ASTM Standards, Vol 15.05.

⁸ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., 19111-5094.

TABLE 2 Alphabetical List of Test Methods

Test Method	Section	ASTM Method	Federal Test Method Standard No. 141
Abrasion resistance	9.1	D 658, D 968	...
Adhesion	9.3	D 2197	...
Application of brushed films	7.1	...	2141
Brushing properties	7.2	...	4321
Chalk resistance	9.5.3	D 659	...
Checking resistance	9.5.4	D 660	6421
Chemical analysis	10.1	...	...
Coarse particles and foreign matter	6.2	D 185	...
Color differences of opaque materials, instrumental evaluation of	8.2	D 2244	...
Color differences of opaque materials, visual evaluation of	8.1	D 1729	4249
Condition in container	6.1	...	3011
Conducting exterior exposures	9.5.1	D 1006	...
Consistency	6.5	D 562	...
Cracking resistance	9.5.5	D 661	6471
Density or weight per gallon	6.3	D 1475	...
Drying properties	7.4	D 1840	...
Elongation	9.2	D 522, D 1737	6221
Erosion resistance	9.5.6	D 662	...
Exterior exposure	9.5	...	...
Fineness of dispersion	6.4	D 1210	...
Flaking resistance	9.5.7	D 772	...
Freeze-thaw resistance	6.7	D 2243	...
Gloss	8.3	D 523	...
Hiding power	8.4	D 2805, D 344	...
Leveling properties	7.5	D 4062	...
Package stability	6.8	D 1849	...
pH measurement	6.6	E 70	...
Pigment analysis	10.3	D 215	...
Producing films of uniform thickness	7.6	D 823	...
Reporting paint film features, characteristic of exterior latex paints	9.5.1	D 1848	...
Resistance to microorganisms	6.9	D 2574	...
Resistance to chemicals	9.4	D 1308	...
Roller coating properties	7.3	...	4335
Volatile content	10.2	D 2369	...
Wood panel description	9.5.2	D 358	...

6.3 Density or Weight per Gallon—The density as measured by weight in pounds per gallon (kilograms per litre) is used to assure product uniformity from batch to batch. In the referenced method, density is expressed as the weight in pounds of 1 U.S. gal (or kilograms per litre) of the paint at a specified temperature. Determine density in accordance with Test Method D 1475 using a calibrated cup.

6.4 Fineness of Dispersion—The more finely a pigment is dispersed, the more efficiently it is being used. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the material down a calibrated, tapered groove in a hardened steel block with the groove varying in depth from 4 to 0 mils (100 to 0 μm) and calibrated Hegman 0-8. The point at which continuous groupings of particles or agglomerates, or both, are visible through the surface of the liquid is taken as the fineness reading. Lower readings in mils or μm or higher readings in Hegman units indicate better fineness of dispersion. Determine fineness of dispersion in accordance with Test Method D 1210.

6.5 Consistency—Consistency is important, relating to application and flow, and should fall within a range agreed

upon. In the referenced method consistency is defined as the load in grams required to produce a specified rate of shear and also expressed in Krebs units. Other methods may be used as agreed upon between the purchaser and the seller. Determine consistency in accordance with Test Method D 562.

6.6 pH—The pH of a water-thinned paint depends on the type of latex used and the general formulation. It may vary from about 5 to 10. pH does not determine the quality of the paint and should not be used to assure product uniformity. A change in pH during storage may indicate poor stability or a change in properties of a water-thinned paint. Determine pH in accordance with Test Method E 70.

6.7 Freeze-Thaw Resistance—Water-thinned paints may be subjected to freezing conditions during shipping and storage. Suitably stabilized paints resist several cycles of freezing and thawing without showing deleterious changes. The referenced method covers the determination of the extent to which water-thinned floor paints, utilizing synthetic latices or synthetic resin emulsions as vehicles, retain their original properties when subjected to freezing and subsequent thawing. Determine freeze-thaw resistance in accordance with Test Method D 2243.

6.8 Package Stability—Since paints cannot normally be used immediately after manufacture, they must remain stable in the can for some time. At normal temperatures, most water-thinned floor paints can be stored for over a year with little change in properties. The referenced method covers the change in consistency and in certain related properties that may take place in packaged latex paint when stored at temperatures above freezing. Determine package stability in accordance with Test Method D 1849.

6.9 Resistance to Microorganisms—Bacteria in a water-borne floor paint can cause gassing, putrefactive or fermentative odors, and loss of viscosity. Determine if the paint contains living bacteria and if it is resistant to attack by bacteria in accordance with Test Method D 2574.

7. Paint Application and Film Formation

7.1 Application of Brushed Films—Brushed films should be smooth and free of seeds, color streaking, and brush marks. Test brush application in accordance with Method 2141 of Federal Test Method Standard No. 141.

7.2 Brushing Properties—The specified method covers a means for determining the brushing properties of coatings. The test is quite subjective; however, someone experienced in the art can produce quite consistent results, particularly in the determination of "drag" qualities. Floor paints are generally applied to horizontal surfaces but evaluation on vertical surfaces may be necessary to determine performance on stair risers, railings, posts, baseboards, etc. Determine the brushing properties in accordance with Method 4321 of Federal Test Method Standard No. 141.

7.3 Roller Coating Properties—Floor paints are frequently applied by roller. The referenced method outlines a procedure for evaluating a material's characteristics when applied by a roller. Determine roller coating properties in accordance with Method 4335 of Federal Test Method Standard No. 141.

7.4 Drying important in stair may be result in dust exterior surf appearance.

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7.5 Leveling when uniform and appeara tory determin liquid coating ance with Te

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8.4 Hiding measure of t however, de influenced b practical tes thickness is visually as c affected by paint. Test precise and a standard. Pa the effects o measured, a

7.4 Drying Properties—The drying time of a floor paint is important in determining when a freshly painted floor or stair may be subjected to traffic. Slow drying of the film may result in dust pickup, poor appearance and, if used on an exterior surface, rain or dew may cause a nonuniform appearance. Generally, water-thinned paints are dry-to-touch in 1 or 2 h when the water has evaporated from the film. Determine drying time in accordance with Test Methods D 1640.

7.5 Leveling Properties—Leveling is an important factor when uniform surfaces are to be produced, as it affects hiding and appearance. The referenced method covers the laboratory determination of the relative leveling characteristics of liquid coatings. Determine leveling characteristics in accordance with Test Method D 4062.

7.6 Producing Films of Uniform Thickness—Test Methods D 823 covers the preparation of various films of uniform thickness essential in conducting tests.

8. Appearance of the Dry Film

8.1 Color Difference of Opaque Materials by Visual Evaluation—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of the light source, illuminating and viewing conditions, size of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color difference in accordance with Practice D 1729.

8.2 Color Differences of Opaque Materials by Instrumental Evaluation—Color difference between a product and the standard can be measured by instrument. Generally the tolerance is agreed upon between the purchaser and the seller. The referenced method covers the calculation of instrumental determinations of small color differences, observable in daylight illumination, between nonfluorescent, nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (8.1) should be used to verify instrumental results. Calculate the color difference measured instrumentally in accordance with Method D 2244.

8.3 Gloss—Water-thinned floor paints generally have a semi-gloss finish which resists penetration of dirt and improves washability. A range from 20 to 40 may be considered typical. Determine the specular gloss in accordance with Test Methods D 523.

8.4 Hiding Power (Dry Opacity)—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness which is influenced by flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, opacity is evaluated visually as compared to a standard paint, and results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test that does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Determine

hiding power in accordance with Test Methods D 344 or D 2805.

9. Properties of the Dry Film

9.1 Abrasion Resistance—Abrasion resistance is a measure of the ability of a dried film to withstand wear from foot traffic and marring from objects rolled or pulled across the surface. Determine abrasion resistance in accordance with Test Methods D 658 or D 968.

9.2 Elongation—Elongation is a measure of the flexibility of a paint film. Determine elongation in accordance with Test Methods D 1737 or D 522.

9.3 Adhesion—Adhesion is the property of the film that resists removal from the substrate when scuffed or scraped. It is an important property in a floor paint. Determine adhesion in accordance with Test Method D 2197.

9.4 Resistance to Chemicals—An important property of a floor paint is its ability to resist spotting, softening, or removal when subjected to household chemicals or strong cleaners. Determine resistance to chemicals in accordance with Test Method D 1308.

9.5 Exterior Exposure—If the paint is intended for use on porches, decks, or outside stairways and railings, tests for resistance to exterior exposure may be required and may include the following:

9.5.1 Conducting Exterior Exposures—In conducting exterior exposures, refer to Practice D 1006 and Classification D 1848.

9.5.2 Wood Panel Description—In establishing exposure performance, use the panels as described in Specification D 358.

9.5.3 Chalking Resistance—Determine the chalk-resistance rating by reference to Test Method D 659.

9.5.4 Checking Resistance—Determine the checking resistance rating by reference to Test Method D 660.

9.5.5 Cracking Resistance—Determine the cracking resistance rating by reference to Test Method D 661.

9.5.6 Erosion Resistance—Determine the erosion resistance rating by reference to Test Method D 662.

9.5.7 Flaking Resistance—Determine the flaking resistance rating by reference to Test Method D 772.

10. Analysis of Paint

10.1 Chemical Analysis—If a specification requires certain raw materials or certain components in a given amount then chemical analysis is required to determine whether the specified components are present and in what amounts. Analysis does not necessarily establish paint quality which can also be greatly affected by manufacturing techniques. Most ASTM analytical methods apply to solvent-reducible coatings. However, some of these can be adapted for analysis of water-borne paints.

10.2 Volatile Content—The percent of volatile matter indicates the thinner loss from the film as it dries. This quantity subtracted from 100 % gives the nonvolatile content. Determine the volatile content in accordance with Test Method D 2369.

10.3 Pigment Analysis—The analysis of pigment may be required if the product is covered by a specification or if it is

agreed between the purchaser and the seller. Analyze the pigment in accordance with selected test procedures from Methods D 215.

11. Index Terms

11.1 This guide is indexed under the following terms: floor paints/coatings.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

1. Scope

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2. References

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¹ These Paint and Subcommittees published
² Annual
³ Annual



Standard Test Methods for Measuring Adhesion by Tape Test¹

This standard is issued under the fixed designation D 3359; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

These methods have been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover procedures for assessing the adhesion of coating films to metallic substrates by applying and removing pressure-sensitive tape over cuts made in the film.

1.2 Test Method A is primarily intended for use at job sites while Test Method B is more suitable for use in the laboratory. Also, Test Method B is not considered suitable for films thicker than 5 mils (125 μm).

NOTE 1—Subject to agreement between the purchaser and the seller, Test Method B can be used for thicker films if wider spaced cuts are employed.

1.3 These test methods are used to establish whether the adhesion of a coating to a substrate is at a generally adequate level. They do not distinguish between higher levels of adhesion for which more sophisticated methods of measurement are required.

NOTE 2—It should be recognized that differences in adherability of the coating surface can affect the results obtained with coatings having the same inherent adhesion.

1.4 In multicoat systems adhesion failure may occur between coats so that the adhesion of the coating system to the substrate is not determined.

1.5 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting³
- D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting²

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as D 3359 - 74. Last previous edition D 3359 - 87.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 02.05 and 06.06.

D 2370 Test Method for Tensile Properties of Organic Coatings²

D 3330 Test Method for Peel Adhesion of Pressure-Sensitive Tape of 180° Angle⁴

D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser²

3. Summary of Test Methods

3.1 *Test Method A*—An X-cut is made in the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0 to 5 scale.

3.2 *Test Method B*—A lattice pattern with either six or eleven cuts in each direction is made in the film to the substrate, pressure-sensitive tape is applied over the lattice and then removed, and adhesion is evaluated by comparison with descriptions and illustrations.

4. Significance and Use

4.1 If a coating is to fulfill its function of protecting or decorating a substrate, it must adhere to it for the expected service life. Because surface preparation (or lack of it) has a drastic effect on adhesion of coatings, a test method for evaluating adhesion to different surface treatments or of different coatings to the same treatment is of considerable use in the industry.

4.2 The limitations of all adhesion methods and the specific limitation of this test method to lower levels of adhesion (see 1.3) should be recognized before using it. The intra- and inter-laboratory precision of this test method is similar to other widely-accepted tests for coated substrates (for example, Test Method D 2370 and Test Method D 4060), but this is partly the result of it being insensitive to all but large differences in adhesion. The limited scale of 0 to 5 was selected deliberately to avoid a false impression of being sensitive.

TEST METHOD A—X-CUT TAPE TEST

5. Apparatus and Materials

5.1 *Cutting Tool*—Sharp razor blade, scalpel, knife or other cutting devices. It is of particular importance that the

⁴ Annual Book of ASTM Standards, Vol 15.09.

⁵ Permacel 99 manufactured by Permacel, New Brunswick, NJ 08903, and available from various Permacel tape distributors, is reported to be suitable for this purpose. The manufacturer of this tape and the manufacturer of the tape used in the interlaboratory study⁵ have advised this subcommittee that the properties of these tapes were changed. Users of it should, therefore, check whether current material gives comparable results to previous supplied material.

cutting edges be in good condition.

5.2 *Cutting Guide*—Steel or other hard metal straightedge to ensure straight cuts.

5.3 *Tape*—One-inch (25-mm) wide semitransparent pressure-sensitive tape with an adhesion strength agreed upon by the supplier and the user is needed. Because of the variability in adhesion strength from batch-to-batch and with time, it is essential that tape from the same batch be used when tests are to be run in different laboratories. If this is not possible the test method should be used only for ranking a series of test coatings.

5.4 *Rubber Eraser*, on the end of a pencil.

5.5 *Illumination*—A light source is helpful in determining whether the cuts have been made through the film to the substrate.

6. Test Specimens

6.1 When this test method is used in the field, the specimen is the coated structure or article on which the adhesion is to be evaluated.

6.2 For laboratory use apply the materials to be tested to panels of the composition and surface conditions on which it is desired to determine the adhesion.

NOTE 3—Applicable test panel description and surface preparation methods are given in Methods D 609 and Practices D 1730 and D 2092.

NOTE 4—Coatings should be applied in accordance with Test Methods D 823, or as agreed upon between the purchaser and the seller.

NOTE 5—If desired or specified, the coated test panels may be subjected to a preliminary exposure such as water immersion, salt spray, or high humidity before conducting the tape test. The conditions and time of exposure will be governed by ultimate coating use or shall be agreed upon between the purchaser and seller.

7. Procedure

7.1 Select an area free of blemishes and minor surface imperfections. For tests in the field, ensure that the surface is clean and dry. Extremes in temperature or relative humidity may affect the adhesion of the tape or the coating.

7.2 Make two cuts in the film each about 1.5 in. (40 mm) long that intersect near their middle with a smaller angle of between 30 and 45°. When making the incisions, use the straightedge and cut through the coating to the substrate in one steady motion.

7.3 Inspect the incisions for reflection of light from the metal substrate to establish that the coating film has been penetrated. If the substrate has not been reached make another X in a different location. Do not attempt to deepen a previous cut as this may affect adhesion along the incision.

7.4 Remove two complete laps of the pressure-sensitive tape from the roll and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 3 in. (75 mm) long.

7.5 Place the center of the tape at the intersection of the cuts with the tape running in the same direction as the smaller angles. Smooth the tape into place by finger in the area of the incisions and then rub firmly with the eraser on the end of a pencil. The color under the transparent tape is a useful indication of when good contact has been made.

7.6 Within 90 ± 30 s of application, remove the tape by seizing the free end and pulling it off rapidly (not jerked) back upon itself at as close to an angle of 180° as possible.

7.7 Inspect the X-cut area for removal of coating from the

substrate or previous coating and rate the adhesion in accordance with the following scale:

- 5A No peeling or removal,
- 4A Trace peeling or removal along incisions or at their intersection,
- 3A Jagged removal along incisions up to $\frac{1}{16}$ in. (1.6 mm) on either side,
- 2A Jagged removal along most of incisions up to $\frac{1}{8}$ in. (3.2 mm) on either side,
- 1A Removal from most of the area of the X under the tape, and
- 0A Removal beyond the area of the X.

7.8 Repeat the test in two other locations on each test panel. For large structures make sufficient tests to ensure that the adhesion evaluation is representative of the whole surface.

7.9 After making several cuts examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone before using again. Discard cutting tools that develop nicks or other defects that tear the film.

8. Report

8.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred that is, between first coat and substrate, between first and second coat, etc.

8.2 For field tests report the structure or article tested, the location and the environmental conditions at the time of testing.

8.3 For test panels report the substrate employed, the type of coating, the method of cure, and the environmental conditions at the time of testing.

8.4 If the adhesion strength of the tape has been determined in accordance with Test Method D 3330, report the results with the adhesion rating(s).

9. Precision and Bias⁶

9.1 In an interlaboratory study of this test method in which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion, the within-laboratories standard deviation was found to be 0.33 and the between-laboratories 0.44. Based on these standard deviations, the following criteria should be used for judging the acceptability of results, at the 95 % confidence level:

9.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than 1 rating unit for two measurements.

9.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by different operators should be considered suspect if they differ by more than 1.5 rating units.

9.2 Bias cannot be established for these test methods.

TEST METHOD B—CROSS-CUT TAPE TEST

10. Apparatus and Materials

10.1 *Cutting Tool*—Sharp razor blade, scalpel, knife or other cutting device having a cutting edge or edges angled.

⁶ Supporting data are available from ASTM Headquarters. Request RR D01-1008.

between 15 and 30° that will make either a single cut or several cuts at once. It is of particular importance that the cutting edge be in good condition.

10.2 *Cutting Guide*—If cuts are made manually (as opposed to a mechanical apparatus) a steel or other hard metal straightedge or template to ensure straight cuts.

10.3 *Rule*—Tempered steel rule graduated in 0.5 mm for measuring individual cuts.

10.4 *Tape*, as described in 5.3.

10.5 *Rubber Eraser*, on the end of a pencil.

10.6 *Illumination*, as described in 5.5.

10.7 *Magnifying Glass*—An illuminated magnifier to be used while making individual cuts and examining the test area.

11. Test Specimens

11.1 Test specimens shall be as described in Section 6.

12. Procedure

12.1 Where required or when agreed upon, subject the specimens to a preliminary test before conducting the tape test (see Note 3). After drying or testing, select an area free of blemishes and minor surface imperfections.

12.2 Place the panel on a firm base and under the illuminated magnifier make parallel cuts as follows:

12.2.1 For coatings having a dry film thickness up to and including 2.0 mils (50 µm) space the cuts 1 mm apart and make eleven cuts unless otherwise agreed upon.

12.2.2 For coatings having a dry film thickness between 2.0 mils (50 µm) and 5 mils (125 µm), space the cuts 2 mm apart and make six cuts. For films thicker than 5 mils use Test Method A.

12.2.3 Make all cuts about ¾ in. (20 mm) long. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. When making successive single cuts with the aid of a guide, place the guide on the uncut area.

12.3 After making the required cuts brush the film lightly with a soft brush or tissue to remove any detached flakes or ribbons of coatings.

12.4 Examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone. Make the additional number of cuts at 90° to and centered on the original cuts.

12.5 Brush the area as before and inspect the incisions for reflection of light from the substrate. If the metal has not been reached make another grid in a different location.

12.6 Remove two complete laps of tape and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece about 3 in. (75 mm) long.

12.7 Place the center of the tape over the grid and in the area of the grid smooth into place by a finger. To ensure good contact with the film rub the tape firmly with the eraser on the end of a pencil. The color under the tape is a useful indication of when good contact has been made.

12.8 Within 90 ± 30 s of application, remove the tape by seizing the free end and rapidly (not jerked) pulling it off at as close to an angle of 180° as possible.

12.9 Inspect the grid area for removal of coating from the substrate or from a previous coating using the illuminated magnifier. Rate the adhesion in accordance with the fol-

lowing scale illustrated in Fig. 1:

- 5B The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
- 4B Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
- 3B Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
- 2B The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
- 1B The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
- 0B Flaking and detachment worse than Grade 1.

12.10 Repeat the test in two other locations on each test panel.

13. Report

13.1 Report the number of tests, their mean and range, and for coating systems, where the failure occurred, that is, between first coat and substrate, between first and second coat, etc.

13.2 Report the substrate employed, the type of coating and the method of cure.

13.3 If the adhesion strength of the tape has been determined in accordance with Test Method D 3330, report the

Classification of Adhesion Test Results

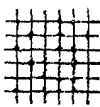
Classification	Surface of cross-cut area from which flaking has occurred. (Example for six parallel cuts)
5B	None
4B	
3B	
2B	
1B	
0B	Greater than 65%

FIG. 1 Classification of Adhesion Test Results

results with the adhesion rating(s).

14. Precision and Bias⁶

14.1 On the basis of two interlaboratory tests of this test method in one of which operators in six laboratories made one adhesion measurement on three panels each of three coatings covering a wide range of adhesion and in the other operators in six laboratories made three measurements on two panels each of four different coatings applied over two other coatings, the pooled standard deviations for within- and between-laboratories were found to be 0.37 and 0.7.

Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1 *Repeatability*—Provided adhesion is uniform over a large surface, results obtained by the same operator should be considered suspect if they differ by more than one rating unit for two measurements.

14.1.2 *Reproducibility*—Two results, each the mean of duplicates or triplicates, obtained by different operators should be considered suspect if they differ by more than two rating units.

14.2 Bias cannot be established for these test methods.

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Standard Practice for Operating Light- and Water-Exposure Apparatus (Unfiltered Open-Flame Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products Using the Dew Cycle¹

This standard is issued under the fixed designation D 3361; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the specific variations in test conditions that are applicable when Method 2 in Recommended Practice G 23 is employed for the exposure of paint and related coatings and materials using the dew cycle with unfiltered open-flame carbon arc.

NOTE 1—Another procedure for exposing these products is covered by Practice D 822 in which specimens are subjected to radiation from the enclosed carbon arc or the filtered open-flame carbon arc. The filters in Practice D 822 remove some of the shorter wavelength ultraviolet radiation not normally received at the earth's surface.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 523 Test Method for Specular Gloss²
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces²
- D 659 Test Method for Evaluating Degree of Chalking of Exterior Paints²
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints²
- D 822 Practice for Conducting Tests on Paint and Related Coatings and Materials Using Filtered Open-Flame Carbon-Arc Light- and Water-Exposure Apparatus²

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, Lacquer, and Related Products on Test Panels²

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings²

D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³

D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴

D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting⁴

D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting⁴

D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²

D 2616 Test Method for Evaluation of Visual Color Difference with a Gray Scale³

D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films²

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵

G 23 Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials⁶

3. Significance and Use

3.1 Organic coatings on exterior exposure are subjected to attack by degrading elements of the weather, particularly ultraviolet light, oxygen, and water. This practice may be used for evaluating the behavior of films exposed in apparatus that produces ultraviolet radiation, high temperatures, and water condensation on the films. This apparatus is used

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Nov. 27, 1987. Published January 1988. Originally published as D 3361 - 74. Last previous edition D 3361 - 81.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

⁶ Annual Book of ASTM Standards, Vols 06.01, 06.03, and 14.02.

to make an early materials comparison of the exterior exposure quality of paints. However, light sources, such as the carbon arc, that emit a significant amount of radiation at wavelengths shorter than those in natural sunlight, may cause results that lead to unrealistic evaluations of weathering properties.

3.2 This test method produces failure of films at a very rapid rate because the test is run without filters. The unfiltered carbon arc produces light that does not exist at the earth's surface. Failure caused by this light may bear no relationship to failures in natural sunlight. It is essential to have a control material of known performance of a similar vehicle type in every test. Substrates that have good thermal insulating properties should not be used because sufficient condensation cannot be obtained.

3.3 As no single light exposure apparatus, with or without water, can be specified as a direct simulation of natural exposure, this practice does not imply expressly, or otherwise, a specific correlation with outdoor exposure. It has, however, been useful in many instances.

3.4 Since climatic conditions vary with respect to time, geography, and topography, it may be expected that the effects of natural exposure will vary accordingly. All materials are not affected equally by the same environment. Results obtained by use of this practice should not be represented as equivalent to those of any outdoor weathering test unless the degree of quantitative correlation has been established for the material in question.

3.5 Variations in results may be expected when operating conditions among similar type instruments vary within accepted limits of this standard procedure.

4. Hazards

4.1 **Warning**—Never look directly at the carbon arc because ultraviolet radiation can damage the eye. Most carbon arc apparatus machines are equipped with door safety switches, but users of old equipment must be certain to turn the operate switch off before opening the test chamber door.

4.2 This light source generates ozone and nitrous oxides. Exhaust from the cabinet should be vented to the atmosphere.

5. Test Specimens

5.1 Unless otherwise agreed upon, choose panels that meet the applicable base panel requirements specified in Test Methods D 609 and Practices D 1730, D 1731, D 1732, or D 2092. Select panel sizes suitable for exhibiting the failure mode to be observed.

5.2 Apply the coatings to flat panels with the base panel material, method of application, coating system, film thickness, and method of drying consistent with the anticipated end use, or as mutually agreed upon between the producer and the user.

5.3 Unless otherwise agreed upon, coat test panels in accordance with Test Methods D 823 and measure the film thickness in accordance with an appropriate procedure selected from Test Methods D 1005, D 1186, D 1212, or D 1400. Nondestructive methods are preferred because panels so measured do not need to be repaired.

5.4 Unless otherwise specified, before exposing coated

panels in the apparatus, condition them at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity for one of the following periods in accordance with the type of coating:

Baked coatings	24 h
Radiation-cured coatings	24 h
All other coatings	7 days min

6. Apparatus

6.1 *Type EH Apparatus* (open-flame carbon arc), of Practice G 23, without filters.⁷

7. Procedure

7.1 Mount the test specimens vertically both above and below the horizontal center line of the source of radiation. Rotate the specimens from upper to lower rack to provide uniform exposure conditions over their surface. Specimens should be exposed approximately as many hours in the top rack as in the bottom rack, but need not be inverted.

7.2 Base temperature measurement and control on the black panel thermometer unit. Support the panel with the thermometer attached in the top row of the specimen drum or rack in the same manner as the test specimens so that it will be subjected to the same influences. If necessary, read black panel temperatures through the window in the test chamber without opening the door. The black panel temperature shall reach a maximum of $145 \pm 9^\circ\text{F}$ ($63 \pm 5^\circ\text{C}$) during the light-on-without-water-spray period of the test cycle and the equilibrium relative humidity of the air in the test chamber shall be $50 \pm 5\%$. The chamber temperature shall be $90 \pm 5^\circ\text{F}$ ($32 \pm 3^\circ\text{C}$) during the light-off-with-water-spray period of the test and the relative humidity of the air shall be $95 \pm 5\%$.

7.3 The water from the rack spray shall strike the back of the test specimens so that the only water on the face of the panels is from the dew formation caused by the chilled water on the back of the panels. The temperature of the water shall be $45 \pm 4^\circ\text{F}$ ($7.2 \pm 2^\circ\text{C}$). The water should have less than 20 ppm of solids.

7.4 Operate the apparatus on a cycle of 60 min dark with water-back spray and 60 min light without water spray.

8. Periods of Exposure

8.1 Use one of the following methods to determine the duration of the exposure under this practice:

8.1.1 A mutually agreed upon specified number of total hours (light and dark hours),

8.1.2 Number of total hours of exposure required to produce a mutually agreed upon amount of change in either the test specimen or an agreed upon standard sample.

9. Evaluation of Results

9.1 Evaluate conditions of exposed test specimens by means of one or more of the following standards: Test Methods D 523, D 610, D 659, D 660, D 661, D 662, D 714, D 772, D 2244, D 2616, D 4214, and E 97 and Practice D 1729. Select methods in accordance with product use requirements.

9.2 Because of possible variations in results as described in

⁷ Apparatus and carbon arcs from the Atlas Electric Devices Co., 4114 Ravenswood Ave., Chicago, IL 60613, have been found suitable for this purpose.

3.5, no reference tests corroborate accompanying reference procedure.

10. Report

10.1 Report
10.1.1 Test results

3.5, no reference should be made to results obtained from the tests conducted in the apparatus using this practice unless accompanied by Section 10 or unless otherwise specified in a reference procedure.

10. Report

10.1 Report the following information:

10.1.1 Type and model of apparatus used and type of

carbon arc,

10.1.2 Total hours of test (dark plus light hours),

10.1.3 Operating black-panel temperatures,

10.1.4 Operating relative humidities,

10.1.5 Test specimen preparation,

10.1.6 Results of evaluation tests, and

10.1.7 Identification of standards used for comparative evaluation.

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Standard Test Method for Film Hardness by Pencil Test¹

This standard is issued under the fixed designation D 3363; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Sections 2 and 3 were editorially renumbered in March 1989.

1. Scope

1.1 This test method covers a procedure for rapid, inexpensive determination of the film hardness of an organic coating on a substrate in terms of drawing leads or pencil leads of known hardness.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 A coated panel is placed on a firm horizontal surface. The pencil is held firmly against the film at a 45° angle (point away from the operator) and pushed away from the operator in a 1/4-in. (6.5-mm) stroke. The process is started with the hardest pencil and continued down the scale of hardness to either of two end points: one, the pencil that will not cut into or gouge the film (pencil hardness), or two, the pencil that will not scratch the film (scratch hardness).

3. Significance and Use

3.1 This test method is especially useful in developmental work and in production control testing in a single laboratory. It should be recognized that the results obtained may vary between different operators and laboratories. Every effort should be made to standardize the hardness of the lead used and the technique followed. If used as a basis for purchase agreement, this test method will achieve maximum precision if a given set of referee pencils be agreed upon between the purchaser and the seller.

4. Apparatus

4.1 A set of calibrated drawing leads (preferred) or equivalent calibrated wood pencils meeting the following scale of hardness:²

6B-5B-4B-3B-2B-B-HB-F-H-2H-3H-4H-5H-6H	
Softer	Harder

The difference between two adjacent leads shall be considered one unit of hardness.

4.2 *Mechanical Lead Holder*, for drawing leads if used.³

4.3 *Mechanical Sharpener*, draftsman-type, is helpful for trimming wood pencils if used.⁴

4.4 *Abrasive Paper*, grit No. 400.

5. Test Specimens and Conditions

5.1 Apply the surface coating by appropriate means to a smooth rigid substrate and cure properly, or use representative panels cut from coated stock. The panels used, the curing conditions, and the age of the coating prior to the test shall be within the limits agreed upon between the purchaser and the seller.

5.2 The film thickness of the coating shall be as specified or as agreed upon between the purchaser and the seller.

5.3 Conduct the test at $77 \pm 3.5^\circ\text{F}$ ($25 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity.

6. Procedure

6.1 For wood pencils, remove approximately 3/16 to 1/4 in. (5 to 6 mm) of wood from the point of each pencil using a draftsman-type mechanical sharpener, being careful to leave an undisturbed, unmarked, smooth cylinder of lead. Holding the pencil or lead holder (when using drawing leads) at an angle of 90° to the abrasive paper, rub the lead against the paper maintaining an exact angle of 90° to the abrasive paper until a flat, smooth and circular cross section is obtained, free of chips or nicks in the edge of the cross section. The desired edge may be attained by cementing the abrasive paper to a flat motor-driven disk. By supporting the pencil at 90° to the rotating disk a uniform flat lead end may be obtained more reproducibly.⁵

6.2 Place the coated panel on a level, firm, horizontal surface. Starting with the hardest lead, hold the pencil or lead holder firmly with the lead against the film at a 45° angle (point away from the operator) and push away from the operator. Exert sufficient uniform pressure downward and forward either to cut or scratch the film or to crumble the

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Oct. 25, 1974. Published November 1974.

² Pencils from many manufacturers have been and may be used in pencil hardness testing but it was apparent to the test group (D01.53.02) that the leads supplied by two manufacturers were more uniform, and reproducible results could be obtained from batch to batch of leads. These are Microtomic, manufactured by Faber Castell, 551 Spring Place Rd., Lewisburg, TN 37091, and Turquoise-T2375, manufactured by Mohawk, Route #30, Perth Road, Amsterdam, NY 12010.

³ Turquoise No. 10 is the preferred holder, however, any holder adequately constructed to eliminate lead slippage may be used.

⁴ Dexter Super 10 Draftsman Type A10D Cutter Assembly, manufactured by Apco Products, Inc., or equivalent, has been found suitable.

⁵ A suitable device for this purpose is available from Gardner Laboratories, Inc., P. O. Box 5728, Bethesda, Md.

edge of the lead. It is suggested that the length of the stroke be $\frac{1}{4}$ in. (6.5 mm).

6.3 Repeat the process down the hardness scale until a pencil is found that will not cut through the film to the substrate (either metal or a previous coat) for a distance of at least $\frac{1}{8}$ in. (3 mm) (see 7.1.1).

NOTE 1—The operator must watch closely for cutting into or scratching the film. Some finishes contain compounds that may tend to lubricate the film. Checks should be made by close visual inspection and by fingernail feel.

NOTE 2—In conducting the test, if the sharp edge of the lead is slightly chipped or crumbled, the lead must be resharpened.

6.4 Continue the process until a pencil is found that will neither cut through nor scratch the surface of the film. Any defacement of the film other than a cut (gouge) is considered a scratch. Record each end point (if applicable) for gouge and scratch hardness (see 7.1).

NOTE 3—With some films, the two end points will be identical.

6.5 Make a minimum of two determinations for gouge hardness (6.3) and scratch hardness (6.4) for each pencil or lead.

7. Report

7.1 Report the following information:

7.1.1 The two end points as follows:

7.1.1.1 *Gouge Hardness*—The hardest pencil that will

leave the film uncut for a stroke length of at least $\frac{1}{8}$ in. (3 mm).

7.1.1.2 *Scratch Hardness*—The hardest pencil that will not rupture or scratch the film.

7.1.2 The make and grade of lead or pencil used, and

7.1.3 Any deviation from standard conditions, including roughness in the finish.

8. Precision

8.1 In an interlaboratory test of this test method with three different films on panels, ten laboratories and operators, and repeated by switching leads and panels between laboratories, the within-laboratory standard deviation was found to be 0.52 and the between-laboratory standard deviation was found to be 0.61. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at a 95 % confidence level:

8.1.1 *Repeatability*—Two results obtained by two operators within a laboratory using the same pencils and panels should be considered suspect if they differ by more than one pencil unit on the scale described in 4.1.

8.1.2 *Operator Reproducibility*—Two results, each the mean of at least two determinations, obtained by operators in different laboratories using the same pencils and panels or different pencils with the same panels should be considered suspect if they differ by more than one pencil unit on the scale described in 4.1.

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Standard Guide for Testing Solvent-Reducible Floor Paints¹

This standard is issued under the fixed designation D 3383; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

[†] NOTE—Sections 3 and 4 were renumbered and the title was changed in October 1988.

1. Scope

1.1 This guide covers the selection and use of procedures for testing solvent-reducible floor paints and enamels. The test methods included are listed in Tables 1 and 2.

1.2 This guide covers the testing of ready-mixed finish coats applied by brush or roller on surfaces subjected to traffic, such as floors, decks, platforms, stairs, and adjacent surfaces such as stair risers and railings.

1.3 Floor paints may be intended for interior use only or may also be suitable for use on exterior surfaces exposed to the weather such as decks, porches, porch steps, and carport floors. Paints intended for both exterior and interior use will require certain test procedures not applicable to paints intended for interior use only. Selection of test methods must be governed by the requirements in each individual case, together with agreement between the purchaser and the seller.

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology of Terms Relating to Paint, Varnish, Lacquer, and Related Products²
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester³
- D 154 Guide for Testing Varnishes⁴
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints⁵
- D 215 Methods of Chemical Analysis of White Linseed Oil Paints⁴
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁴
- D 358 Specification for Wood to Be Used as Panels in Weathering Tests of Coatings⁴
- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive⁴

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.42 on Architectural Finishes.

Current edition approved May 25 and Oct. 26, 1979. Published December 1979. Originally published as D 3383-75. Last previous edition D 3383-75.

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vols 05.01, 06.01, and 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

- D 659 Method of Evaluating Degree of Chalking of Exterior Paints⁴
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints⁴
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁴
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints⁴
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints⁴
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁴
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁴
- D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood⁴
- D 1014 Test Method for Conducting Exterior Exposure Tests of Paints on Steel⁴
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁴
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁴
- D 1471 Test Method for Two-Parameter, 60-deg Specular Gloss⁶
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁴
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁴
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁷
- D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁸
- D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion⁴
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
- D 2245 Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints⁹
- D 2369 Test Method for Volatile Content of Coatings⁴
- D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁴

⁶ Discontinued; see 1975 Annual Book of ASTM Standards, Part 27.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

⁹ Annual Book of ASTM Standards, Vol 06.03.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Methods	Federal Test Method Standard No. 141
Liquid paint properties:			
Skimming	6.1	D 154	3021
Condition in container	6.2		3011.1
Coarse particles and foreign matter	6.3	D 185	4091
Density or weight per gallon	6.4	D 1475	4184.1
Fineness of dispersion	6.5	D 1210	4411.1
Consistency	6.6	D 562	4281
Absorption	6.7		4421
Flash point	6.8	D 93 or D 3278	
Paint application and film formation:			
Application of brushed film	7.1		2141.1
Brushing properties	7.2		4321.1
Roller coating properties	7.3		4335
Drying properties	7.4	D 1640	4061.1
Leveling properties	7.5	D 2801	
Producing films of uniform thickness	7.6	D 823	2121, 2162
Appearance of the dry film:			
Color differences of opaque materials, instrumental evaluation of	8.2	D 2244	6123
Color differences of opaque materials, visual evaluation of	8.1	D 1729	4249.1
Gloss	8.3	D 523, D 1471	6101
Hiding power	8.4	D 2805, D 344	
Properties of the dry film:			
Abrasion resistance	9.1	D 658, D 968	
Elongation (flexibility)	9.2	D 1737, D 522	6221
Adhesion	9.3	D 2197	6303.1
Resistance to chemicals	9.4	D 1308	
Exterior exposure	9.5		
Conducting exterior exposures on wood	9.5.1	D 1006	6161.1
Conducting exterior exposures on steel	9.5.1	D 1014	6160
Wood panel description	9.5.2	D 358	2031
Chalk resistance	9.5.3	D 659	6411
Checking resistance	9.5.4	D 660	6421
Cracking resistance	9.5.5	D 661	6471
Erosion resistance	9.5.6	D 662	6431
Flaking resistance	9.5.7	D 772	6441
Analysis of paint:			
Chemical analysis	10.1		
Volatile content	10.2	D 2369	4041.1
Pigment content	10.3	D 2371	4021, 4022
Pigment analysis	10.4	D 215	7261
Identification of vehicle solids	10.5	D 2621	
Identification of oils	10.6	D 2245	7501

D 2621 Test Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints⁴

D 2801 Test Method for Leveling Characteristics of Paints by Draw-Down Method¹⁰

D 2805 Test Method for Hiding Power of Paints by Reflectometry⁴

D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus⁹

E 105 Recommended Practice for Probability Sampling of Materials⁷

2.2 U.S. Federal Test Methods Standard 141:¹¹

1021 Inspection (General)

2121 Application of Dipped Films

2141.1 Application of Brushed Films

3011.1 Condition in Container

4321.1 Brush Properties

4335 Roller Coating Properties

4421 Absorption Test

3. Definitions

3.1 For definitions of terms used in this guide refer to Definitions D 16.

4. Conditions Affecting Performance of Solvent-Thinned Floor Paints

4.1 Practical requirements and performance of solvent-thinned floor paints may vary with:

4.1.1 Substrate type, such as type and quality of wood, concrete, metal, or composition flooring.

4.1.2 Substrate condition such as porosity, hardness, smoothness, and degree of cleanliness, or in the case of unpainted concrete, alkalinity.

4.1.3 Environmental conditions such as temperature and humidity at the time of application and during drying.

4.1.4 *Substrate Aspects*—If construction defects are such that excessive moisture makes its way through the substrate, or if the substrate is in direct contact with damp ground, blistering, flaking, or peeling may result.

5. Sampling

5.1 Prior to sampling, the condition of the container should be established since damage to it may cause evapora-

¹⁰ Discontinued; see 1989 Annual Book of ASTM Standards, Vol 06.01.

¹¹ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

TABLE 2 Alphabetical List of Test Methods

Test Methods	Section	ASTM Test Method	Federal Test Method Standard No. 141
Abrasion resistance	9.1	D 968	
Absorption	6.7		4421
Adhesion	9.3	D 2197	6303.1
Application of brushed films	7.1		2141.1
Brushing properties	7.2		4321.1
Chalk resistance	9.5.3	D 659	6411
Checking resistance	9.5.4	D 660	6421
Chemical analysis	10.1		
Coarse particles and foreign matter	6.3	D 185	4091
Color differences of opaque materials, instrumental evaluation	8.1	D 2244	6123
Color differences of opaque materials, visual evaluation	8.2	D 1729	4249.1
Condition in container	6.2		3011.1
Conducting exterior exposures on wood	9.5.1	D 1008	6161.1
Conducting exterior exposures on steel	9.5.1	D 1014	6160
Consistency	6.6	D 562	4281
Cracking resistance	9.5.5	D 661	6471
Density or weight per gallon	6.4	D 1475	4181.1
Drying properties	7.4	D 1640	4061.1
Elongation (flexibility)	9.2	D 1737, D 522	6221
Erosion resistance	9.5.6	D 662	6431
Exterior exposure	9.5		
Fineness of dispersion	6.5	D 1210	4411.1
Flaking resistance	9.5.7	D 772	6441
Flash point	6.8	D 93 or D 3278	
Gloss	8.3	D 523, D 1471	6101
Hiding power	8.4	D 2805, D 344	
Leveling properties	7.5	D 2801	
Oils, identification of	10.6	D 2245	7501
Pigment analysis	10.4	D 215	7261
Pigment content	10.3	D 2371	4021, 4022
Producing films of uniform thickness	7.6	D 823	2121, 2162
Resistance to chemicals	9.4	D 1308	
Roller coating properties	7.3		4335
Skimming	6.1	D 154	3021
Vehicle solids, identification of	10.5	D 2621	
Volatile content	10.2	D 2369	4041.1
Wood panel description	9.5.2	D 358	2031

tion, skinning, or other undesirable effects in the coating. Determine the condition of the coating in accordance with 6.1 and 6.2.

5.2 Sample in accordance with 4.2.1 of Method 1021 of U.S. Federal Test Method Standard No. 141. Determine the weight per gallon in accordance with Test Method D 1475. Repeat this procedure until successive readings agree within 0.1 lb (45 g) or as agreed upon between the purchaser and the seller. Samples for testing may then be taken.

5.3 Specify the amount of sample, the package sizes, and identification codes to assure a representative sample. A 1-gal (4-L) sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling plan consult Recommended Practice E 105.

6. Liquid Paint Properties

6.1 *Skimming*—Coatings containing a binder that dries by oxidation may be subject to skin formation in a partially filled can. Since skins are insoluble in the material, they must be removed before use. The referenced test method indicates the tendency of a paint to skin. Examine the original sample for skins, both on the surface and in its mass. On a well-mixed, skin-free portion of the sample, perform a skinning test in accordance with Guide D 154.

6.2 *Condition in Container*—Thickening, settling, and separation are undesirable and objectionable if a paint cannot be reconditioned and made suitable for application

with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties of paints after storage. Determine the condition in the container in accordance with Method 3011.1 of Federal Test Method Standard No. 141.

6.3 *Coarse Particles and Foreign Matter*—Paints must be free of coarse particles to form uniform films of good appearance, typical maximum being 1 weight % of total paint. The specified test with a No. 325 (45- μ m) screen gives the percent of these particles in a paint. Determine coarse particles and foreign matter in accordance with Test Methods D 185.

6.4 *Density or Weight per Gallon*—The density as measured by weight per gallon or kilogram per litre is used to assure product uniformity from batch to batch. In the referenced test method, the density is expressed as the weight in pounds avoirdupois of 1 U. S. gal (kilogram/litre) of the paint at a specified temperature. A calibrated weight-per-gallon cup is used. Determine density in accordance with Test Method D 1475.

6.5 *Fineness of Dispersion*—The more finely a pigment is dispersed, the more efficiently it is being used. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the material down a calibrated, tapered groove in a hardened steel block with the groove varying in depth from 4 to 0 mils (100 to 0 μ m). The point at which continuous groupings of particles or agglom-

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erates, or both, protrude through the surface of the liquid is taken as the fineness reading. Lower readings in mils or micrometres or higher readings in Hegman units indicate better fineness of dispersion. Determine fineness of dispersion in accordance with Test Method D 1210.

6.6 Consistency—Consistency is important, relating to application and flow, and should fall within a range agreed upon. In the referenced test method, consistency is defined as the load in grams required to produce a specified rate of shear. Determine consistency in accordance with Test Method D 562.

6.7 Absorption—The referenced method provides a rapid determination of the relative penetration of the binder into a porous surface. It provides a rough measure of the wetting and penetrating properties of liquid materials. Loss of binder and the resultant change in pigment volume concentration, as the film dries, are indicated by the test. Determine absorption in accordance with Method 4421 of Federal Test Method Standard No. 141.

6.8 Flash Point—When the flash point of a material is required for shipping information, use Test Methods D 93, Part B, or D 3278.

7. Paint Application and Film Formation

7.1 Application of Brushed Films—Brushed films should be smooth and free from seeds, color streaking, and brush marks. Test application of a brushed film in accordance with Method 2141.1 of Federal Test Method Standard No. 141.

7.2 Brushing Properties—The specified method covers a means for determining the brushing properties of coatings. The test is quite subjective; however, someone experienced in the art can produce quite consistent results, particularly in the determination of "drag" qualities. Floor paints are generally applied to horizontal surfaces but evaluation on vertical surfaces may be necessary to determine performance on stair risers, railings, posts, etc. Determine the brushing properties in accordance with Method 4321.1 of Federal Test Method Standard No. 141.

7.3 Roller Coating Properties—Floor enamels are frequently applied by roller. The referenced method outlines a procedure for making an evaluation of a material's characteristics when applied by a roller. Determine roller coating properties in accordance with Method 4335 of Federal Test Method Standard No. 141.

7.4 Drying Properties—The drying time of a floor paint is important in determining when a freshly painted floor or stair may be subjected to traffic. Slow drying of the film may result in dust pickup, poor appearance, and, if used on an exterior surface, rain or dew may cause a nonuniform appearance. The test can also be used to determine whether or not the drying properties of the paint have changed during storage in the container. Determine drying time in accordance with Test Method D 1640.

7.5 Leveling Properties—Leveling is an important factor when uniform surfaces are to be produced, as it affects hiding and appearance. The referenced test method covers the laboratory determination of the relative leveling characteristics of liquid coatings. Determine leveling characteristics in accordance with Test Method D 2801.

7.6 Producing Films of Uniform Thickness—The following test method covers the preparation of various films of

uniform thickness essential in conducting tests. Prepare films in accordance with Test Methods D 823.

8. Appearance of The Dry Film

8.1 Color Differences of Opaque Materials by Visual Evaluation—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, size of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color difference in accordance with Practice D 1729.

8.2 Color Differences of Opaque Materials by Instrumental Evaluation—Color difference between a product and the standard can be measured by instrument. Generally the tolerance is agreed upon between the purchaser and the seller. The referenced method covers the instrumental determination of small color differences, observable in daylight illumination, between nonfluorescent, nonmetameric, opaque surfaces such as paint specimens. If metamerism is suspected, visual evaluation (8.1) should be used to verify instrumental results. Conduct instrumental evaluation of color difference in accordance with Method D 2244.

8.3 Gloss—Floor enamels generally have a high initial 60-deg gloss reading when first applied. Determine the specular gloss in accordance with Test Methods D 523 or D 1471.

8.4 Hiding Power (Dry Opacity)—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness which is influenced by flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, opacity is evaluated visually as compared to a standard paint, and results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test which does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Determine hiding power in accordance with Test Methods D 344 or D 2805.

9. Properties of the Dry Film

9.1 Abrasion Resistance—Abrasion resistance is a measure of the ability of a dried film to withstand wear from foot traffic and marring from objects rolled or pulled across the surface. Determine abrasion resistance in accordance with Test Methods D 658 or D 968.

9.2 Elongation—Elongation is a measure of the flexibility of paint films. Determining elongation in accordance with Test Methods D 1737 or D 522.

9.3 Adhesion—Adhesion is the property of the film which resists removal from the substrate when scuffed or scraped. It is an important property in a floor paint. Determine adhesion in accordance with Test Methods D 2197.

9.4 Resistance to Chemicals—An important property of a floor paint is its ability to resist spotting, softening, or removal when subjected to household chemicals or strong

cleaners. Determine resistance to chemicals in accordance with Test Method D 1308.

9.5 *Exterior Exposure*—If the paint is intended for use on porches, decks, or outside stairways and railings, tests for resistance to exterior exposure may be required.

9.5.1 *Conducting Exterior Exposures*—In conducting exterior exposures, refer to Practice D 1006 or Test Method D 1014.

9.5.2 *Wood Panel Description*—In establishing exposure performance, use the panels as described in Specification D 358.

9.5.3 *Chalk Resistance*—Determine the chalk-resistance rating by reference to Method D 659.

9.5.4 *Checking Resistance*—Determine the checking resistance rating by reference to Test Method D 660.

9.5.5 *Cracking Resistance*—Determine the cracking resistance rating by reference to Test Method D 661.

9.5.6 *Erosion Resistance*—Determine the erosion resistance rating by reference to Test Method D 662.

9.5.7 *Flaking Resistance*—Determine the flaking resistance rating by reference to Test Method D 772.

10. Analysis of Paint

10.1 *Chemical Analysis*—If a specification requires certain raw materials or certain components in a given amount then chemical analysis is required. Chemical analysis determines whether the specified components are present and if they are, in what amounts. It does not necessarily establish

paint quality which can also be greatly affected by manufacturing techniques. No single schematic analysis is comprehensive enough to cover the wide variety of floor enamel composition.

10.2 *Volatile Content*—The percent of volatile matter indicates the thinner loss from the film as it dries. This quantity subtracted from 100 % gives the nonvolatile content. Determine the volatile content in accordance with Test Method D 2369.

10.3 *Pigment Content*—Pigment gives the hiding and color and influences many other properties of a coating. Determine the percent pigment in accordance with Test Method D 2371.

10.4 *Pigment Analysis*—The analysis of pigment may be required if the product is covered by a specification, or if it is agreed between the purchaser and the seller. Analyze the pigment in accordance with selected test procedures from Method D 215 and other appropriate ASTM Test methods.

10.5 *Identification of Vehicle Solids*—The type of nonvolatile vehicle used in a coating has a great influence on its properties. The referenced test method gives a procedure for identifying the main components of the binder. Determine the identity of the vehicle solids in accordance with Method D 2621.

10.6 *Identification of Oils*—The composition of the nonvolatile vehicle can, if desired, be further broken down into the types of oils present. Determine the identity of oils in accordance with Method D 2245.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Method of Evaluating the Lightfastness of Printed Matter¹

This standard is issued under the fixed designation D 3424; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers evaluation of the lightfastness of printed matter. Two methods of exposure, direct to artificial light and under glass to natural light, and three methods of rating the fastness are provided.

2. Referenced Documents

2.1 ASTM Standards:

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates³

D 2616 Test Method for Evaluation of Visual Color Difference With a Gray Scale²

G 24 Practice for Conducting Natural Light Exposures Under Glass²

G 25 Practice for Operating Enclosed Carbon-Arc Type Apparatus for Light Exposure of Nonmetallic Materials⁴

G 27 Practice for Operating Xenon-Arc Type Apparatus for Light Exposure of Nonmetallic Materials⁴

3. Significance

3.1 Lightfastness of printed matter is of particular importance in uses such as posters, billboard prints, advertising matter, and display materials generally.

4. Apparatus

4.1 *Method A, Exposure to Artificial Light*—The apparatus to be used is described in Section 3 of Practice G 27. An alternative light source is described in Section 3 of Practice G 25. The spectrum of the xenon arc is closer to the spectrum of daylight.

4.2 *Method B, Exposure to Natural Light Under Glass*—The apparatus to be used is described in Section 3 of Practice G 24.

5. Test Specimens

5.1 The type and number of test specimens to be used shall be defined in specifications covering the product being tested or shall be mutually agreed upon by the purchaser and the seller.

6. Procedure

6.1 Mask approximately one quarter of each print with a material opaque to light. Expose the prints for a period mutually agreed upon by the purchaser and the seller to the required conditions.

6.2 After exposure, compare the unexposed portion to the exposed part using one of the following three procedures as agreed upon by the purchaser and the seller:

6.2.1 Visual rating against a control exposed at the same time.

6.2.2 Visual rating using gray scale color chips in accordance with Method D 2616.

6.2.3 Instrumental measurement in accordance with Method D 2244.

7. Report

7.1 When a report is required, the designation of the specimen and the description or identification of the method used to prepare the specimen should be included.

7.2 The report will depend upon the procedure used for evaluating the results and should be agreed upon by the purchaser and the seller.

7.2.1 For Procedure 6.2.1, visual rating against a control exposed at the same time, viewing condition should be included as described in Practice D 1729. Report the specimen as being equal to or having more or less light fastness than the control.

7.2.2 For Procedure 6.2.2, visual rating using gray scale color chips, report information as in Method D 2616.

7.2.3 For Procedure 6.2.3, instrument measurement, report information as in Method D 2244.

8. Precision

8.1 For precision of the rating method, see Method D 2244 or Method D 2616, where applicable.

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials, and is the direct responsibility of Subcommittee D 01.56 on Printing Inks.

Current edition approved July 25, 1975. Published October 1975.

² Annual Book of ASTM Standards, Vol 14.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Discontinued, see 1979 Annual Book of ASTM Standards, Part 41.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

1. Scope

1.1 This standard covers the test methods for testing enamels. and 2. A Selection of the test methods governed by individual countries and the

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Standard Guide for Testing Solvent-Reducible Interior Semigloss Wall and Trim Enamels¹

This standard is issued under the fixed designation D 3425; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout, including the title, in October 1988.

1. Scope

1.1 This guide covers the selection and use of procedures for testing solvent-reducible interior wall and trim semigloss enamels. The test methods included are listed in Tables 1 and 2. All of these tests may not be required for each enamel. Selection of the test methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester³
- D 154 Guide for Testing Varnishes⁴
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints⁵
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁴
- D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁴
- D 1038 Definitions of Terms Relating to Veneer and Plywood⁶
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁴
- D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings⁴
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁴

- D 1543 Test Method for Color Permanence of White Architectural Enamels⁴
- D 1554 Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials⁶
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁴
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁷
- D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁸
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁴
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
- D 2369 Test Method for Volatile Content of Coatings⁴
- D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁴
- D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings⁴
- D 2698 Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging⁴
- D 2801 Test Method for Leveling Characteristics of Paint by Draw-Down Method⁹
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁴
- D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus¹⁰
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry¹¹
- E 105 Recommended Practice for Probability Sampling of Materials⁷
- 2.2 U. S. Federal Test Methods Standard 141:¹²
 - 1021 Sampling General
 - 2112 Preparation of Gypsum Wallboard Panels
 - 2131 Application of Sprayed Films
 - 2141 Application of Brushed Films
 - 3011.1 Condition in Container
 - 4203.1 Reducibility and Dilution Stability

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials, and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Aug. 1, 1980. Published December 1980. Originally published as D 3425 - 75. Last previous edition D 3425 - 79.

² Annual Book of ASTM Standards, Vols 06.01, 06.02 and 06.03.

³ Annual Book of ASTM Standards, Vols 05.01, 06.01 and 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁶ Annual Book of ASTM Standards, Vol 04.09.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

⁹ Discontinued; see 1989 Annual Book of ASTM Standards, Vol 06.01.

¹⁰ Annual Book of ASTM Standards, Vol 06.03.

¹¹ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

¹² Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
Liquid enamel properties:			
Skimming	6.1	D 154	3021
Condition in container	6.2	...	3011.1
Coarse particles and foreign matter	6.3	D 185	4091
Weight per gallon or density	6.4	D 1475	4184.1
Fineness of dispersion	6.5	D 1210	4411.1
Odor	6.6	...	4401
Consistency (viscosity)	6.7	D 562	4281
Rheological properties of non-newtonian liquids	6.8	D 2196	...
Dilution stability	6.9	...	4203.1
Absorption	6.10	...	4421
Flash point	6.11	D 93, D 3278	4293
Color compatibility	6.12	...	...
Enamel application and film formation:			
Application of brushed films	7.1	...	2141.1, 4321.1
Application by roller	7.2	...	2112
Application of sprayed films	7.3	...	2131
Drying properties	7.4	D 1640	4061.1
Leveling properties	7.5	D 2801	...
Working properties	7.6	...	4541
Sag resistance	7.7	...	4494
Wet film thickness	7.8	D 1212	...
Producing films of uniform thickness	7.9	D 823	2121, 2162
Appearance of dry enamel film:			
Color difference of opaque materials, visual evaluation	8.1	D 2244	4249.1
Color difference of opaque materials, instrument evaluation	8.2	D 1729	6123
Directional reflectance	8.3	E 97	6121
Gloss (60 deg specular)	8.4	D 523	6101
Hiding power	8.5	D 344, D 2805	...
Color change of white architectural enamels	8.6	D 1543	...
Properties of dry enamel film:			
Elongation (flexibility)	9.1	D 1737, D 522	6221
Washability	9.2	...	6141
Analysis of enamel:			
Chemical analysis	10.1	...	...
Volatile content	10.2	D 2369	4041.1
Pigment content	10.3	D 2371	4021, 4022
Analysis of pigment			
Nonvolatile content (volume percent)	10.4	...	...
	10.5	D 2697	4311

4321.1 Brushing Properties

4401 Odor Test

4421 Absorption Test

4494 Sag Test (Multinotch Blade)

4541 Working Properties and Appearance of Dried Film

6141 Washability of Paints

3. Definitions

3.1 For definition of terms used in this guide, refer to Definitions D 16, D 1554, and D 1038.

4. Conditions Affecting Performance of Solvent-Type Interior Semigloss Wall and Trim Enamel

4.1 Performance of solvent-reducible interior semigloss wall and trim enamels may vary with:

TABLE 2 Alphabetical List of Test Methods

Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
Absorption	6.10	...	4421
Analysis of pigment	10.4	...	...
Application of brushed films	7.1	...	2141.1, 4321.1
Application by roller	7.2	...	2112
Application of sprayed films	7.3	...	2131
Chemical analysis	10.1	...	...
Coarse particles and foreign matter	6.3	D 185	4091
Color change of white architectural enamels	8.6	D 1543	...
Color compatibility	6.12	...	...
Color difference of opaque materials, instrument evaluation	8.2	D 2244	6123
Color difference of opaque materials, visual evaluation	8.1	D 1729	4249.1
Condition in container	6.2	...	3011.1
Consistency (viscosity)	6.7	D 562	4281
Dilution stability	6.9	...	4203.1
Directional reflectance	8.3	E 97	6121
Drying properties	7.4	D 1640	4061.1
Elongation (flexibility)	9.1	D 1737, D 522	6221
Fineness of dispersion	6.5	D 1210	4411.1
Flash point	6.11	D 93, D 3278	4293
Gloss (60 deg specular)	8.4	D 523	6101
Hiding power	8.5	D 344, D 2805	...
Leveling properties	7.5	D 2801	...
Nonvolatile content (volume percent)	10.5	D 2697	4311
Odor	6.6	...	4401
Pigment content	10.3	D 2371, D 2698	4021, 4022
Producing films of uniform thickness	7.9	D 823	2121, 2162
Rheological properties of non-Newtonian liquids	6.8	D 2196	...
Sag resistance	7.7	...	4494
Sampling	5	E 105	...
Skimming	6.1	D 154	3021
Volatile content	10.2	D 2369	4041.1
Washability	9.2	...	6141
Weight per gallon or density	6.4	D 1475	4184.1
Wet film thickness	7.8	D 1212	...
Working properties	7.6	...	4541

4.1.1 Substrate, such as type and quality of drywall, plaster, wood, or hardboard.

4.1.2 Condition of substrates—presence of grease, dirt, and mold; also porosity, adhesion, and general condition of previous coating.

4.1.3 Preparation of previously painted substrates including detergent cleaning, solvent etching, and sanding.

4.1.4 Type and quality of primer or undercoat and time of drying before topcoating.

4.1.5 Environmental conditions, both general and specific, at the time of enamel application and immediately after application.

5. Sampling

5.1 The condition of the container prior to sampling, should be established since damage to it may cause evaporation, skinning, or other undesirable effects. Determine the condition of the enamel in accordance with 6.1 and 6.2.

5.2 Sample in accordance with Section 4.2.1 of Method 1021 of Federal Test Method Standard No. 141. Determine

the weight per gallon in accordance with Test Method D 1475. Repeat this procedure until successive readings agree within 0.1 lb (45 g) or as agreed between the purchaser and the seller. Then take samples for testing.

5.3 Specify the amount of sample, the package sizes, and identification codes to assure a representative sample. A 1-gal (4-L) sample is usually sufficient for the recommended tests but for guidance in selecting a sample plan consult Recommended Practice E 105.

6. Liquid Enamel Properties

6.1 *Skimming*—Enamels containing a binder, that dries by oxidation may be subject to skin formation in a partially filled can or by diffusion of air into a filled can. Since skins are insoluble in the enamel, they must be removed before use. This test in a partially filled container indicates the tendency of an enamel to skin. A typical minimum time for skinning in accordance with the referenced test method is 48 h. Examine the original sample for skins, both on the surface and in its mass. On a well-mixed, skin-free portion of the sample, perform a skinning test in accordance with Guide D 154.

6.2 *Condition in Container*—Thickening, settling, and separation are undesirable and objectionable if an enamel, after storage, cannot be readily reconditioned and made suitable for application with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties after storage. Determine the condition in the container in accordance with Method 3011.1 of Federal Test Method Standard No. 141.

6.3 *Coarse Particles and Foreign Matter*—Enamels must be free of oversize particles and foreign matter to form a uniform film of good appearance, a typical maximum being 0.5 weight % of total enamel. The specified test with a No. 325 (45- μ m) screen gives the percent of these particles in an enamel. Determine coarse particles and foreign matter in accordance with Test Method D 185.

6.4 *Weight per Gallon*—The density of an enamel as measured by weight per gallon (or kilograms per litre) is used to assure product uniformity from batch to batch and provides a check against the theoretical weight calculated from the formula. The referenced test method gives a procedure for measurement of the density of an enamel. The density is expressed as weight in pounds of 1 U.S. gal (kilograms per litre) of the enamel at a specified temperature. A calibrated weight-per-gallon cup is used. Determine weight per gallon in accordance with Test Method D 1475.

6.5 *Fineness of Dispersion*—One method for measuring the degree of dispersion is to draw the enamel down in a tapered groove in a hardened steel block with the groove varying in depth from 4 to 0 mils (100 to 0 μ m). The point at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the wet film is taken as the fineness reading. Lower readings in mils or micrometres or higher readings in Hegman units indicate better dispersion. A typical fineness of dispersion requirement is a reading of 1.0 mil (25 μ m) or 6 Hegman for interior semigloss wall and trim enamels. Determine fineness of dispersion in accordance with Test Method D 1210.

6.6 *Odor*—Some solvent combinations produce obnoxious odors, particularly when painting indoors with inadequate

ventilation and under adverse temperature-humidity conditions. Test for odor in accordance with Method 4401 of Federal Test Method Standard No. 141.

6.7 *Consistency*—Enamels of a given type should fall within a consistency range agreed upon. In the referenced test method, consistency is defined as the load in grams required to produce a specified rate of shear. Enamels for professional painters are usually formulated at a higher consistency range than consumer enamels. A typical range is 75 to 90-Krebs unit (KU) for consumer enamels and 90 to 100 KU for professional painter enamels. Determine the consistency of the product in accordance with Test Method D 562.

6.8 *Rheological Properties of Non-Newtonian Liquids*—Rheological properties are related to the application and flow properties of the liquid coating. The referenced test method covers the determination of the rheological properties of an enamel and is particularly suited for use with enamels that display thixotropic characteristics. In fact, viscosity under varying conditions of time and rotational speed of the spindle is measured. Determine the rheological properties in accordance with Test Method D 2196.

6.9 *Dilution Stability*—The diluent suggested for reduction should be readily incorporated into the enamel without excessive stirring or shaking. This test is a measure of the stability of an enamel that has been reduced to a desired viscosity, for example, for spray application. Determine dilution stability in accordance with Method 4203 of Federal Test Method Standard No. 141.

6.10 *Absorption*—The referenced method provides a rapid means for measuring the relative penetration of the binder into a porous surface. It provides a rough measure of the wetting and penetrating quality of liquid materials. Loss of binder and the resultant change in pigment volume concentration, as the film dries, are indicated by this test. Determine the absorption in accordance with Method 4421 of Federal Test Method Standard No. 141.

6.11 *Flash Point*—Organic solvents used in enamels have characteristic temperatures at which they will support combustion. This temperature is known as the flash point. It is often used for danger classification in shipment by common carriers. It is also used to determine conditions of storage to meet fire regulations and also the safety requirements of the Occupational Safety and Health Act (OSHA). Determine the flash point in accordance with Test Methods D 93 or D 3278.

6.12 *Color Compatibility*—If tinting colors are not adequately compatible with enamel tint base, lighter, darker, or nonuniform shades of color will develop. Tintability of white bases with colorants of standardized tinting strength is a trade requirement. The test method shall be agreed upon between the purchaser and the seller. An ASTM test method is now being developed.

7. Enamel Application and Film Formation

7.1 *Application by Brush*—Brushed films should be smooth and free of seeds and on vertical surfaces should show no sagging, color streaking, or excessive brush marks. Test application of brushed films in accordance with Method 2141.1 of Federal Test Method Standard No. 141.

7.2 *Application by Roller*—Walls are frequently painted

with rollers that produce slight stipple effects. Test roller coating properties in accordance with Method 2112 of Federal Test Method Standard No. 141.

7.3 Application by Spray—Interior paints and enamels are sometimes applied by spray. Both air and airless spray are used in commercial work. Test in accordance with Method 2131 of Federal Test Method Standard No. 141.

7.4 Drying Properties—The drying time of an enamel is determined by the composition of the enamel and by atmospheric conditions during drying. Insufficient drying may result in dirt pickup causing a poor appearance. Typical drying times are $\frac{1}{3}$ to 2-h set-to-touch and 18-h dry-hard. The test can also be used to determine whether the enamel has lost its drying properties during storage or whether drier was included in the product at the time of manufacture. Determine the drying time in accordance with Test Methods D 1640.

7.5 Leveling Properties—Leveling is a factor in hiding and appearance of wall and trim enamels. The referenced test method covers the laboratory determination of the relative leveling characteristics of liquid coatings. Determine leveling property in accordance with Test Method D 2801.

7.6 Working Properties—Working properties of an enamel are generally compared to a standard or described by requirement in the product specification. Test working properties in accordance with Method 4541 of Federal Test Method Standard No. 141.

7.7 Sag Resistance—Some enamels sag and form curtains before the film sets. Test for sag resistance in accordance with Method 4494 of Federal Test Method Standard No. 141.

7.8 Wet Film Thickness—Measurement of wet film thickness is useful in calculating spreading capacity or adjusting application to an agreed square feet per gallon (or square metres per litre). Determine wet film thickness in accordance with Method D 1212.

7.9 Producing Films of Uniform Thickness—The following test method covers the preparation of enamel films of uniform thickness essential in conducting various tests. Prepare films in accordance with Test Method D 823.

8. Appearance of Dry Enamel Film

8.1 Color Difference of Opaque Materials by Visual Evaluation—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced practice covers the illuminating and viewing condition to be used in the visual evaluation of color differences. Determine color difference in accordance with Practice D 1729.

8.2 Color Difference of Opaque Materials by Instrument Evaluation—Color difference between a product and the standard can be measured by instrument. Generally the tolerance is agreed upon between the purchaser and the seller, and may also be required if a production specification is involved. Determine the color difference in accordance with Method D 2244.

8.3 Directional Reflectance—This property is a measure of light reflected from the surface of the enamel. It usually is assigned a value in specifications for white and pastel shades. A typical minimum reflectance value is 86 for white.

Determine daylight directional reflectance in accordance with Test Method E 97.

8.4 Gloss (60-deg Specular)—Semigloss enamels are particularly sensitive to enamel hold-out of primers and undercoats. Low or uneven gloss readings are indicative of the defect. Interior semigloss enamels after drying 48 h are typically in the range from 40 to 70 when applied to glass. Gloss measurements taken after 7 days are more indicative of the final gloss of the dried film. Determine the specular gloss in accordance with Test Method D 523.

8.5 Hiding Power (Dry Opacity)—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness which is influenced by flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, opacity is evaluated visually as compared to a standard paint, and results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test which does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Determine hiding power in accordance with Test Methods D 344 or D 2805.

8.6 Color Change of White Architectural Enamels—Color permanence is an important requirement in interior white semigloss enamels. Lack of permanence is usually caused by after-yellowing. Determine color change in accordance with Test Method D 1543.

9. Properties of Dry Enamel Film

9.1 Elongation (Flexibility)—Elongation is a measure of flexibility of an enamel film. Determine elongation in accordance with Test Method D 1737 or D 522.

9.2 Washability—Washability is an important property of semigloss enamels in service. It is determined from gloss and reflectance measurements before and after removal of stains. Determine washability in accordance with Method 6141 of Federal Test Method Standard No. 141.

10. Analysis of Enamel

10.1 Chemical Analysis—If a specification requires certain raw materials or certain components in given amount then analysis is required. Chemical analysis is primarily a measure of uniformity. It does not necessarily establish paint quality which can also be greatly affected by manufacturing techniques. No single schematic analysis is comprehensive enough to cover the wide variety of semigloss wall and trim enamels.

10.2 Volatile Content—The percent of volatile matter indicates the thinner loss from the film as it dries. Determine volatile content of the paint in accordance with Test Method D 2369.

10.3 Pigment Content—Pigment gives the hiding and color and influences many other properties of the enamel. Determine percent pigment in accordance with Test Method D 2371.

10.4 Analysis of Pigment—The analysis of pigment may be required if the enamel is covered by a specification or if it is agreed between the purchaser and the seller. Determine

analysis of pigment in accordance with selected ASTM test methods.

10.5 *Nonvolatile Content (Volume Percent)*—Nonvolatile content volume percent is a useful figure in calculating

coverage or spreading capacity per gallon (or litre) at a specified dry film thickness. Determine nonvolatile content volume percent of the paint in accordance with Test Method D 2697.

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Standard Test Method for Washability Properties of Interior Architectural Coatings¹

This standard is issued under the fixed designation D 3450; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the relative ease of removing soilent discolorations from the dried film of an interior coating by washing with either an abrasive or nonabrasive cleaner.

1.2 This test method is limited to coatings having a CIE-Y reflectance of 60 % or more, as measured in accordance with Test Method E 97.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 562 Test Method for Consistency of Paints Using the Stormer Viscometer²

D 1193 Specification for Reagent Water³

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems²

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵

3. Terminology

3.1 Description of Term Specific to This Standard:

3.1.1 *soilent*—a discoloring substance that clings to the surface of a coating, whose dispersed color component is not in solution and therefore does not penetrate into the film.

3.1.1.1 *Discussion*—Soilent differs from stain in that the colorant of a stain is in solution, and therefore, can penetrate into the film.

4. Summary of Test Method

4.1 The test material is applied to a black plastic panel and allowed to dry for 7 days. The reflectance of the film is measured (R_1) and then a soilent consisting of carbon black dispersed in mineral oil is applied on the film. The panel is

placed on a glass plate in a washability machine and the film is washed with either an abrasive or nonabrasive scrub medium for 100 cycles. The panel is rinsed and dried and the reflectance in the stained area is read (R_2). The ratio of the reflectance, R_2/R_1 , is a measure of the degree to which the soilent has been removed.

5. Significance and Use

5.1 Interior architectural paints are often discolored by dirt and other soilent materials. This test method covers the relative ease and completeness of removal of a specific soilent from such a surface by scrubbing. The greater the ease of soil removal with a minimum of film erosion, the greater the useful service life is expected to be.

5.2 Results obtained by this test method do not necessarily correlate with all types of soilent.

5.3 Semigloss finishes generally require only the nonabrasive type cleaner for good soilent removal, whereas flat paints may require the abrasive type.

6. Apparatus

6.1 *Washability Tester*,⁶ also referred to as washability machine.

6.1.1 Accessory Apparatus:

6.1.1.1 *Glass Plate*, measured to fit, 17 $\frac{7}{8}$ by 6 $\frac{1}{2}$ by $\frac{1}{4}$ in. (454 by 165 by 6.3 mm).

6.1.1.2 *Stainless Steel Pan*.

6.1.1.3 *"C" Clamps*.

6.1.1.4 *Scale*, standard.

6.1.1.5 *White Blotter*.

6.2 *U-Shaped Film Caster*,⁷ having both a 7-mil (0.18-mm) clearance by 132 mm width and a 10-mil (0.25-mm) clearance by 138 mm width.

6.3 *Film Applicator*, 3 in. (76 mm) width with 3-mil (76- μ m) clearance.

6.4 *Suction Plate* for drawdowns (large size).⁸

6.5 45°, 0° *Reflectometer*, with green filter, as specified in Test Method E 97.

6.6 *Balance*, with 0.1-g scale graduation.

6.7 *Fineness of Dispersion Gage*, as specified in Test Method D 1210.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved August 31, 1990. Published November 1990. Originally published as D 3450 - 75. Last previous edition D 3450 - 86.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

⁵ *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

⁶ Model AG 8100, available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910, and Model "D" available from Paul N. Gardner Company, Inc., 316 N. E. First St., Pompano Beach, FL 33060 have been found suitable for this purpose. Other straight-line washability testers may be adapted to meet the requirements of this method. See RR D01-1065.

⁷ The Dow film caster has been found satisfactory for this purpose and is available from BYK-Gardner.

⁸ Available from BYK-Gardner. An equivalent may be used.

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1. Reagents and Materials

7.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁹ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

7.3 Black Plastic Panels.¹⁰

7.4 Sponge and Holder.¹¹

7.5 Masking Tape.

7.6 Soilant Medium,¹² consisting of the following:

	grams
Mineral oil, USP heavy	60
Odorless mineral spirits	32
Carbon black ¹³	8
	100

7.6.1 Mix components and disperse to a Hegman fineness of 7 (0.5 mils or 13 μ m) or when measured in accordance with Test Method D 1210.

7.7 Scrub Media (Note 1).

7.7.1 Nonabrasive Medium¹² consisting of the following:

	grams
Water	89.6 ⁴
Hydroxyethyl cellulose ¹⁴	2.0
Detergent ¹⁵	4.0
Trisodium phosphate, anhydrous	4.0
Acetic acid glacial	0.3 ^B
Preservative ¹⁶	0.1
	100.0

7.7.2 Abrasive Medium¹², consisting of the following:

	grams
Water	49.4 ^C
Hydroxyethyl cellulose ¹⁴	0.8
Ammonium hydroxide 28 %	—
Detergent ¹⁵	2.0
Trisodium phosphate, anhydrous	2.0
Silica ¹⁷	45.0
Acetic acid, glacial	0.7 ^D
Preservative ¹⁶	0.1
	100.0

⁴ Vary to achieve a final consistency of 165 to 220 g (75 to 85 Krebs Units) with a Stormer Viscometer in accordance with Test Method D 562.

⁹ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

¹⁰ Leneta, P-121-10N dull black plastic panels 6½ by 17 in. by 10 mils (432 by 165 by 0.25 mm) in size, obtainable from the Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ are suitable for this purpose. An equivalent may be used.

¹¹ Sponge is a cellulosic type, coarse pore grade, ¾ by 3 by 1½ in. (95 by 76 by 38 mm) meeting Federal Spec. L-S-626, Type II; available also from O'Cel-O Div., General Mills, Inc., 305 Sawyer Ave., Tonawanda, NY 14150. Brass holder measuring ¾ by 3 by 1 in. (95 by 76 by 25.4 mm).

¹² Laboratory standardized stain and scrub media have been found suitable and are available from the Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ. An equivalent may be used.

¹³ Printex U Beads, obtainable from DeGussa Corp., 65 Challenger Road, Ridgefield Park, NJ 07660, has been found suitable for this purpose. An equivalent may be used.

^B Vary to achieve a final pH from 9.5 to 10.0 in accordance with Test Method E 70.

^C Vary to achieve a final consistency of 475 to 600 g (110 to 120 Krebs Units) with the Stormer Viscometer in accordance with Test Method D 562.

^D Vary to achieve a final pH of 9.5 to 10.0 in accordance with Test Method E 70.

7.8 Slowly add the hydroxyethyl cellulose to the water while stirring mechanically. Stir until uniform and then slowly add 2 to 3 drops of 28 % ammonium hydroxide solution while mixing and continue mixing until the solution turns clear. In the order shown, add the other ingredients separately, stirring continuously. Be sure each item is uniformly dispersed before adding the next one. Add the silica slowly to ensure uniform dispersion. Finally, add the preservative and adjust the pH with glacial acetic acid.

NOTE 1—When a referee test is made, prepare fresh medium or use standardized scrub medium¹¹ from a previously unopened container that is no more than 1 year old.

8. Preparation of Apparatus

8.1 Washability Machine—The washability machine should be leveled before use and operated at 37 ± 1 cpm; each cycle consisting of a complete forward and reverse stroke. Adjust tautness of the cables with a spring scale to approximately 5 lb.

8.2 Sponge and Holder—Add sufficient weight to the holder in the form of lead sheets or other flat weights to give a combined weight of 1500 g, including the dry sponge.

NOTE 2—Check the compression of the damp sponge under the 1500-g weight to ensure that the holder does not drag along the panel and tear the film.

9. Procedure

9.1 Clean the top of the glass plate (or preferably the suction plate) and both sides of the black plastic panel to be sure they are free of specks. Place the black panel on the plate and tape one end to the plate. Smooth the panel along the plate to ensure a close fit.

9.2 Stir the test material thoroughly and strain to remove all skins and particles. Draw down the material on the panel, starting from the taped end of the panel. For latex paints, the 10-mil (0.25-mm) side of the applicator will generally be satisfactory, whereas the 7-mil (0.18-mm) side can be used for solvent-based coatings. However, the film thickness of paint to be applied may be varied as mutually agreed upon between the purchaser and the seller. The rate of application should be fairly slow, 3 to 4 s from end to end, to prevent

¹⁴ Hydroxyethyl cellulose having a molar substitution (MS) value from 1.8 to 2.5 and a viscosity of a 2 % solution in the range from 4400 to 6500 cps. Cellosize QP-4400 is available from Union Carbide Corp., P.O. Box 8720, South Charleston, WV 25303 and Natrosol 250M, available from Aqualon Co., 2711 Centerville Rd., Wilmington, DE 19850 have been found suitable for this purpose.

¹⁵ Octyl phenoxy polyethoxyethanol, as represented by Triton X-100, obtainable from Rohm & Haas Co., 2 Executive Campus, Suite 320, Cherry Hill, NJ 08002, has been found satisfactory for this purpose. An equivalent may be used.

¹⁶ 1,3,5-triethyl hexahydro-sym-triazine (Vancide TH), obtainable from R. T. Vanderbilt Co., 30 Winfield St., Norwalk, CT 06855, has been found satisfactory. An equivalent may be used.

¹⁷ Silica No. 22, obtainable from Whittaker, Clark and Daniels, Inc., 100 Church St., New York, NY 10007, has been found satisfactory for this purpose. This silica is an amorphous grade of 99+ % silicon dioxide. Average particle size is 2.95 μ m with 92 % being less than 40 μ m. Surface area is 7600 cm²/g, and will give a pH from 6.8 to 7.2 in reagent water. An equivalent may be used.

pinholes or holidays in the film. Air dry in a horizontal position for 7 days in a room preferably maintained at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, or under conditions mutually agreeable to the purchaser and the seller.

9.3 On the last day of drying, measure the directional reflectance (green filter) of a portion of the panel in accordance with Test Method E 97. Record this value as R_1 .

9.4 After reading the reflectance, use the applicator with the 3-mil (76- μm) clearance to draw down a film of soilant medium perpendicular to the coating film and in the area where the reflectance was measured. Permit the stained panel to dry for 16 to 24 h under the same drying conditions specified in 9.2.

9.5 Clean the glass surface on which the test panel is to be mounted and also the back of the test panel. Attach the test panel to the glass plate and place the mounted test panel, coated side up, in the pan. Clamp the assembly securely to the pan with the "C" clamps, centering the pan so that the sponge boat can move lengthwise on the panel.

9.6 Remove the sponge from the holder and soak it in reagent water at room temperature. Remove the sponge and squeeze repeatedly with one hand until no more water drips from the sponge. Replace the sponge in the holder and pour $15 \pm 1\text{ mL}$ of water on the exposed face of the sponge. Stir the scrub medium and spread uniformly across the face of the sponge $10.0 \pm 0.1\text{ g}$ of nonabrasive scrub medium. (Use a spatula if necessary, when applying the abrasive scrub medium, to apply uniformly over the sponge.)

9.7 Attach the sponge holder, with the sponge and cleaner face down, to the cables of the washability machine. Add 5 mL of water on each side of the holder in the path of the sponge. Start the motor and allow the sponge to travel 25 cycles at the rate of $37 \pm 1\text{ cpm}$. Ensure that the sponge and holder are level during the run, otherwise uneven wear will occur on the test panel. At the end of 25 cycles, stop the machine, remove the sponge, clean in running water, squeeze dry, and resoak in water. Wipe off excess stain outside the test area. Repeat the procedure starting in 9.6 with "remove the sponge and squeeze it repeatedly. . . ." for an additional 25 cycles. Repeat the 25-cycle procedure for a total of 100 cycles, each time trying to start and stop the sponge outside the stained area of the panel.

9.8 Remove the test panel from the pan, rinse with running tap water, gently moving the palm of the hand over the path of the sponge to remove clinging particles of scrub medium. Blot the panel and then allow to dry at room temperature. After the panel has dried thoroughly, wipe off excess stain outside the test area and measure reflectance in the soiled and washed area of the panel. Record as R_2 .

9.9 Repeat the test on a second panel and, for each, calculate the reflectance recovery in percent as $(R_2/R_1) \times 100$.

9.10 If the mean reflectance recovery for the two panels is less than 96 %, repeat the test on another set of panels using the abrasive scrub medium specified in 7.7.2 and report the results with both mediums.

10. Report

10.1 Report the mean of the two determinations and the type of scrub medium used.

10.2 Report any deviations from the standard procedure.

10.3 Note if erosion occurred in the soiled area.

11. Precision and Bias

11.1 On the basis of an interlaboratory study in which operators in six laboratories tested six coatings, including solvent and water-based systems, and covering a range of pigment volume concentrations (PVC's), the following criteria should be used for judging the acceptability of the results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 1.5 % in reflectance recovery with the abrasive cleaner and 9 % in reflectance recovery with the nonabrasive cleaner.

11.1.2 *Reproducibility*—Two results, each the mean of two measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 6 % in reflectance recovery with the abrasive cleaner and 25 % with the nonabrasive cleaner.

11.2 *Bias*—The concept of bias is not applicable to this test method.

12. Keywords

12.1 soilant resistance; washability

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Standard Practices for Testing Polymeric Powders and Powder Coatings¹

This standard is issued under the fixed designation D 3451; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last revision or approval. A superscript (c) indicates an editorial change since the last revision or approval.

¹ NOTE—Footnote numbers 8 through 18 inclusive were renumbered editorially in December 1987.

1. Scope

1.1 These practices cover the selection and use of procedures for testing polymeric powders and powder coatings. The test methods included are listed in Table 1. Where more than one test method is listed for the same characteristic, no attempt is made to indicate superiority of one method over another. Selection of the methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.

1.2 These practices describe the testing of polymeric powders as applied by electrostatic spray, fluidized bed, or any other applicable method.

1.3 These practices apply to proper and safe packaging, shipping and receiving, and storage and handling during use and application of polymeric powders.

1.4 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

- 2.1 *ASTM Standards:*
- B 117 Method of Salt Spray (Fog) Testing²
- D 153 Test Methods for Specific Gravity of Pigments³
- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁴
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces⁴
- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive⁴
- D 659 Test Method for Evaluating Degree of Chalking of Exterior Paint⁴
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paint⁴

¹ These practices are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D 01.51 on Powder Coatings.

Current edition approved June 25, 1976. Published August 1976. Originally published as D 3451-75. Last previous edition D 3451-75.

² Annual Book of ASTM Standards, Vol 13.02 and 06.01.

³ Annual Book of ASTM Standards, Vol 16.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁴
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints⁴
- D 714 Test Method for Evaluating Degree of Blistering of Paints⁴
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paint⁴
- D 792 Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement⁵
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion⁴
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁴
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁴
- D 1044 Test Method for Resistance of Transparent Plastics to Surface Abrasion⁵
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁴
- D 1193 Specification for Reagent Water⁶
- D 1308 Test Method for the Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁴
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base⁴
- D 1474 Test Methods for Indentation Hardness of Organic Coatings⁴
- D 1535 Test Method for Specifying Color by the Munsell System⁷
- D 1644 Test Methods for Nonvolatile Content of Varnishes⁴
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁷
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁸
- D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting⁸
- D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting⁸
- D 1733 Method of Preparation of Aluminum-Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products⁹

⁵ Annual Book of ASTM Standards, Vol 08.01.

⁶ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁹ Discontinued; see 1980 Annual Book of ASTM Standards, Vol 06.01.

TABLE 1 List of Test Methods

	Section	ASTM method		Section	ASTM method
Polymeric Powder Properties:			Gravimeter	24	D 3170
			Color, pigmented coatings	25	
	8		Visual	25.2	D 1535
	8.1	D 1921		25.3	E 308
Particle size and distribution					
Multiple sieve and analysis					

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TABLE 1 List of Test Methods

ASTM	Section	ASTM	Section
D 3170	24	D 1921	8.1
D 1536	25.2	D 1921	8.2
E 308	25.3	D 1921	8.3
D 2244	26.2	D 1921	8.4
D 2246	27	D 1921	9.2
D 522	28	D 1921	10
D 1737	28.2	D 1921	11
D 2803	29	D 1921	12
D 1400	20.5	D 1921	13
D 1166	20.5	D 1921	14
D 1005	20.5	D 1921	15
D 523	30	D 1921	16
D 1474	31	D 1921	17
D 1474	31.1	D 1921	18
D 1474	31.1	D 1921	18.1
D 1474	31.1	D 1921	18.2
D 1474	31.1	D 1921	18.3
D 2794	32	D 1921	19
D 714	33.2.1	D 1921	21
D 659	33.2.2	D 1921	21
D 660	33.2.3	D 1921	21
D 661	33.2.4	D 1921	21
D 610	33.2.5	D 1921	21
D 662	33.2.6	D 1921	21
D 772	33.2.7	D 1921	21
D 2081	34	D 1921	21
B 117	35	D 1921	21
D 1736	36.3	D 1921	21
D 870	37	D 1921	21
D 3170	24	D 1921	8
D 1536	25.2	D 1921	8.1
E 308	25.3	D 1921	8.2
D 2244	26.2	D 1921	8.3
D 2246	27	D 1921	8.4
D 522	28	D 1921	9.2
D 1737	28.2	D 1921	10
D 2803	29	D 1921	11
D 1400	20.5	D 1921	12
D 1166	20.5	D 1921	13
D 1005	20.5	D 1921	14
D 523	30	D 1921	15
D 1474	31	D 1921	16
D 1474	31.1	D 1921	17
D 1474	31.1	D 1921	18
D 1474	31.1	D 1921	18.1
D 1474	31.1	D 1921	18.2
D 1474	31.1	D 1921	18.3
D 2794	32	D 1921	19
D 714	33.2.1	D 1921	21
D 659	33.2.2	D 1921	21
D 660	33.2.3	D 1921	21
D 661	33.2.4	D 1921	21
D 610	33.2.5	D 1921	21
D 662	33.2.6	D 1921	21
D 772	33.2.7	D 1921	21
D 2081	34	D 1921	21
B 117	35	D 1921	21
D 1736	36.3	D 1921	21
D 870	37	D 1921	21

D 2794 Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)⁴

D 2803 Test Method for Fullform Corrosion Resistance of Organic Coatings on Metal⁴

D 2967 Test Method for Edge Coverage of Coating Powders¹²

D 3170 Test Method for Chipping Resistance of Coatings¹

D 3359 Test Methods for Measuring Adhesion by Tape Test¹

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁷

E 308 Method for Computing the Colors of Objects By Using the CIE System⁷

3.1 All tests shall be made in diffused light (not direct sunlight) and at 23 ± 2°C and 50 ± 5 % relative humidity, after conditioning 16 h unless otherwise specified.

4. Definitions

4.1 bulk density—mass per unit volume of a material in powder form including the air trapped between particles.

4.2 coating powder—finely divided particles of organic polymer, either thermoplastic or thermosetting, which generally contain pigments, fillers, and additives and which

D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus⁴

D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus¹⁰

D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Material¹¹

D 1898 Practice for Sampling of Plastics¹¹

D 1921 Test Method for Particle Size (Sieve Analysis) of Plastic Materials¹¹

D 2091 Test Method for Print Resistance of Lacquers⁴

D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting⁴

D 2197 Test Method for Adhesion of Organic Coatings by Scraping Adhesion⁴

D 2201 Test Method for Preparation of Hot-Dipped Non-passivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁴

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴

D 2246 Test Method for Finishes on Primed Metallic Substrates for Humidity-Thermal Cycle Cracking⁴

D 2248 Practice for Detergent Resistance of Organic Finishes⁴

D 2454 Practice for Determining the Effect of Overbaking on Organic Coatings⁴

¹⁰ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

¹¹ Annual Book of ASTM Standards, Vol 08.02.

¹² Annual Book of ASTM Standards, Vol 10.02.

remain finely divided during storage under suitable conditions.

4.3 *film formation from a coating powder*—the forming of a continuous film by melting powder particles and coalescing them by the application of energy. For thermosetting materials, a chemical reaction, either condensation or addition, also takes place. This fused film has the uniformity, color, toughness, and other properties associated with protective and decorative coatings.

4.4 *fluidity*—the ability of a powder to move freely, uniformly, and continuously (somewhat like a liquid) when subjected to certain conditions of pressure, temperature, and velocity of a carrier gas.

4.5 *gel time*—interval required at a given temperature for a powder to be transformed from a dry solid to a gel-like state.

4.6 *particle size*—average diameter of an object having irregular boundaries that can be described in an artificial way as having a diameter.

4.7 *particle-size distribution*—arrangement of particle size measurements on a powder in groups of specified diameters.

4.8 *pourability*—the ability of a powder to flow uniformly or to be continuously poured from a container at a steady rate.

4.9 *powder coatings*—coatings which are protective or decorative, or both, formed by the application of a coating powder (4.2) to a substrate and fused into a continuous film by the application of heat or radiant energy.

4.10 *storage stability*—the ability of a powder to maintain uniform physical and chemical properties after being subjected to specified storage conditions.

4.11 *volatile content*—the quantity expressed as weight percent of the powder which is lost under specified conditions of temperatures and time.

4.12 *cloud chamber technique*—method of moving a charged or uncharged object through a charged or uncharged cloud of powder in an enclosed chamber.

4.13 *electrostatic deposition*—technique of moving and charging powder so that it is deposited onto an oppositely charged substrate by one of the following methods.

4.13.1 *fluidized bed technique*—method of moving a grounded object over or through an oppositely charged fluidized powder.

4.13.2 *spray technique*—method of spraying and charging powder so that it is deposited onto an oppositely charged substrate.

4.14 *nonelectrostatic deposition*—technique of moving powder onto a substrate which may be heated above the fusion point of the powdered material.

4.14.1 *spray technique*—method of spraying powder onto a substrate which may be heated above the fusion point of the powdered material.

4.14.2 *fluidized bed technique*—method of dipping a hot object into a container holding the powder suspended in a gas stream so that it resembles a fluid and allowing the powder to melt onto the object being coated.

4.14.3 *flame spread technique*—method of applying powder using a compressed gas and melting the powder in a

flame before the powder impinges on the surface.

5. Sampling

5.1 Sample the powder in accordance with Practice D 1898.

5.2 Prepare specimens as required for the specific tests on the coating.

6. Equipment

6.1 Use the equipment as specified in each method of test.

7. Conditions Affecting Polymeric Powder or Powder Coatings, or Both

7.1 Practical requirements and performance of powder and powder coating may vary with:

7.1.1 *Substrate Type*—Ferrous, nonferrous types, plastic, or elastomeric.

7.1.2 *Substrate Weathering*—Weathering of the substrate will probably adversely affect the performance.

7.1.3 The type, quality, and suitability of the metal treatment or primer used under the powder coating and the time before coating application.

7.1.4 Application conditions.

7.1.5 Contaminants on the surface of the substrate.

7.1.6 Damage to container, size of container, storage time, excessive temperature, and temperature fluctuations which may cause settling, caking, or chemical change.

POLYMERIC POWDER PROPERTIES

8. Particle Size and Distribution

8.1 *Multiple Sieve Analysis*:

8.1.1 This method employs multiple sieves to determine particle sizes and their distribution. Standard 200-mm (8-in.) diameter sieves and a mechanical shaker are used. A representative quantity of the sample is sifted through a series of sieves and the amount retained on each sieve is weighed and calculated as percent of the total specimen.

8.1.2 *Apparatus*:

8.1.2.1 *Sieves*—Half-height sieves, 200 mm (8 in.) in diameter, conforming to the requirements of Specification E 11. A selection of sieves encompassing the expected range of particle sizes together with a cover and a bottom pan are required.

8.1.2.2 *Mechanical Sieve Shaker with Automatic Time Switch*¹³—The mechanical sieve-shaking device shall be capable of imparting uniform rotary motion and a tapping action at the rate of 150 ± 10 taps/min.

8.1.2.3 *Balance*—A laboratory balance with a minimum capacity of 500 g sensitive to 0.1 g, for weighing the specimen and the residues retained on the sieves.

8.1.3 *Procedure*:

8.1.3.1 If the residues are not to be transferred for weighing, weigh each of the selected sieves and the bottom pan to 0.1 g. Nest the desired sieves in order of diminishing openings with the coarsest sieve on top and the pan on the bottom.

¹³ Ro-Tap Sieve Shaker has been found suitable for this purpose and is available from W. S. Tyler Co., Cleveland, Ohio 44114. An equivalent may be used.

8.1.3.2 Weigh out a 100 ± 0.1 -g specimen and transfer it to the top sieve of the stack.

NOTE 1—If necessary this test may be made on a specimen of any size from 50 to 200 g. The weight of specimen used shall be stated in the report.

8.1.3.3 Place the cover on the top sieve, and place the stack in the mechanical sieve shaker. Operate the shaker for $10 \text{ min} \pm 15 \text{ s}$.

8.1.3.4 After shaking, carefully separate the stack of sieves, beginning at the top, and weigh the quantity of material retained on each sieve and that contained in the pan to 0.1 g. This may be accomplished either by transferring the fractions to the balance or by weighing the sieve or pan and its contents, and subtracting the tare weight from the total. If the material is transferred to the balance, carefully brush the sieve on both sides to ensure that adhering particles are transferred.

8.1.3.5 Calculate the percent in each fraction as follows:

$$\% = (R/S) \times 100$$

where:

R = residue weight, g, and

S = specimen weight, g.

NOTE 2—Ordinarily there is a small loss of dust as indicated by the cumulative total weight being less than 100 %. If this loss is not over 2 %, the amount reported through the finest sieve shall be increased until the total of all portions of the sample equals 100 %. If the cumulative total is less than 98 %, repeat the test.

8.1.4 Report—The report shall include the following:

8.1.4.1 Complete identification of the sample,

8.1.4.2 Specimen weight,

8.1.4.3 Percent of material retained on each sieve,

8.1.4.4 If required, the total cumulative percent of material retained on each sieve and in the pan,

8.1.4.5 Temperature, and

8.1.4.6 Relative humidity.

8.2 Vacuum Sieve Analysis:

8.2.1 Summary of Method:

8.2.1.1 In this method sieving is accomplished by aerodynamics so that there is no reduction in particle size and absolutely no wear on the sieves.

8.2.1.2 The housing holds a standard 200-mm (8-in.) sieve and a transparent sieve cover. A slit nozzle rotates slowly below the sieve. An air current, produced by a standard vacuum cleaner-type device, is blown upwards through a hollow shaft and the slit nozzle to the sieve, where it clears the screen. The particles thus suspended in air between the sieve and the sieve cover are separated as the air current circulates. The fine materials are blown through the sieve and into a filter bag, while the coarse materials remain on top of the sieve. A manometer indicates the vacuum inside the housing.

8.2.2 Apparatus:

8.2.2.1 Sieves—As described in 8.1.2.1 and transparent sieve cover.

8.2.2.2 Vacuum Sieve Apparatus—A vacuum sieving device¹⁴ capable of maintaining inside the housing a vacuum

of 5 ± 0.5 in. (127 ± 12.7 mm) of water as measured by a manometer. The slit nozzle which rotates below the sieve shall revolve at a speed of 25 ± 2 rpm.

8.2.2.3 Balance—See 8.1.2.3.

8.2.3 Procedure:

8.2.3.1 If the residues are not to be transferred for weighing, weigh each of the selected sieves to 0.1 g.

8.2.3.2 Weigh out a 100 ± 0.1 g specimen and transfer it to one of the sieves.

NOTE 3—If necessary, this test may be made on a specimen of any size from 10 to 50 g. The weight of specimen used shall be stated in the report. For small sieve openings, 1 to 10 g is recommended.

8.2.3.3 Place the transparent cover on the sieve and place the sieve in position on the vacuum sieving apparatus. Operate the sieving apparatus for $5 \text{ min} \pm 15 \text{ s}$ at a vacuum of 5 ± 0.5 in. (127 ± 12.7 mm) of water.

8.2.3.4 Remove the sieve and cover from the apparatus and weigh the quantity of material retained on the sieve and adhering to the cover to the nearest 0.1 g. This may be accomplished either by transferring the retained material to the balance or by weighing the sieve, cover, and contents, and subtracting the tare weight from the total. If the material is transferred to the balance carefully brush the sieve on both sides, and removed all powder from the cover to ensure that all adhering particles are transferred.

8.2.3.5 Repeat the procedure for each sieve used.

8.2.4 Calculate the percent retained on each sieve as follows:

$$\% = (R/S) \times 100$$

where:

R = residue weight, g, and

S = specimen weight, g.

8.2.5 Report—The report shall include the following:

8.2.5.1 Complete identification of the sample,

8.2.5.2 Specimen weight,

8.2.5.3 Percent of material retained on each sieve,

8.2.5.4 Temperature, and

8.2.5.5 Relative humidity.

8.3 Sonic Sifter Analysis:

8.3.1 Summary of Method:

8.3.1.1 In the sonic sifter, the sieves are stationary and agitation is imparted to the particles by an oscillating column of air. Sieve wear and particle attrition are minimal.

8.3.1.2 The sonic sifter consists of a sieving chamber, a diaphragm at the top vibrating at 60 Hz, and a motor with the necessary controls. The amplitude of vibration is adjustable to the nature of the specimen.

8.3.2 Apparatus:

8.3.2.1 Sieves—See 8.1.2.1.

8.3.2.2 Sonic Sifter—A sonic sieving device¹⁵ utilizing a diaphragm vibrating at 60 Hz with controls to adjust the sift amplitude and the pulse amplitude.

8.3.2.3 Balance—An analytical balance with a minimum capacity of 150 g sensitive to 1 mg for weighing the specimen and the residues retained on the sieves.

8.3.3 Procedure:

¹⁴ Alpine Air Jet Sieve, Model 200, has been found suitable for this purpose and is available from Alpine American Corp., Michigan Drive, Natick, Mass. 01760. An equivalent may be used.

¹⁵ Model L3P Sonic Sifter has been found suitable for this purpose and is available from ATM Corp., P.O. Box 2405, Milwaukee, Wisc. 53214. An equivalent may be used.

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8.3.3.1 Weigh and record the weight of each sieve, and of the collar, cover, and fines collector.

8.3.3.2 Weigh out a 2.5 ± 0.001 -g specimen.

8.3.3.3 Stack the sieves in descending order, coarsest on top, finest on the bottom, and transfer the specimen to the top sieve.

8.3.3.4 Insert the sieve stack assembly in the sieve chamber and lock. Set the sift amplitude at 3 and the pulse amplitude at 10. Operate the sifter for $3 \text{ min} \pm 15 \text{ s}$.

8.3.3.5 Weigh each sieve, the fines collector, cover, and the collar, subtract the tare weight for each, and calculate the percent retained on each sieve.

NOTE 4—Include any material adhering to the cover and the collar in the weights for the top sieve.

8.3.4 Report—The report shall include the following:

8.3.4.1 Complete identification of the sample;

8.3.4.2 Specimen weight,

8.3.4.3 Percent of material retained on each sieve,

8.3.4.4 Temperature, and

8.3.4.5 Relative humidity.

8.4 Electronic Counting Analysis:

8.4.1 Summary of Method:

8.4.1.1 This method, one of several found valuable for the measurement of particle size, covers the determination of the particle size distribution of powder coatings (0.6 to $80.0 \mu\text{m}$) using the electronic particle counter.¹⁶ This instrument uses an electric current path of small dimensions which is modulated by individual particle passage through an aperture, and produces individual pulses of amplitude proportional to the particle volume.

8.4.1.2 The powder is carefully dispersed ultrasonically or by some other appropriate method and a portion is added to a filtered electrolyte. Particle counts are obtained at several selected particle size levels and converted to relative weight fractions assuming constant particle density.

8.4.1.3 The instrument response is essentially to particle volume (liquid displacement); therefore equivalent spherical diameter is commonly used to express the particle size.

8.4.2 Apparatus:

8.4.2.1 Electronic Particle Counter,¹⁶ detecting particles using electric current through a liquid resistor.

8.4.2.2 Aperture Tube, $200\text{-}\mu\text{m}$ diameter. The diameter required is dependent upon the particle size distribution of the sample. Generally any given tube will cover a particle size range from 2 to 40 % of its aperture diameter.

8.4.2.3 Beaker, 250 to 500-ml, capable of maintaining all particles uniformly in suspension (for example, baffled or round bottom).

8.4.2.4 Ultrasonic Agitator.

8.4.2.5 Membrane Filtering Device, $0.45\text{-}\mu\text{m}$ filters.¹⁷

8.4.3 Reagents:

8.4.3.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications

of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.¹⁸ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.4.3.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

8.4.3.3 Dispersing Medium—5 % solution of octyl phenoxy polyethoxyethanol¹⁹ in reagent water.

8.4.3.4 Electrolyte—Dissolve 10.0 g of reagent grade sodium chloride (NaCl) in 1000 ml of reagent water, add some bactericide such as 70 mg of sodium trichlorophenate then filter twice through the membrane (8.4.2.5).

8.4.3.5 Wash Water—Reagent water filtered twice.

8.4.4 Procedure:

8.4.4.1 Precalibrate the aperture and electrolyte combination following the manufacturer's instruction manual.

NOTE 5—Calibration should be carried out using a lycopodium of microspheres suspension of $18.04\text{-}\mu\text{m}$ diameter with the half-count procedure as given in the instruction manual. It is necessary to have mutual agreement on monosized system diameters for interlaboratory comparisons.

8.4.4.2 Cone and quarter the sample until a specimen of convenient size is obtained.

8.4.4.3 Disperse the specimen by adding dispersing medium from the eye-dropper. Use gentle spatulation until a pasty consistency is obtained. Pick up a small portion of this mixture (about 0.1 ml more or less depending on the particle size of the powder) on a small spatula and rinse with wash water into a 25-ml beaker containing 5 ml of dispersing medium. Place the beaker in the ultrasonic agitator for 30 s and then rinse into 200 ml of electrolyte in the 250 to 500-ml beaker. Fill the beaker to capacity and place in the counting position of the counter stand.

8.4.4.4 Fill the aperture tube with electrolyte. Check the aperture for blockage and clear with a brush if necessary. Set the instrument controls for the smallest particle appropriate for the aperture in use. Typical instrument settings are shown in Figs. 1, 2, or 3.

8.4.4.5 Take three cumulative counts with 500- μl volume manometer and calculate the mean, which should be between 2700 and 5000. If it is not, adjust the particle concentration until it is. If more powder is added, it must be treated the same as the original specimen, except it may be rinsed directly into the beaker after ultrasonic agitation. If the suspension needs dilution, drain some of the suspension through a clean glass tube while agitating and then refill the beaker with electrolyte.

8.4.4.6 Set the instrument controls for normal accumulation of differential data. Typical instrument settings are shown in Figs. 1, 2, and 3. Take three counts at each size level for averaging.

¹⁸ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

¹⁹ Triton X-100 has been found suitable for this purpose and is available from Rohm and Haas Co., Independence Square, Philadelphia, PA. An equivalent may be used.

¹⁶ Models ZB with M2 accessory, Model B with Model M accessory, Model T, Model TA, and Model TALL have been suitable for this purpose and are available from Coulter Electronics, Inc., 590 W. 20 St., Hialeah, FL 33010. An equivalent may be used.

¹⁷ The membrane filters may be Millipore, Gelman, General Electric, Nuclepore, Selas Flotronics metal type, or their equivalent.

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FIG. 1 Model "B" or "Z" Typical Worksheet.

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Model T Worksheet									
SAMPLE									
ELECTROLYTE Isoton			DISPERSANT Triton X-100 5%						
EQUIPMENT		SERIAL		Aper. Dia.	Ser. No.	CALIBRATION DATA		Part. Dia.	W
ORGANIZATION		DATE		200				18.04	7
OPERATOR		DATE							
$k = d \sqrt{\frac{2W}{A}}$				± I A	3.2	$\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^2 \frac{(W_2 - W_1)}{W_1}$ For Model T			
FOR MODEL T				CALIB. A	241				
				APERTURE DIA.	200				
SAMPLE DATA									
Geometric Mean μ^3	Volume μ^3	Diameter μ	Channel (W)						
.00575	.004091	.198							
.0115	.008181	.250							
.0231	.01636	.315							
.0452	.03272	.397							
.0925	.06545	.500							
.1851	.1309	.630							
.3702	.2618	.794							
.7405	.5236	1.00							
1.481	1.047	1.26							
2.962	2.094	1.59							
5.924	4.189	2.00							
11.85	8.378	2.52							
23.70	16.76	3.17							
47.39	33.51	4.00							
94.78	67.02	5.04							
189.6	134.0	6.35							
379.1	268.1	8.00							
758.3	536.2	10.08							
1516.	1072.	12.7							
3033.	2145.	16.0							
6065.	4289.	20.2							
12.13 x 10 ³	8579.	25.4							
24.27 x 10 ³	17.16 x 10 ³	32.0							
48.54 x 10 ³	34.31 x 10 ³	40.3							
97.18 x 10 ³	68.63 x 10 ³	50.8							
194.4 x 10 ³	137.3 x 10 ³	64.0							
388.7 x 10 ³	274.5 x 10 ³	80.6							
777.4 x 10 ³	549.0 x 10 ³	101.6							
1.555 x 10 ⁶	1.098 x 10 ⁶	128.							
3.109 x 10 ⁶	2.196 x 10 ⁶	161.							
6.219 x 10 ⁶	4.392 x 10 ⁶	203.							
12.44 x 10 ⁶	8.784 x 10 ⁶	256.							
24.89 x 10 ⁶	17.57 x 10 ⁶	322.							
49.75 x 10 ⁶	35.14 x 10 ⁶	406.							
99.50 x 10 ⁶	70.27 x 10 ⁶	512.							
199.0 x 10 ⁶	140.6 x 10 ⁶	645.							
398.0 x 10 ⁶	281.1 x 10 ⁶	812.							
796.0 x 10 ⁶	562.2 x 10 ⁶	1024.							

NOTE—Typical settings for smallest particle (8.4.4.4).
Cumulative count data readout required for (8.4.4.5).

FIG. 2 Model "T" Typical Worksheet

9.1.2.3 At 24-h intervals perform the following tests until failure or as agreed upon between the purchaser and the seller.

(a) Particle size and particle size distribution (see Section 8).

(b) Gel time (see Section 14).

(c) Spray a panel of the powder coating and bake as specified. Compare with a panel prepared with unheated powder.

(d) If there is noticeable caking, test for pourability by Method D 1895.

9.1.3 Report:

9.1.3.1 Duration of test,

9.1.3.2 Particle size distribution before and after testing,

9.1.3.3 Gel time before and after testing,

9.1.3.4 General appearance of panels coated with aged and unaged powders, and

9.1.3.5 Degree of caking.

Model TA & TAIL Worksheet

Model TA & TAIL Worksheet									
SAMPLE _____									
ELECTROLYTE Isoton			DISPERSANT Triton X-100 5%						
EQUIPMENT	SERIAL	Aper. Dia.	Ser. No.	CALIBRATION DATA	Part. Dia.	W	± I A	A	
ORGANIZATION		200			18.04	9	4.0	167.8	
OPERATOR	DATE								
± I A		4.0			$\frac{A_2}{A_1} = \left(\frac{d_1}{d_2}\right)^3 2^{(W_2 - W_1)}$ For Model TA				
CALIB. A		189							
APERTURE DIA.		200							
SAMPLE DATA									
Geometric Mean μ^3	Volume μ^3	Diameter μ	Channel (W)						
.00575	.004091	.198							
.0115	.008181	.250							
.0231	.01636	.315							
.0462	.03272	.397							
.0925	.06545	.500							
.1851	.1309	.630							
.3702	.2618	.794							
.7405	.5236	1.00							
1.481	1.047	1.26							
2.962	2.094	1.59							
5.924	4.189	2.00							
11.85	8.378	2.52							
23.70	16.76	3.17							
47.39	33.51	4.00	3*						
94.78	67.02	5.04	4						
189.6	134.0	6.35	5						
379.1	268.1	8.00	6						
758.3	536.2	10.08	7						
1516.	1072.	12.7	8						
3033.	2145.	16.0	9						
6066.	4289.	20.2	10						
12.13 x 10³	8579.	25.4	11						
24.27 x 10³	17.16 x 10³	32.0	12						
48.54 x 10³	34.31 x 10³	40.3	13						
97.18 x 10³	68.63 x 10³	50.8	14						
194.4 x 10³	137.3 x 10³	64.0	15						
388.7 x 10³	274.5 x 10³	80.6	16						
777.4 x 10³	549.0 x 10³	101.6							
1.555 x 10⁶	1.098 x 10⁶	128.							
3.109 x 10⁶	2.196 x 10⁶	161.							
6.219 x 10⁶	4.392 x 10⁶	203.							
12.44 x 10⁶	8.784 x 10⁶	256.							
24.88 x 10⁶	17.57 x 10⁶	322.							
49.75 x 10⁶	35.14 x 10⁶	406.							
99.50 x 10⁶	70.27 x 10⁶	512.							
199.0 x 10⁶	140.6 x 10⁶	645.							
398.0 x 10⁶	281.1 x 10⁶	812.							
796.0 x 10⁶	562.2 x 10⁶	1024.							

NOTE—Typical setting for smallest particle (8.4.4.4).
Cumulative count data readout required for (8.4.4.5).

FIG. 3 Model TA and TAI Typical Worksheet

9.2 Package Stability (Moisture):

9.2.1 *Open-Dish Method*—Weigh 10 g of powder into an uncovered 50-mm diameter aluminum dish and place at $40 \pm 3^\circ\text{C}$, $95 \pm 5\%$ relative humidity, or conditions as agreed upon between the purchaser and the seller. Check for caking or change of gel time after 48 h.

10. Pourability

10.1 Test for pourability in accordance with Test Method

D 1895.

11. Fluidity

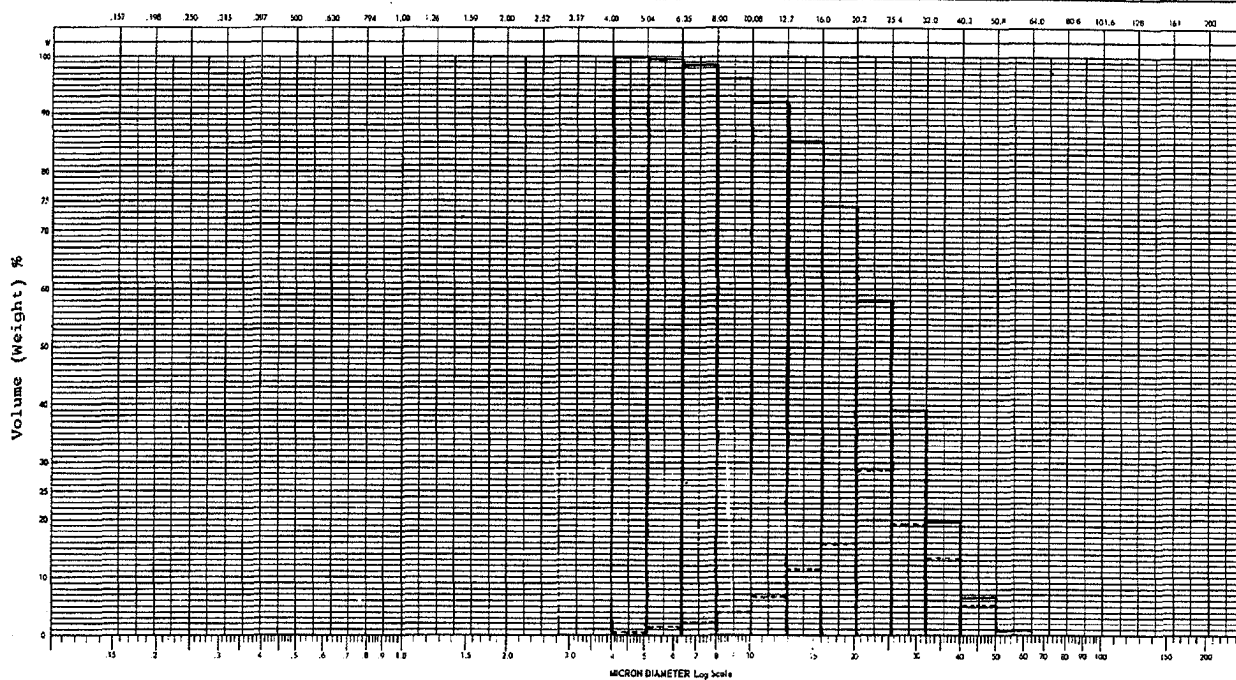
11.1 A method is under consideration.

12. Nonvolatile Content

12.1 Apparatus:

12.1.1 *Analytical Balance*, sensitive to 0.1 mg.

PARTICLE SIZE ANALYSIS										.15 - 200 μ % PERCENT	
ORGANIZATION				$L = d \sqrt{\frac{2}{\pi}}$ $\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^3$ when $W_2 = W_1$ $\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^3$ when $R_2 = W_1$				SAMPLE SETTINGS			
OPERATOR				FOR MODEL T				FOR MODEL TA			
EQUIPMENT				APERTURE	SERIAL	PART NO.		W	$\pm$ IA	A	DL
SAMPLE				ELECTROLYTE		DISPERGANT					



Cumulative Value Plot - - - - Differential Value Plot
FIG. 4 Size Distribution Curve on Semilog Paper

12.1.2 *Aluminum Dishes*, 60 mm in diameter and 17 mm deep.

12.2 Procedure:

12.2.1 Weigh two aluminum dishes to 0.1 mg and weigh by difference two specimens of 1.9 to 2.1 g or as otherwise specified of coating powder into them.

12.2.2 By gentle tilting spread the powder uniformly over the bottom of the dishes.

12.2.3 Heat the dishes and contents in an oven for 2 h at $60 \pm 2^\circ\text{C}$ or at $100 \pm 2^\circ\text{C}$ or at the temperature specified between the purchaser and the seller. Cool in a desiccator and weigh.

12.3 *Calculation*—Calculate the nonvolatile content as follows:

$$\text{Nonvolatile content, weight \%} = (C - A) 100/S$$

where:

A = weight of dish, g,

S = weight of specimen, g, and

C = weight of dish and contents after heating, g.

13. Volatile Content at Baking or Fusion Temperature

13.1 Use the apparatus and procedure given in Section 12 but heat the specimen at the normal baking temperature and

time unless otherwise specified.

13.2 Calculate the percent volatile matter by subtracting the nonvolatile content from 100.

14. Gel Time or Stroke Curve (for Thermosetting Powders Only)

14.1 Apparatus:

14.1.1 *Hot Plate*, suitable for use at 200°C .

14.1.2 *Stop Watch or Electric Timer*, 0.1 s interval.

14.1.3 *Wooden Tongue Depressor*, 6 by 1 in. (150 by 25 mm) or *Wooden Splints*, 6 by 1/4 in. (150 by 6 mm).

14.1.4 *Aluminum Foil*.

14.1.5 *Analytic Balance*.

14.1.6 *Pyrometer*, suitable for use at 200°C .

14.2 Procedure:

14.2.1 Measure 0.5 ± 0.1 g of powder.

14.2.2 Cover the hot plate with aluminum foil or use a non-silicon mold release lubricant. Set the heat to $190 \pm 1^\circ\text{C}$ or as agreed between the purchaser and seller, and check with the pyrometer.

NOTE 6—A shroud may be required to prevent air circulation from cooling the hot plate.

14.2.3 Drop the powder specimen on the foil and start the

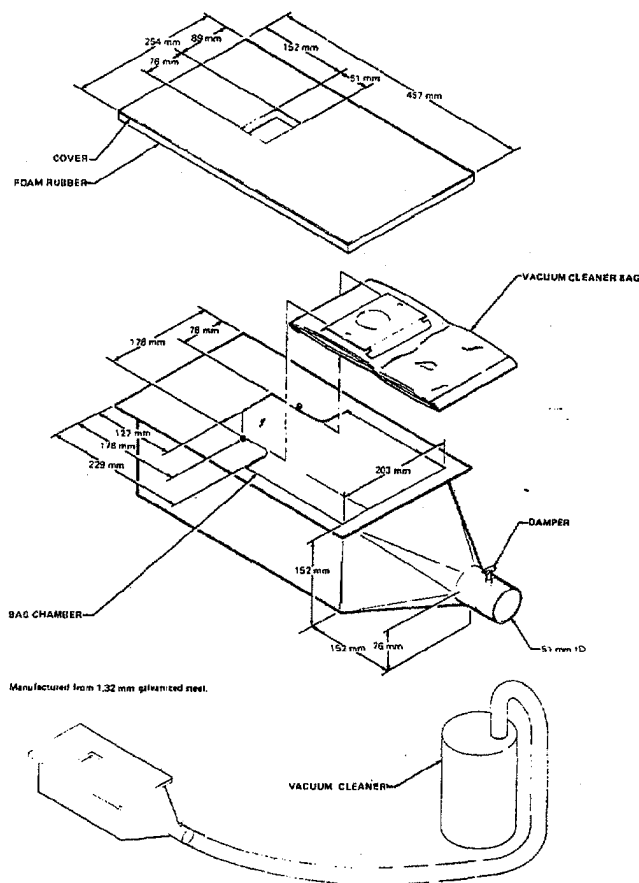


FIG. 5 Powder Flowmeter

timer. Rub the powder with the edge of the tongue depressor in 1 in. (25 mm) diameter circular strokes. Continue this motion with slight pressure until a solid gel is produced as found by lifting the depressor at least 2 in. (50 mm) from the hot plate. If the material produces a continuous filament from the plate to the depressor it has not gelled.

14.2.4 Stop the timer when the strand breaks readily. Repeat the test with a fresh specimen. Calculate and report the mean gel time.

15. Melting Point Determination

15.1 Apparatus:

15.1.1 *Kofler Hot Bench*,²⁰ hot bench agreed upon between the purchaser and the seller.

15.1.2 Calibrating Test Substances:

	Melting Point, °C
Azobenzene	68 ± 1
Naphthalene	80 ± 0.5
Benzoic acid	122 ± 1

15.1.3 *Paint Brush*, stiff bristle, 1/2-in. (12.7-mm).

15.2 Procedure:

15.2.1 Calibrate the apparatus as follows. Allow 60 min for the warm up of the heating bar. Sprinkle a calibrating substance having the closest melting point to that of the powder on the heating bar. Observe the sharp division between solid and liquid. Place the pointer between these two divisions. Slide the reading device to the melting point of the calibrating substance.

15.2.2 Sprinkle the specimen over the heating bar in a uniform manner. Observe the specimen after 1 to 2 min. Brush the material towards the lower temperature and note the location where particles of powder adhere to the bar when brushed lightly. Place the pointer at this point. Read the temperature and report in degrees Celsius.

NOTE 7—Powder will fuse and "set" on the hot bar. Remove the powder quickly and thoroughly before the setting occurs. Do not use abrasives to clean the bar.

16. Viscosity Measurement for Molten Powder Coatings by Weissenberg Rheogoniometer

16.1 *Summary of Method*—This method covers the determination of the viscosity of powder coatings at baking temperatures and consists simply of preheating the cone and plate of a Weissenberg rheogoniometer, pouring an adequate sample of powder onto the plate, closing the apparatus and recording revolutions per minute and the corresponding

²⁰ Kofler hot bench has been found suitable and is available from Reichert Co., Vienna, Austria. An equivalent may be used.

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²¹ Weis

Technical

steady torque transducer deflection.

16.2 *Apparatus*—Weissenberg rheogoniometer²¹ with at least capability for constant-speed rotational shear, high-temperature electric oven, and cone and plate shearing surfaces.

16.3 Procedure:

16.3.1 By consulting the rheogoniometer instruction manual, select the cone, plate, and torsion wire adequate to measure the largest anticipated viscosity in no more than 10 s. After alignment of the cone and plate, use the electric oven to bring them to the baking temperature of the coating (as agreed upon between the purchaser and the seller), or to a few degrees above that temperature.

16.3.2 As quickly as possible, with the air bearing functioning, open the oven, raise the cone a few inches, pour sufficient powder to fill the volume between the cone and the plate onto the center of the plate, and lower the cone sufficiently to close the oven. Continue to lower the cone until the proper gap between the cone and plate, as specified by the manufacturer of the rheogoniometer, has been reached.

16.3.3 Start the plate rotating at 1 to 5 rpm, with the torque transducer at the largest possible full-scale setting. **Caution**—Stop the plate rotation should the full-scale torque be exceeded since this may damage the instrument. Start again at a lower rpm. Record torque reading at this rpm. At several lower rotational speeds, record rpm, steady torque reading and the ratio of torque to rpm until the ratio of these two becomes essentially constant, indicating a low shear rate Newtonian (constant viscosity) behavior.

16.3.4 If lack of sensitivity prevents measurements at low shear rates, repeat the above procedure with a more sensitive torsion wire. If there is no low shear rate Newtonian region evident, the powder may be crosslinking or increasing in viscosity because of loss of volatiles. Repeated measurements on the same specimen at a given shear rate should indicate this. Several measurements made quickly at very low shear rates on fresh specimens may allow calculation of viscosities before crosslinking or volatile loss causes a significant change.

16.4 *Report*—The viscosity and shear rate values obtained, the value of the low shear rate Newtonian viscosity and the temperature at which the test was conducted.

16.5 *Precision*—Repeatability and reproducibility of viscosity readings at any given shear rate should be within 10 % relative. However, sample age and storage history may well affect the results obtained.

17. Inclined Plate Flow Test

17.1 Apparatus:

17.1.1 *Constant-Temperature Convection Electric Oven*, thermostatically controlled to maintain temperature of $150 \pm 2^\circ\text{C}$ or as agreed upon between the purchaser and the seller.

17.1.2 *Metal Plate Rack Assembly*, 8 by 11 in. (203 by 280 mm) that fits into inner oven chamber and is capable of being maintained in either a horizontal position or at a

65-deg angle by means of an exterior lever.

17.1.3 *Tin Plate Panel*, 8 by 11 in. (203 by 280 mm).

17.1.4 *Analytical Balance*, sensitive to 1.0 mg.

17.1.5 *Steel Pellet Mold Press*²² and knockout rod to make pellets 0.25 in. (6 mm) thick by 0.50-in. (12.7-mm) diameter.

17.1.6 *Stop Watch*.

17.1.7 *Steel Rule*.

17.2 Procedure:

17.2.1 Weigh out 0.50 g of coating powder and transfer to the steel pellet mold. Press to 0.25-in. (6-mm) thickness and push the pellet out of the mold with the knockout.

17.2.2 Place the pellet on a tin plate panel in a 1/2-in. circle scribed or marked on the panel.

17.2.3 Place the panel holding the pellet(s) on the metal rack assembly in the oven in a horizontal position. Close the oven door quickly. Allow the assembly to remain in the horizontal position for 3 min.

17.2.4 Tilt the rack assembly holding the panel to 65 deg from the horizontal without opening the oven door and allowing the oven to cool. Allow the rack and panel to remain in this position for 30 min.

17.2.5 After 30 min remove the panel from the oven and allow to cool to room temperature. Measure the amount of flow from the original diameter and line scribed on the panel.

17.3 *Report*—Maximum flow in millimetres from the original diameter.

NOTE 8—A control pellet if possible of known reactivity and flow should be run in each test. Sample material should be stored in sealed polyethylene bags prior to use and likewise pressed pellets should be stored in a desiccator or sealed bags to prevent moisture absorption.

NOTE 9—The 0.5-g charge should be sufficient for most powders with a cured specific gravity of 1.1 to 1.4. For heavier resins, it may be necessary to increase the pellet weight. If so, this should be recorded as a variance in specimen size.

18. Density and Specific Gravity

18.1 *Apparent Density*—Determine in accordance with Method A of Test Method D 1895.

18.2 *Bulk Factor*—Determine in accordance with Test Method D 1895.

18.3 *Specific Gravity*—Determine in accordance with Test Method D 153 or as follows.

18.3.1 In place of the pycnometer, a weight-per-gallon cup may be substituted. Because of its wide opening, the cup allows ready transfer of powder without loss. In addition the powder can be more readily wet with kerosine (or other suitable carrier agreed upon between the purchaser and the seller) by thorough use of a glass stirrer to remove the entrapped air. Care must be exercised to return any powder particles adhering to the glass rod to the weight per gallon cup to ensure accuracy of the method.

²² Parr Calorimeter Pellet Press, Cam and Cover type, stainless steel die with standard cavity for making 1/2-in. diameter pellets up to 1/2 in. thickness obtainable from Fisher Scientific Co., Pittsburgh, PA, has been found suitable. An equivalent may be used.

²¹ Weissenberg Rheogoniometer is available from Diano Corp., Midwest Technical Center, 1866 Production Dr, Louisville, KY 40299.

APPLICATION PROPERTIES

19. Determining the Relative Deposition Efficiency of Organic Polymeric Powders on Moving Targets

19.1 *Summary of Method*—The method essentially consists of spraying powder at a known flow rate on a number of similarly shaped targets moving at a known rate past a powder spraying device mounted in a spray booth.

19.2 Apparatus:

19.2.1 *Set of Targets*, consisting of 15 flat steel panels 6 by 36 by 0.063 in. (152 by 915 by 1.6 mm) wrapped in aluminum foil. (Use other targets as agreed upon between the purchaser and the seller.) The panels should have holes drilled at one end for hanging from a conveyor. The aluminum foil should be of the lowest possible temper and should be 13½ by 37 by 0.0015 in. (340 by 940 by 0.04 mm) (these characteristics have been found to be the best for easy wrapping of the panels). Other tempers, thicknesses, and dimensions may be used, however (for example, regular commercial aluminum foil is satisfactory).

19.2.2 *Fifteen Target Hangers* constructed so that the targets will hang from the conveyor in one plane within ±½ in. (±13 mm) with the flat surfaces perpendicular to the source of the powder spray. The target hangers should be long enough so the source of the powder spray can be pointed at the center of the targets.

19.2.3 *Powder Flowmeter*, consisting of a vacuum cleaner attached to a small vacuum chamber (powder receiver) constructed as shown in Fig. 5.

19.2.4 *Balance*, sensitive to 100 mg.

19.2.5 *Balance*, sensitive to 1 mg.

19.2.6 *Stop Watch*, accurate and readable to ⅓ s.

19.2.7 *Powder Spray System*.

19.3 Procedure:

19.3.1 Cut the number of aluminum-foil wrappings necessary for the desired number of tests and number (with a felt tip pen, grease pencil, etc.) on one corner. If the available balance can weigh the entire target, weigh the panels with the aluminum-foil wrappings. If the balance cannot weigh the entire target, weigh the aluminum-foil wrappings alone. Record the numbers and corresponding weights.

19.3.2 Wrap the targets with the foil. The best method of doing this is simply to lay the foil on a flat surface with the numbered side down. Place the target on the foil so that it is centered on the foil and ½ to 1 in. (13 to 25 mm) from the long edge of the foil. For convenience, the top of the target (where the hanger holes are) should be at the numbered end of the foil wrap. Fold the ½ to 1-in. (13 to 25-mm) piece of foil over the edge of the target, creasing it firmly. Then turn the panel over so that it wraps the foil around itself, and crease the new edge firmly. Then turn the panel over again (this will fold the remaining foil over) and crease it firmly.

19.3.3 Determine the conveyor speed by measuring the distance between two points on the conveyor track at least 10 ft (3.05 m) apart and by measuring, with the stop watch, the time it takes a location on the conveyor chain to travel the distance between the two points. If possible, adjust the conveyor speed to as close to 10 ft/min as possible (±2 %).

19.3.4 If possible, hang the 15 targets on the conveyor on 12 in. (305 mm) centers far enough ahead of the spray booth to allow the conveyor to come up to speed (at least 1 min

travel) or for this test, 10 ft (3.05 m).

19.3.5 Break up any powder agglomerates in the powder by mixing or sieving before adding to the powder hopper. Determine the powder flow by first weighing the vacuum cleaner bag with the balance and then installing it in the bag chamber. The vacuum cleaner bag alignment holes should go on the alignment pins on the bag chamber. Install the cover on the bag chamber (simply place on bag chamber). Start the vacuum cleaner and adjust the damper so that very little air is felt moving through the hole in the cover (vacuum in the vacuum bag should be 0.3 to 0.7 in. (8 to 17 mm) of water, vacuum in the flowmeter should be 1.5 to 2.5 in. (38 to 64 mm) of water).

19.3.6 Insert the powder spray device into the opening in the top of the powder flowmeter and turn on the dust collector. Turn on the powder spraying device for 30 or 60 s. (Caution—Be sure high voltage is turned off or, if that is not possible, at the lowest possible adjustment), measuring the time with the stop watch. Disassemble the powder flowmeter and reweigh the vacuum cleaner bag on the balance. The increase in weight divided by the time the powder was flowing is the rate of powder flow. The powder in the vacuum cleaner bag can be recovered simply by tearing the bag in half and dumping the powder in the booth or hopper.

19.3.7 Adjust the powder flow, if possible, to 300 g/min ± 5 % by successive process changes. Also adjust or record, or both, all the process variables possible including the following:

19.3.7.1 The powder and its state of use,

19.3.7.2 The particle size distribution if available,

19.3.7.3 The high-voltage power supply output potential and output current,

19.3.7.4 The resistor in the gun,

19.3.7.5 The potential of the powder gun's charging tip,

19.3.7.6 The distance between the target and the charging tip,

19.3.7.7 The pattern adjustment mechanism (measure such things as the relative locations of the parts),

19.3.7.8 The conveyor speed,

19.3.7.9 The powder flow,

19.3.7.10 The powder collector system air flow or some pressure which changes as a function of air flow only,

19.3.7.11 The powder transfer air flow and air pressure,

19.3.7.12 The powder quantity control pressure or whatever variable controls this,

19.3.7.13 For fluidized beds; air flow, air pressure, the amount of powder, and bed depth,

19.3.7.14 Other variables such as vortex air, dosing air, powder density air, pattern air, etc., and

19.3.7.15 The atmospheric variables: temperature, barometric pressure, and humidity.

19.3.8 Mount and level the powder spray device in the spray booth so that it is aimed at the centers of the targets as they pass. Position it so that its charging tip is 8 ± ½ in. (203 ± 13 mm) from the plane in which the targets will pass.

19.3.9 Turn on the powder flow, adjust the high voltage to 60 ± 1 kV (use other voltages as agreed upon between the purchaser and the seller), turn on the powder collection system, etc. Start the conveyor and spray the targets after all other systems seem to be operating normally and powder is spraying into the spray booth.

19.3.10 After remove the targets from the powder off of the targets in the booth. This is also an excellent method for the removal of powder retained after preweighed 5-Place this in the

19.3.11 After and pull the targets as soon as possible. This facilitates because the targets become handle. If necessary, after the foil wrapping.

19.3.12 Weighings from the each end of because they relative to the powder added powder and powder was wiped off to

19.3.13 Calculate the following equation: % E

where:
% E = percent
 $\bar{W}_p$ = mean
 V_c = coefficient
 D_T = distance
 Q_p = powder
% R = percent (1)

19.3.14 coating, calculate following equation:

where:
 V = volume
 $\bar{W}_p$ = weight
 n = number
 $\bar{W}_p$ = mean

19.3.14.1 indicates that This is due to the fact that, sometimes powder collection results agree

19.3.10 After spraying the targets, stop the conveyor and remove the targets, taking care not to knock or jar the powder off of them. If it is desired to cure the powder, place the targets in an oven for the time and temperature recommended for the specific powder being used. If necessary, this is also an excellent time to determine the weight percent retained after curing the powder. Weigh 1 g of powder in a preweighed 5-in. (127-mm) diameter aluminum foil "pan." Place this in the oven along with the targets.

19.3.11 After curing, remove the targets from the oven and pull the aluminum foil down 1 to 2 ft (305 to 610 mm) as soon as possible using asbestos or thick workman's gloves. This facilitates the final removal of the aluminum foil because the coated foil tends to contract slightly with cooling and become very difficult to remove when cool enough to handle. If necessary, also remove the pan of powder from the oven. After they are reasonably cool, remove the aluminum-foil wrappings, if they are to be weighed alone.

19.3.12 Weigh the center 11 targets or aluminum wrappings from the center 11 targets excluding the two targets on each end of the target set. (The end targets are excluded because they tend to collect excessive amounts of powder relative to the other targets.) Determine the amount of powder added to each target. If necessary, weigh the pan of powder and determine the weight percent retained. If the powder was not cured, the targets may be blown off and then wiped off to prepare them for the next test.

19.3.13 Calculate the mean weight of powder deposited. Calculate the percent deposition efficiency using the following equation:

$$\% E = (\bar{W}_p \times V_c \times 10\,000) / (D_T \times Q_p \times \% R)$$

where:

$\% E$ = percent deposition efficiency,

$\bar{W}_p$ = mean powder deposition, g,

V_c = conveyor speed cm/min,

D_T = distance between target centers, cm,

Q_p = powder flow, g/min, and

$\% R$ = percent by weight powder retained after curing (100 % if not cured).

19.3.14 To determine the variation in application of the coating, calculate the coefficient of variation using the following equation:

$$V = \frac{(\sum W_p^2 - n \bar{W}_p^2)^{1/2}}{\bar{W}_p}$$

where:

V = coefficient of variation %,

$\bar{W}_p$ = weight of powder on a foil wrapping, g,

n = number of targets used (11), and

$\bar{W}_p$ = mean powder deposition, g.

19.3.14.1 If the coefficient of variation exceeds 10 % it indicates that the powder cloud is fluctuating excessively. This is due usually to poor (slugging) powder pump operation, sometimes to unstable booth air flow profiles, and sometimes to unstable air flow patterns created by spray gun powder cloud control system.

19.3.15 Repeat the above procedure until two consecutive results agree within ± 10 %. Use the set of conditions under

which these results were obtained as a standard to which to relate future work. It is wise to run the test within the standard conditions at the beginning and end of each day. If results do not agree within ± 10 % the day's work should be suspect. Results will vary from day to day and even during the day due to variables beyond the user's control. Long-term variations can even be as large as 50 %. Tests run with different powders or under different conditions should have the results reported in terms of the standard test results. For example, if the efficiency results under the standard conditions were 50 % with one powder and 75 % with another powder, the second powder would be 50 % more efficient than the first powder.

19.4 Report:

19.4.1 Report the variables and their values as recorded for 19.3.7. Report the percent deposition efficiency and its coefficient of variation, the percent efficiency compared to the average percent efficiency obtained using the standard conditions on the day of the test.

19.5 *Precision*—Deposition efficiency results should be repeatable within ± 10 % during a day.

PHYSICAL PROPERTIES OF POWDER COATING

20. Panel Preparation

20.1 *Treatment of the Substrate*—Clean and prepare test panels in accordance with one of the following standard test methods or recommended practices or as agreed upon between the purchaser and the seller:

20.1.1 Method D 609.

20.1.2 Practices D 1730.

20.1.3 Practices D 1731.

20.1.4 Practices D 1732.

20.1.5 Method D 1733.

20.1.6 Practices D 2092.

20.1.7 Test Method D 2201.

20.2 *Priming and Sealing*—In many instances, the use of a primer, primer surfacer or sealer is required. The type, application, and treatment of any undercoat system should be agreed upon between the purchaser and the seller.

20.3 *Application of Powder Coatings*—The coatings may be applied by fluidized bed, electrostatic spray, or other methods.

20.4 Curing of Powder Coatings:

20.4.1 Fuse or bake the powder coating to a uniform film according to the established schedule and temperature and age as agreed upon between the purchaser and the seller before running tests.

20.4.2 The powder coating should be overbaked to determine the time/temperature effect on the physical and chemical properties in accordance with Recommended Practice D 2454.

20.5 *Measurement of Film Thickness*—Since the properties of a powder coating can vary considerably with its thickness, it is important to know the film thickness. Measure the film thickness in accordance with Test Method D 1005, D 1186, or D 1400.

21. Abrasion Resistance

21.1 Determine the abrasion resistance in accordance with Test Method D 658, D 968, or D 1044.

22. Adhesion

22.1 The powder coatings of a specified thickness and over a specified substrate as agreed upon between the purchaser and the seller is subjected to an adhesion test to determine the degree of attachment the coating has to the substrate.

22.2 Determine the adhesion of the powder coating to the specified substrate in accordance with Test Methods D 2197 or D 3359.

23. Chemical Resistance

23.1 Coating systems frequently come into contact with various chemicals which may have an effect on the properties of the system. Failure, when it occurs, is usually in the form of discoloration, change in gloss, blistering, softening, swelling, or loss of adhesion.

23.2 *Household Chemical Resistance*—Determine the effect of chemicals in accordance with Test Method D 1308.

23.3 *Detergent Resistance*—Determine the resistance to failure when immersed in a detergent solution in accordance with Practice D 2248.

24. Chip Resistance

24.1 In many end uses, the ability of a powder coating to withstand sudden impact from stones, gravel, etc., without being loosened from the substrate is important.

24.2 Determine chip resistance in accordance with Test Method D 3170.

25. Color—Pigmented Coatings

25.1 The colors of opaque objects such as coated surfaces may be specified by visual or instrumental requirements.

25.2 For visual evaluation, determine the color of a coated surface in accordance with Method D 1535.

25.3 Determine the color of a coated surface instrumentally in accordance with Method E 308.

26. Color Difference—Pigmented Coatings

26.1 The color difference between two homogeneously colored opaque films may be determined by visual evaluation or by instrumental means.

26.2 Determine color differences visually in accordance with Practice D 1729 or instrumentally by Method D 2244.

27. Cracking Resistance

27.1 The cracking resistance test is designed to give an indication of the resistance of a coating system to cracking and checking caused by temperature and humidity changes. Some factors which may affect results are type of substrate, substrate thickness, undercoat, topcoat, and film thickness.

27.2 Determine cracking resistance in accordance with Test Method D 2246.

28. Elongation

28.1 An elongation test may give an indication of the flexibility of a powder coating. It can also show whether there is any change in flexibility due to the aging of the film.

28.2 Determine elongation in accordance with Test Method D 522 or D 1737.

29. Filiform Corrosion Resistance

29.1 Filiform corrosion is a type of corrosion that occurs under coatings on metal substrates and is characterized by a definite thread-like structure and directional growth.

29.2 Determine the susceptibility of a powder coating on a metal substrate to this type of corrosion by Test Method D 2803.

30. Gloss

30.1 Determine the gloss of a powder coating in accordance with Test Method D 523.

31. Hardness

31.1 Determine the hardness of powder coating in accordance with Test Method D 1474 using either Method A (Knoop Indentation Hardness) or Method B (Pfund Indentation Hardness).

31.2 Other methods of determining hardness are widely used in some industries and may be used as agreed upon between the purchaser and the seller.

32. Impact Resistance

32.1 Films formed from powder coatings may be subject to sudden impact in certain end uses. Determine impact resistance in accordance with Test Method D 2794.

33. Outdoor Exposures

33.1 While the accelerated tests given elsewhere in this recommended practice are intended to enable prediction of probable performance, actual outdoor exposures should be made on coatings intended for exterior use. Usage of paint systems is so varied that no one set of conditions (length or place of exposure) can be given in this practice to cover all situations. These conditions as well as the type of substrate, substrate preparation, etc., should be agreed upon between the purchaser and the seller. However, it is suggested that unless otherwise agreed upon, panels for outdoor exposure should be prepared in accordance with Section 19 of this practice.

33.2 Many properties of powder coating films should be evaluated periodically throughout the outdoor exposure period. These properties may be evaluated as follows:

33.2.1 *Blistering*—Test Method D 714.

33.2.2 *Chalking*—Test Method D 659.

33.2.3 *Checking*—Method D 660.

33.2.4 *Cracking*—Test Method D 661.

33.2.5 *Rusting*—Method D 610.

33.2.6 *Erosion*—Test Method D 662.

33.2.7 *Flaking*—Test Method D 772.

34. Print Resistance

34.1 A print test may be used to determine the degree of thermoplasticity or solvent retention of a film and hence whether the product can be safely stacked or packaged and, in the case of a thermoplastic film, at what temperature the film prints or mars. The print test can also determine the degree of marring due to pressure.

34.2 Determine the imprinting and thermoplasticity of a powder coating film in accordance with Test Method D 2091.

35. Salt Spray

35.1 Salt spray determining the conditions of the accelerated atmosphere, pH, composition, physical parameters of the substrate, the coating is scribed within the cabinets and panels and the upon between.

35.2 Test in Method B 117

36. Water Resistance

36.1 Test in determining the high humidity usually evident adhesion which of the ab

36.2 Determine of high humidity

36.3 Determine of water immersion test is best soaked in water

37. Safety and Health

37.1 This aging, shipping and application decorative volumes of paint

37.2 When chemical aspects: Administrative

37.3 Packaging 37.3.1 Containers

plastic-lined

37.3.2 Labeled as to manufacturer

37.4 Shipping 37.4.1 Packing

manner to be

37.4.2 Bundled in a manner Precautions of the powder

37.5 Storage

35. Salt Spray Resistance

35.1 Salt spray testing of coatings may be helpful in determining their resistance to failure in service under conditions of high humidity and salt concentrations. Under the accelerated conditions of the laboratory test the temperature, pH, concentration of the salt solution and other physical parameters can be controlled. The selection of the substrate, the coating system, the manner in which the coating is scribed, the location or position of the panels within the cabinet, the length of the test, the inspection of panels and the method of reporting results must be agreed upon between the purchaser and the seller.

35.2 Test for salt spray resistance in accordance with Method B 117.

36. Water Resistance

36.1 Testing of coating systems with water is helpful in determining their resistance to failure under conditions of high humidity or water immersion. Failure in water tests is usually evidenced by blistering, dulling, softening, or loss of adhesion which does not disappear or recover upon evaporation of the absorbed water.

36.2 Determine the resistance to failure under conditions of high humidity in accordance with Practice D 1735.

36.3 Determine the resistance to failure under conditions of water immersion in accordance with Practice D 870. This test is best suited for coating systems that will actually be soaked in water during service.

37. Safety and Handling Precautions

37.1 This recommended practice covers the safe packaging, shipping, receiving, storage, and handling during use and application of organic powders used for protective and decorative coatings. Procedures for handling large and small volumes of powder are included.

37.2 Where required, attention shall be directed to ecological aspects and pertinent Occupational Safety and Health Administration (OSHA) regulations.

37.3 Packaging:

37.3.1 *Container*—For small and medium operations, containers designed to give moisture protection, such as plastic-lined boxes or drums, should be used.

37.3.2 *Labeling*—All containers should be clearly identified as to type material, color, batch number, and date of manufacture.

37.4 Shipping and Receiving:

37.4.1 *Palletizing*—Containers should be palletized in a manner to avoid crushing or packing.

37.4.2 *Bulk Shipment*—Large shipments should be handled in a manner agreeable to the purchaser and the seller. Precautions should be taken to avoid hard packing or caking of the powder.

37.5 Storage:

37.5.1 All containers must be sealed when not in use.

37.5.2 Temperature and relative humidity during storage should not exceed 80°F (27°C) and 50 %, respectively, or as specified on the label.

37.6 Precautions in Handling and Application:

37.6.1 All metal equipment must be properly grounded.

37.6.2 The fluid bed must be cleaned periodically to ensure proper operation.

37.6.3 Under certain conditions organic powders may be subject to dust explosions. Trade association bulletins such as the National Fire Protection Association Bulletins No. 33, Spray Finishing and No. 654, Dust Explosion Prevention—Plastics Industry, may be helpful in identifying conditions and minimizing their occurrences.

37.6.4 Spraying:

37.6.4.1 Spray booths, dust collectors, and ducts should be built with smooth surfaces to facilitate cleaning. If possible, surfaces should be of a material to which the powder will not stick.

37.6.4.2 During nonoperating periods all equipment should be flushed with air or the manufacturer's recommended procedure should be followed.

37.6.5 *Blending*—If reclaimed or recycled powder is to be blended with virgin material, a suitable device for sifting, drying, and blending should be used.

37.6.6 *Dust Collection and Ventilation*—All handling and application equipment should be fitted with a suitable dust collector and filter system.

37.6.7 Suitable electrical grounding is required. Precautions are necessary to ensure ratio of powder to air are held below or above the explosive mixture.

37.7 Ecology:

37.7.1 Exhaust stacks should be equipped with dust collectors or absorbers or both to avoid air pollution.

37.7.2 *Waste Disposal*—Powder must be disposed of in the manner governed by local laws and regulations.

37.8 OSHA Requirements:

37.8.1 *Operator Safety*—All personnel should be equipped with suitable air masks, gloves, and any other specified devices for personal protection.

38. Data Report

38.1 The report shall include the following:

38.1.1 Complete identification of the powder coating material tested,

38.1.2 Description of specimens, including numbers, preparation method, and thickness of coating,

38.1.3 Temperature of test if other than specified by test method,

38.1.4 Type and manufacture of test equipment,

38.1.5 All specific information called for on the individual test method report,

38.1.6 Test results as calculated or observed values on the basis of data, median or mean value, and

38.1.7 Date of test.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Determining by Exterior Exposure Tests the Susceptibility of Paint Films to Microbiological Attack¹

This standard is issued under the fixed designation D 3456; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This practice provides guidelines for determining the susceptibility of paint films to microbiological attack on exterior exposure. While it is recognized that various organisms may occur on an exposed coating, the specific types of organisms are mainly of academic interest. The degree to which microbiological discoloration occurs is the primary concern.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood²
- D 1849 Test Method for Package Stability of Paint²
- D 3274 Test Method of Evaluating Degree of Surface Disfigurement of Paint Films by Microbial (Fungal or Algal) Growth or Soil and Dirt Accumulation²

3. Summary of Practice

3.1 Simple observation of a coated object subjected to exterior exposure is considered a practical and reliable method for determining the degree that microorganisms discolor the coating. However, this applies to a specific coated object exposed under a given set of conditions. It should be recognized that there are critical factors that influence the amount of fungal growth that may occur on the same coated object when exposed to other conditions. These factors include the geographic location, local atmospheric conditions such as the dust and pollen content of the air, angle of exposure, degree to which the coating is subjected to weathering, effects of moisture and sunlight, the substrates on which the coating is applied, and the coatings in the paint system under test. The latter factor includes the stability of the coating while packaged in the container, as well as the composition of the coatings included in the total system and

the thickness of each coating applied. Thus, while microorganisms occur on the surface of the last film applied, the degree of microbiological growth that will occur is also influenced by the composition of the undercoats. All the above factors should be considered in the selection of a coating resistant to discoloration by microorganisms.

4. Significance and Use

4.1 The growth of fungi and algae in and on the surface of paint films represents a major cause of discoloration or disfigurement of painted surfaces. This practice covers the preparation of coatings for testing, their application on substrates, and the arrangement of the coated panels on exterior test fences to determine the degree of microbiological attack that may occur on the surface of the coatings over a period of time. This practice is intended to provide guidelines for, and a discussion of, the various factors critical in selection of exterior coatings resistant to discoloration or disfigurement by algae and fungi.

5. Preparation and Application of Coatings

5.1 *Conditioning of Coatings Prior to Application*—Individual coatings to be used in the paint system should be properly aged under suitable conditions prior to testing. Hydrolysis, amalgamation, absorption, and other physical and chemical changes that may have a profound influence on the resistance of a coating to microorganisms usually increase with increasing temperature. It is recognized that actual storage periods of paints prior to use may vary from one to several years, and the peak temperature encountered may be as warm as 160°F (70°C). However, a recommended conditioning period consists of 1 year at room temperatures or 1 month at 125°F (50°C) as in Test Method D 1849. The conditioning of coatings prior to testing shall be agreeable to the producer and the user in the case of a referee test.

5.2 *Preparation of Coatings for Application*—Prior to application of the various coatings to be included in the total paint system, thoroughly reconstitute each coating by appropriate mixing or shaking. At the time of application, there must be no settling, incompatibility, or other stability problem observable in the coating in the container.

5.3 *Application of Paint System*—Apply each coating in the total paint system in an appropriate manner to provide a specified and reasonably uniform film thickness. The presence and thickness of different coatings in the complete system can have a pronounced effect on the degree of microbiological discoloration that will occur. Thus, each

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¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint, and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.28 on Biodeterioration.

Current edition approved March 27, 1986. Published May 1986. Originally published as D 3456 - 75. Last previous edition D 3456 - 75 (1981)^{ε1}

² Annual Book of ASTM Standards, Vol 06.01.

paint in the system must be applied as recommended by the manufacturer. It is important that the procedure and the user agree on the type of coatings and the spreading rate of each coating in the final test film. The drying time between coats and the curing time of the total system prior to exposure should also be specified. Recommended practice for house paint is 2 days between coats and no less than 7 days and not longer than 1 month prior to the exterior exposure. Industrial and industrial maintenance coatings may require a different practice that should be acceptable to the producer and the user.

5.4. On each substrate use a test area not less than 48 in.² (310 cm²) in size. When only one type of substrate is being used, expose at least duplicate panels. When more than one substrate is employed for each exposure condition, duplicate panels of each substrate are not usually required. In case of a referee test, it is recommended that replication and size of test area be agreed on between the producer and the user. Common practice in the industry is to use 6 in. (nominal) by 36-in. (152 by 915-mm) panels for house paint exposures and to use 12-in. (305-mm) metal panels of various widths for exposures of industrial and industrial maintenance coatings.

6. Substrates for Testing

6.1 *General Considerations*—The substrate on which a paint system is exposed can have a significant effect on the degree of microbiological discoloration that may occur. Coated wood surfaces generally support more mold and algae than do coatings on metals or masonry surfaces. This is perhaps due to some nutrients and greater porosity for retention of available moisture in wood surfaces. The type of metal substrate can have either an adverse or beneficial influence on the growth of fungi and algae. Some metals can inactivate certain microbiocides, thereby allowing greater microbiological discoloration. It should also be recognized that metals may also reduce the growth of microorganisms because of toxic compounds resulting from weathering of the metal. Masonry surfaces generally inhibit microbiological growth because of their alkaline nature. However, this same characteristic can contribute to microbiological discoloration by hydrolyzing alkali-sensitive microbiocides that may have been used in the coating. The recommended substrates for testing coatings for resistance to discoloration by microorganisms vary according to the intended use of the coatings. In referee cases, the substrate for testing shall be mutually agreeable to the producer and the user. Industrial coatings should be evaluated on the surface for which they are designed. Trade sales and industrial maintenance coatings are general-purpose coatings and should perform on a variety of substrates. For such coatings, test exposures on the following substrates are recommended for the indicated reasons.

6.2 *Wood Substrates*—Sapwood of pine and fir generally is considered conducive to growth of microorganisms. This may be due to nutrients in the wood and to the low dimensional stability, resulting in microcracking of coatings applied on the wood with subsequent mold growth in these cracks. Plywood, hardboard, and other wood-derived products support varying degrees of fungal growth depending on the nutrient value, degree of moisture absorption, and

dimensional stability of the base material. Redwood tends to have better dimensional stability and otherwise has insignificant effect on the microbiological growth on coatings applied over it. Cedar lumber generally contains compounds that aid in resisting microbiological growth. Both cedar and redwood contain colored extractives that can bleed through coatings to discolor the surface. Some of these extractives can also be nutrients that contribute to microbiological growth, resulting in added discoloration.

6.3 *Metal Substrates*—Iron, galvanized steel, and aluminum are common substrates for paints. Iron and zinc compounds generally inhibit microbiological growth. On the other hand, these metals may react with certain microbiocides to reduce the microbiological inhibition. Certain microbiocides can also cause discolored corrosion products or loss of adhesion by the coating on these surfaces. Aluminum is rather chemically inert and does not itself promote microbiological growth. It may, however, cause loss of microbiological resistance of coatings containing certain mercury compounds because of the amalgamation reaction by aluminum and mercury. This can result in loss of adhesion.

6.4 *Masonry Substrates*—The extremes of masonry surfaces generally consist of two conditions: fresh surfaces, which are relatively alkaline and free of fungi and algae, and weathered surfaces that are less alkaline and may be discolored because of microbiological growth. Weathered masonry surfaces represent useful test surfaces since microbiological contamination can grow through inadequately preserved coatings from the underside. Weathered masonry surfaces also offer a relatively uniform surface from panel to panel. Such uniformity is useful in statistically determining the relative effectiveness of various coatings or of various wash solutions for cleaning or "sterilizing" a surface before repainting. Exposures of coatings on both clean masonry panels and weathered panels can provide useful results and both are recommended as test substrates.

6.5 *Moldy "Mildewed" Repaint Surfaces*—Weathered paint films that are discolored by microbiological growth are also useful in determining the efficacy of wash solutions to clean or "sterilize" an old paint film prior to repainting. Such moldy repaint surfaces also are useful in determining the resistance of a coating system to discoloration due to microorganisms growing through the paint system from the underside.

7. Arrangement of Coated Panels on Exterior Test Fences

7.1 Paint systems should offer long-term resistance to microbiological growth both in completely exposed areas and in shady, protected areas. Where sunlight and moisture are in abundance, chalking will occur. However, chalking is slow to occur in shady areas. Thus, an effective microbiocide for a coating must have long-term light stability, heat resistance, and be sufficiently soluble and toxic to cause microbiological inhibition, and it must have limited solubility or leachability from the coating system such that it remains in the system for sufficiently long periods of time. Ideally, it should not induce chalking for cleaning of the surface, since such chalking will cause fading or tinted paints.

7.2 The most realistic conditions of exposure can best be realized on the exterior of houses. Unfortunately, buildings

with the various substrates and located in desirable localities and geographic locations are seldom available. However, comparisons of various coating systems can be made by exposing coated panels on exterior test fences. Positions of exposure should vary to include completely exposed weathering conditions and also protection from weathering. The exposed conditions are useful in determining the influence of temperature, sunlight, moisture, various substrates, and subsequent chalking on a coating. The protected, shady exposures are useful in determining the relative microbiological inhibition of coatings in the absence of chalking.

7.3 Construction of test fences for *protected or shady conditions* can be similar to that given in Practice D 1006 but modified to provide for a larger test area in the protection of an eave. A protected area under an eave facing north best represents the desired conditions. This eave should have a minimum of 18 in. (455 mm) overhang. The panels should be exposed in a lapped position as would be encountered with wood siding on a house. The test area for each system should begin immediately under the eave and continue down the test fence to a point at least 2 ft (610 mm) below an imaginary horizontal line, which is derived by projecting at a 45° angle from the outer edge of the eave, downward and inward to the test panels. Thus, an 18-in. eave would require a minimum of 3.5 ft (1.06 m) of test panels, beginning immediately under the eave down to the bottom edge of the exposure area. The inverted horizontal surface of the soffit under the northerly eave is an ideal test environment as well. Also, this area does not usually collect excessive dirt. Thus, any microbiological discoloration is readily apparent.

7.4 Construction of test fences for *exposed conditions* can be similar to that in Practice D 1006 but modified to provide for offset panels held at an angle of 5° (±5°) off vertical facing south. Construction should allow exposures such that moisture falling on the test area of one panel will not drip on the next panels below.

8. Geographic Location of Test Fences

8.1 The climatic and environmental conditions of the test fences should be similar to those of the areas in which the

paint system is intended for use. For coatings to be used nationally, it is desirable to expose in warm and humid environments. Exposures should be made in both shady conditions and in direct sunlight. It should be recognized that the microflora occurring in different parts of a country will vary so that mold and algae encountered in one test location may not necessarily be those of another.

9. Periods of Exposure

9.1 The time of the year when the initial exposure is first made can be critical. Because of differences in weather from year to year, results of exposures from one year to another may also vary. In order to compare the tendency for two or more paint systems to become discolored through microbiological growth, it is advisable that the paint systems be exposed at the same time. It is frequently desirable to include both a positive and a negative control. The negative (fail) control is a paint system known to discolor quite severely under standard exposure conditions and may be used on each panel. A positive (pass) control is a paint system that will perform reasonably well in the same known exposure conditions.

10. Ratings for Microbiological Discoloration

10.1 Ratings should be made in accordance with Test Method D 3274.

10.2 It is desirable to obtain mold ratings prior to initiation of chalking and erosion, since it is prior to this occurrence that a paint film is most prone to discoloration by spore-cluster type (dirt-like) mold growth. Chalking can occur within several months, or it may never occur, depending on the paint formulation and its exposure to weathering conditions. Therefore, mold ratings are suggested after 3, 6, 9, 12, 18, and 24 months and yearly thereafter. Make yearly inspections immediately following seasonal growing conditions. Most extensive microbiological growth usually develops during periods of warm and humid weather. Thus, make any ratings of microbiological discoloration immediately following the warm, humid period of the year.

11. Keywords

11.1 exterior, exposure, microbiological paint films

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1. Scope

1.1 This standard covers the method for determining the relative microbiological inhibition of coatings in the absence of chalking.

1.2 This standard is applicable to the evaluation of paint systems under standard exposure conditions and may be used on each panel.

1.3 This standard is applicable to the evaluation of paint systems that will perform reasonably well in the same known exposure conditions.

1.4 This standard is applicable to the evaluation of paint systems that will perform reasonably well in the same known exposure conditions.

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3.1 This standard is applicable to the evaluation of paint systems under standard exposure conditions and may be used on each panel.

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Standard Test Method for Humid-Dry Cycling for Coatings on Wood and Wood Products¹

This standard is issued under the fixed designation D 3459; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the evaluation of coatings designed for use on interior wood and wood products substrates by exposure alternately to low and high humidity at an elevated temperature.

1.2 This test method is applicable to any coated material or product that is affected either entirely or partly by changes in atmospheric relative humidity.

1.3 This test method applies only to those coatings applied in sufficient quantity to form a continuous film.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D333 Test Methods for Clear and Pigmented Lacquers²
- D714 Test Method for Evaluating Degree of Blistering of Paints²
- D1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D2571 Guide for Testing Wood Furniture Lacquers²
- D2691 Test Methods for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products²
- E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens³

3. Significance and Use

3.1 Wood substrates and the coatings applied to them expand and contract to different degrees as the humidity changes, causing stresses that may produce checks, cracks, splits, blisters, swelling, loss of adhesion, and various changes in surface appearance. This test method is intended for use where the coating is applied in sufficient quantity to form a continuous film on the wood or wood product substrate. It is not possible to make any direct correlation between the

results obtained from humid/dry cycling and the results expected in a specific period of time in service because of variations resulting from geographic locations, location within the building, care of the panel (such as waxing), and variations within materials involved.

4. Test Panels and Panel Preparation

4.1 Test panels shall be regular production finished panels if available.

4.2 If regular production finished panels are not available the purchaser and the seller shall agree upon the following variables: type of wood or wood substrate, sanding method(s) and paper(s), sealer(s) or toner(s), filler(s), stain, primer or basecoat, print(s), and topcoat(s), methods of application, and the number of surfaces to be coated. If rotary-cut veneered panels are used, it should also be agreed whether the veneer is to be open or close faced.

5. Apparatus

5.1 *Oven*—a gravity-convection or forced-ventilation electrically heated oven meeting the requirements of Specification E 145 and providing a continuous temperature of $122 \pm 3.5^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$).

5.2 *Constant Elevated-Temperature and Constant-Humidity Chamber*, maintained at a relative humidity of $97 \pm 2\%$ and a temperature of $122 \pm 3.5^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$).

5.3 *Constant-Temperature and Constant-Humidity Chamber*, maintained at a relative humidity of $50 \pm 5\%$ and a temperature of $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$).

6. Test Specimens

6.1 Test specimens shall be large enough to be representative of the material or product and to permit easy observation of possible defects. This usually means a minimum area of approximately 12 by 12 in. (300 by 300 mm).

6.2 Unexposed control specimens of each coating or coating system or product tested shall be held in reserve in a constant-temperature and constant-humidity area (5.3) to prevent any changes.

6.3 Whenever possible, comparative control specimens of a similar material or product with known service characteristics should also be exposed.

6.4 Three specimens are usually adequate unless a lack of uniformity is suspected in the substrate, coating, or coating system.

7. Conditioning

7.1 Place the specimens in a conditioned room or chamber (5.3) so that air is free to circulate on all sides of

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 3459 - 75. Last previous edition D 3459 - 75 (1981)⁴.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.02.

each specimen so as to avoid localized overheating. Allow them to remain there 14 days.

8. Procedure

8.1 Measure the dry film thickness of the coating system on the control panel to the nearest 0.1 mil (2.5 μm) by an appropriate method such as Test Methods D 1005, Methods D 2691, or an Optical Surfer Analyzer.⁴ The results of humid dry cycling are directly affected by the dry film thickness as well as by the coating system itself.

8.2 After conditioning measure thickness of each specimen to the nearest mil (25 μm) at a marked location about 1 in. (25 mm) from one edge and midway along one side at the beginning and after each change of conditions.

8.3 Place the specimens in the oven maintained at $120 \pm 3.5^\circ\text{F}$ ($50 \pm 2^\circ\text{C}$) (5.1) so that air is free to circulate on all sides of each specimen to prevent localized overheating. After 48 h remove the specimens from the oven and place them in the high-humidity elevated-temperature chamber (5.2) so that air is free to circulate on all sides of each specimen. Leave the specimens in the humidity cabinet 48 h. The period of 48 h of dry heat followed by 48 h of humid heat constitutes one cycle. Expose the specimens to the number of cycles as agreed upon between the purchaser and the seller.

8.4 At each change of conditions during cycling, visually inspect each specimen under a strong light and at various

angles to the light for possible damage or change. Note any change in or damage to each specimen. Damage may be found in the base material (plywood, hardboard, flakeboard, solid wood, etc.) or in the coating, in a finish or overlay on the substrate material. Some defects that may occur as a result of humid/dry cycling are as follows: checks, cracks, splits, blisters, (see Test Method D 714) raised grain, localized lumps or swelling, photographing of substrate through the coating, loss of particle adhesion, loss of adhesion in glue lines, change in the dimensions of the overlay, changes in surface color, gloss, or hardness, cohesion loss in the coating. If open-faced rotary-cut veneer has been used, checking may result from this fact.

9. Report

9.1 Report the following information:

9.1.1 Complete description of the test material and the comparative control material,

9.1.2 Size and number of the specimens,

9.1.3 Dry film thickness of the material under test and the control (comparison material).

9.1.4 Number of cycles agreed upon between the purchaser and the seller,

9.1.5 Any deviations from the standard procedure, and

9.1.6 Defects or changes as described under 8.4.

10. Precision

10.1 Since the observed changes can take several forms no numerical values have been developed, and meaningful estimates of precision cannot be given. Round-robin tests showed that cooperators can obtain satisfactory agreement in ranking of results.

⁴ Gardner, H. A. and Sward G. G., *Paint Testing Manual*, 13th edition, ASTM STP 500, ASTM, 1972.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

1. Scope

1.1 This test method covers the determination of the rate of evaporation of Shell Th applied varnish, carbonates, and rate of use.

Method A
Method B

1.3 This test method is used to determine the volume of the dispensed syringe. 1.4 This test method is used to determine the address of the res applied applic hazard

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Standard Test Methods for Evaporation Rates of Volatile Liquids by Shell Thin-Film Evaporometer¹

This standard is issued under the fixed designation D 3539; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These test methods cover the determination of the rate of evaporation of volatile liquids of low viscosity using the Shell Thin-Film Evaporometer. The test methods have been applied to a wide range of volatile liquids, including paint, varnish, and lacquer solvents and thinners to various hydrocarbons and to insecticide spray-base oils.

1.2 The test methods for the determination of evaporation rate using the thin-film evaporometer are:

	Sections
Method A ^{2,3} —Manual Recording	5 to 11
Method B—Automatic Recording	12 to 17

1.3 The test methods are limited only by the viscosity of the volatile liquid which must be sufficiently low to permit the dispensing of an accurately measured specimen from a syringe.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Notes 1 and 2.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 891 Test Methods for Specific Gravity of Liquid Industrial Chemicals⁴
E 1 Specification for ASTM Thermometers⁵

3. Summary of Method

3.1 A known volume of liquid is spread on a known area of filter paper that is suspended from a sensitive balance in a

cabinet. Dried air or nitrogen at 25°C is passed through the cabinet at a known rate. The loss of weight of the filter paper/liquid is determined and plotted against time.

4. Significance and Use

4.1 The rate of evaporation of volatile liquids from a solution or dispersion is important because it affects the rate of deposition of a film and flow during deposition, and thereby controls the structure and appearance of the film. In the formulation of paints and related products, solvents are chosen based on the evaporation characteristics appropriate to the application technique and the curing temperature.

METHOD A—EVAPORATION RATE USING THE MANUAL THIN-FILM EVAPOROMETER

5. Apparatus

5.1 *Evaporometer*, thin-film evaporometer⁶ as shown in Fig. 1.

5.2 *Constant-Temperature Cabinet* for evaporometer.

NOTE 1—*Precaution:* In instances with the solvents and other volatile materials normally tested using this apparatus and under the conditions specified in this method, the concentration of solvent or other flammable material being exhausted into the laboratory atmosphere will be significantly below any concentration that could be hazardous, that is, a lower flammable limit. However, it may be desirable to locate the instrument and cabinet in a laboratory exhaust hood if the routine handling of certain materials may present a hazard due to toxicity, extreme volatility, or flammability.

5.3 *Interval Timer: Stop Watch or Electric Timer*—A timer that gives an audible signal at 10 or 20-s intervals and that gives a warning signal approximately 3 s before the end of the interval is preferred.

5.4 *Filter Paper Disk*—Fast, open-texture filter paper, 90 mm in diameter, with a circle approximately 60 mm in diameter (and concentric with the edge) lightly drawn on the paper with a pencil.

5.5 *Syringe*—A 1.00-mL hypodermic syringe equipped with a 9-in. (225-mm) needle of 0.050-in. (1.3-mm) outside diameter stainless steel tubing.⁷ Due to manufacturing variations, the syringe should be calibrated before use.

5.6 *Dehumidification Equipment*—A suggested setup is given in a schematic diagram, Fig. 2.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 3539 - 76. Last previous edition D 3539 - 76.

² These test methods are essentially the same as the one developed by the New York Society for Paint Technology. The Precision section was added by ASTM Subcommittee D01.24 and is based upon the data of the New York Society for Paint Technology.

³ See "Comparative Evaporation Rates of Solvents: II," New York Club, Technical Subcommittee No. 66, *Official Digest*, 28, No. 382, 1956, p. 1060.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

⁵ *Annual Book of ASTM Standards*, Vols 05.03 and 14.03.

⁶ The manual Shell thin-film evaporometer is no longer available.

⁷ Syringe: Becton, Dickinson and Co., No. 1YT available from Fisher Scientific Co. Needle: Special Syringe Needle Type LNR, 18 gage, 9 in. long blunt round end, no bevel—available on special order from Becton, Dickinson and Co. through Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15239.

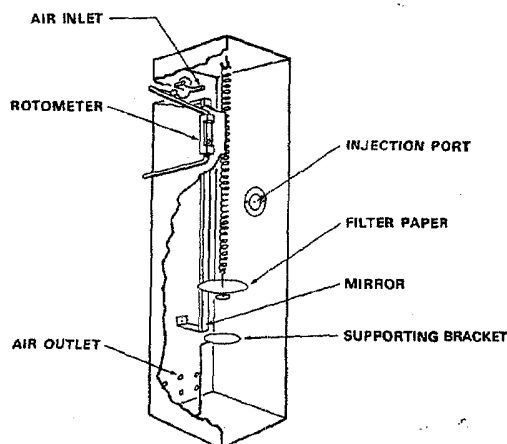


FIG. 1 Details of the Thin-Film Evaporometer

NOTE 2—Precaution: Use of this dehumidification apparatus requires the safety practices relative to the handling, use, and disposal of hazardous acids and caustics be observed. When handling these materials, protective eye or face, or both, shields and protective clothing are recommended.

NOTE 3—In those instances where dry nitrogen is available it may be used directly instead of air and thus eliminate the dehumidification equipment. The use of nitrogen does not alter the evaporation rate.

5.7 *Hygrometer (or other humidity-sensing device)*, capable of indicating low humidities.

5.8 *Thermometers*, of suitable accuracy such as ASTM Bomb Calorimeter thermometer 56C having a range from 19 to 35°C, subdivisions 0.02°C or Thermometer 56F (66 to 95°F with 0.05°F subdivisions), and conforming to the requirements of Specification E 1.

6. Preparation of Evaporometer

6.1 Place the filter paper disk on the wire support, threading the hook through a small hole in the center of the paper. Attach the hook to the steel spring below the sighting disk and allow the paper and the paper support to hang therefrom.

6.2 Close the evaporometer and cabinet doors and allow the temperature in both chambers and the humidity to

equilibrate at the following test conditions:

Cabinet and evaporation temperature: $77 \pm 0.5^\circ\text{F}$ ($25 \pm 0.25^\circ\text{C}$)
Evaporometer humidity: 0 to 5 % relative humidity

Approximately 2 h are required for the humidity to drop to less than 5 %.

6.3 Adjust the air flow to 21 L/min (center of ball float opposite correct mark on the rotometer scale).

7. Conditioning

7.1 Bring the sample or a portion of it to an equilibrium temperature of $77 \pm 1.0^\circ\text{F}$ ($25 \pm 0.5^\circ\text{C}$) in a constant-temperature bath. Determine the specific gravity of the sample at this temperature in accordance with Test Methods D 891.

8. Procedure

8.1 Record the position of the filter paper as indicated by alignment of the sighting disk with its mirror image. This is the no-load position.

8.2 Raise the wire mesh bracket until the bottom of the disk support rests lightly on it.

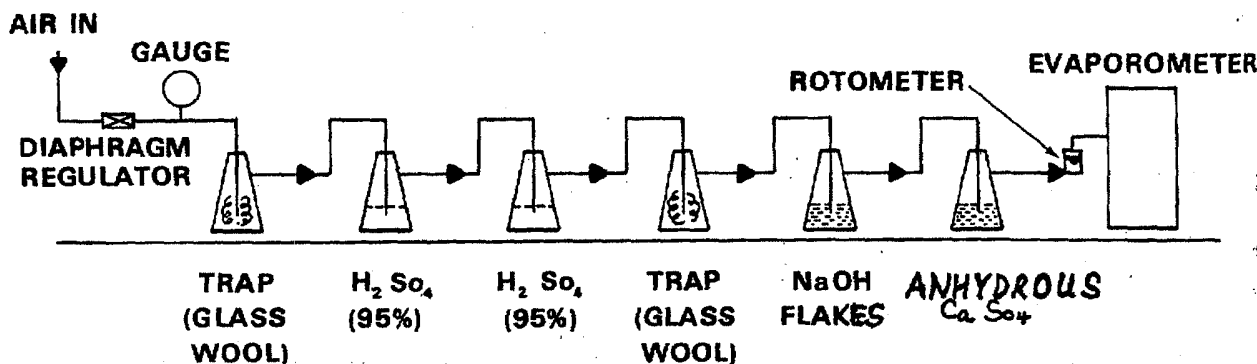
8.3 Withdraw into the syringe 0.70 mL of the solvent which is at $77 \pm 1.0^\circ\text{F}$ ($25 \pm 0.5^\circ\text{C}$). Make certain that all air bubbles are expelled from the syringe and the needle before application of the specimen to the filter paper.

8.4 Insert the hypodermic needle into the small opening on the right-hand side of the instrument and position the needle tip so that it almost touches the disk and is just over the line that was drawn.

8.5 Start applying the solvent to the disk. As the first drop hits the disk, start the timer. The solvent should be applied at a uniform rate in ± 2 s and as evenly as possible along the drawn line. To ensure consistent specimen size, touch the tip of the hypodermic needle to the filter paper to dispense the last drop of solvent.

8.6 Immediately lower the wire mesh bracket away from the disk support. Obtain the first reading of the position of the sighting disk at 40 s and then every 20 s. Record the time and the scale reading on the report form. A sample report form is shown in Annex A2.

NOTE 4—With very slow evaporating solvents, it is not necessary to take readings as often as every 20 s. The operator can determine a



NOTE—One-litre flasks should be used throughout.

FIG. 2 Diagram of Dehumidification Apparatus

suitable time interval after the first 200 s.

8.7 Stop the timer when the sighting disk has returned to the original unloaded position.

NOTE 5—The filter paper may be reused provided the solvent leaves no appreciable residue in evaporating.

9. Calculation

9.1 Calculate the evaporation rate as follows:

$$ER = \frac{C}{S} \times 100(B - Z)$$

where:

$S = V \times D$ and $Z = N - (S/C)$

ER = evaporation rate, wt %,

C = spring constant, g/cm elongation,

S = specimen weight,

$V = 0.70$ mL aliquot volatile liquid at $77 \pm 1.0^\circ\text{F}$ ($25 \pm 0.5^\circ\text{C}$),

D = density of volatile liquid at $77 \pm 1.0^\circ\text{F}$ ($25 \pm 0.5^\circ\text{C}$) (Taken as equivalent to specific gravity but with units of g/mL)

B = scale reading taken during evaporation of aliquot,

Z = zero percent evaporated, scale reading = $N - (S/C)$ and

N = no-load scale reading (100 % evaporated reading).

9.2 Plot the percent evaporated against elapsed time in seconds and draw a smooth curve through the points. From the curve, determine at 10 weight % increments to 90 % and for 95 and 100 % evaporation the time in seconds to the nearest value as follows:

Approximate Elapsed Time to 100 % Evaporated Point, s	Report to Nearest Indicated Value, s
Less than 300	1
300 to 600	5
600 to 1800	10
1800 to 3600	30
3600 to 7200	60
More than 7200	nearest 2 % of indicated value

NOTE 6—The curve drawn through the various points should pass through zero or the origin. If it passes to the right of the origin, the delivery time was in excess of 12 s or an aliquot larger than that specified was delivered. If it passes to the left of the origin, then the aliquot was smaller than specified.

10. Report

10.1 Report the elapsed time in seconds at 10 weight % intervals through 90 % and for 95 and 100 % evaporation, and the relative evaporation rate (*n*-butyl acetate = 1.0). Relative evaporation rate is calculated from the 90 weight % evaporated times for the test solvent and for *n*-butyl acetate (99 % ester).

11. Precision⁸

11.1 On the basis of an interlaboratory study of the test method in which operators in six laboratories determined the 90 % evaporation point of six solvents covering a broad range in evaporation rate, the between-laboratories coefficient of variation was found to be 6.3 % relative at 24 degrees

of freedom after discarding two divergent values. On the basis of the results obtained by three laboratories on three of the solvents having 90 % evaporated times of 200 to 600 s, the within-laboratory coefficient of variation was found to be 0.83 % relative at 18 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—For solvents with 90 % evaporation times of 200 to 600 s, two results, each the mean of two determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 2.5 %.

11.1.2 *Reproducibility*—Two results, each the mean of two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 18.2 %.

METHOD B—EVAPORATION RATE USING THIN-FILM EVAPOROMETER, AUTOMATIC RECORDING

12. Apparatus

12.1 *Evaporometer*, Automatic Thin-Film Evaporometer,⁹ as shown in Fig. 3.

12.2 *Filter Paper Disk*—See 5.4.

12.3 *Syringe*—See 5.5.

12.4 *Dehumidification Equipment*—See 5.6.

12.5 *Strip Chart Recorder*—Any strip chart recorder capable of recording the output signal (0 to 15 mA) from the electronic optical weight-sensing device. The recorder should provide a range of chart speeds including ¼ to 2 in. (6.3 to 50 mm)/min. It is also desirable for the recorder to accommodate 2 or more mA ranges in order to regulate the sensitivity of measurement.

13. Preparation of Evaporometer

13.1 Place the filter paper disk on the wire frame threading the hook through a small hole in the center of the paper. Attach the wire frame to the support hook in the evaporometer.

13.2 Close the evaporometer and cabinet doors and equilibrate both chambers as in 6.2.

13.3 Adjust the air flow to 21 L/min.

14. Conditioning of Sample

14.1 See 7.1.

15. Procedure

15.1 When all components (including the filter paper in place) are at equilibrium, adjust the recording pen to a prominent "zero" position near the edge of the chart on the recorder; then turn the switch for the chart motor to the OFF position. This constitutes the "zero" load and time position for the test.

⁹ The automatic Shell thin-film evaporometer, Apparatus Catalog No. F1522 is available from the FLC Instruments, Inc., 2055 Comprehensive Drive, Aurora, IL 60505. Another evaporometer, the Arizona Instrument EV-1, is available (Arizona Instrument Corp., 2078 East University Drive, Tempe, AZ 85281-4098). Precision data is not yet available, but the instrument operates in the same manner as that described in this test method and is reported to give similar results.

⁸ Supporting data are available from ASTM Headquarters. Request RR:D01-1003.

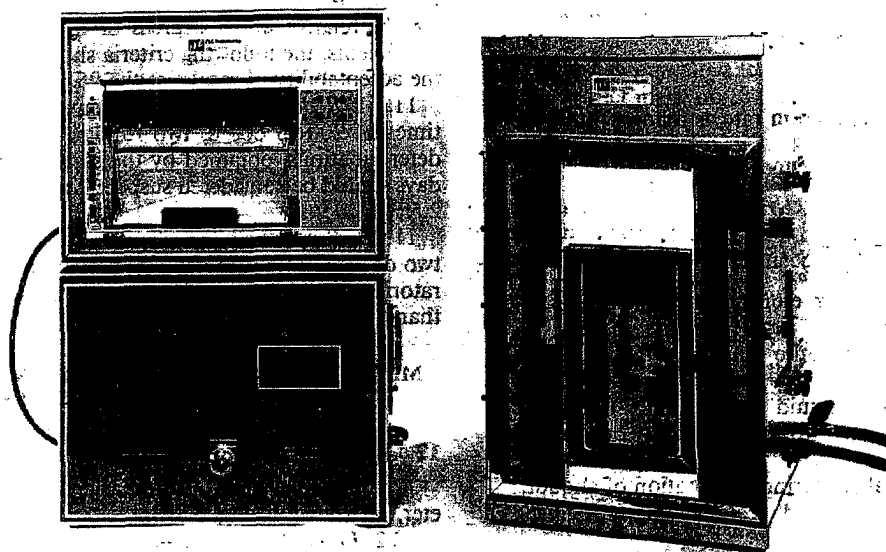


FIG. 3 Automatic Thin-Film Evaporometer

NOTE 7—The milliamperage range and chart speed should be selected, if possible, so that the dimensions of the weight and time axes of the plotted curve are approximately the same length.

15.2 Measure 0.70 mL of test sample into the hypodermic syringe (see 8.3).

15.3 Open the small side door on the right hand side of the insulating cabinet and insert the hypodermic needle through the rubber porthole until the needle tip almost touches the disk and is just over the penciled line.

NOTE 8—Care must be exercised to avoid depressing the plunger of the syringe during this operation. Otherwise the solvent will be accidentally and prematurely dispensed onto the paper before the strip chart recorder is started.

15.4 When all is ready turn the strip chart motor switch to the ON position and simultaneously start distribution of the specimen onto the filter paper. The complete specimen should be dispensed uniformly in 10 ± 2 s along the line. The recorder pen will "advance" immediately to an "apex" position equivalent to the total weight of the specimen, less that portion that evaporated during the application period. The pen will gradually return to its original position as the solvent evaporates and the chart advances. The evaporation is complete when the recording pen has returned to its original "no-load" position.

NOTE 9—It is common for the final portion of the curve to exhibit a "tailing-off." This is due to artifacts of the method such as (1) hydrogen bonding of the last traces of solvent with the cellulose fibers of the filter paper and (2) a gradual diminution of the area of the filter paper wet by solvent (that is, in the final stage of evaporation, drying of the paper progresses from the outer edge toward the center of the disk). Thus, it is common practice for the evaporation cycle to be considered "complete"

when the recording pen returns to 99.5 % of the original displacement.

16. Calculations and Reporting

16.1 Determine the evaporation time in seconds at 10 % weight % increments to 90 %, and for 95 to 100 % from the evaporation curve as follows:

16.1.1 Divide the theoretical recording pen displacement for the total specimen into ten equal units along the weight axis of the evaporation curve; then project the established 10 % divisions to corresponding intersecting points on the evaporation curve. The 0 % evaporated or full-load point at zero time can be obtained either by extrapolation of the evaporation curve back to zero evaporation time or calculation using the weight of the sample and the calibration data for the instrument (See Annex A3). The routine calculation of the 0 % evaporated, full-load point is recommended as a check for correct specimen size.

16.1.2 Multiply the distance along the time axis from the zero starting time by the chart speed factor in seconds giving the total elapsed time for each defined point along the curve. For example, at a chart speed of 1 in./min and a 20 % evaporation point at 3.30 in. the evaporation time is 198 s ($60 \times 3.30 = 198$). Use the procedure given in 9.2 to round results.

16.2 An alternative method of reporting evaporation results is to express an Area Evaporation Rate in terms of grams evaporated per second per square centimetre of evaporating surface. This method is not exact because the evaporation rate is not linear throughout the complete evaporation period, but it is a useful approximation to represent the general volatility of a solvent. The calculation of evaporation rate is as follows:

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$$\text{Area Evaporation Rate, } R = \frac{C \times B}{A - B} \times 10^8$$

where:

R = evaporation rate, $\text{g/cm}^2 \cdot \text{s} \times 10^8$,

C = a factor = 0.00438 mL/cm^2 obtained from:

0.70 mL = specimen size,

0.80 = increment between the 10 % and 90 % evaporation points (the first and last 10 % increment are disregarded),

128 cm^2 = total evaporating surface of 90-mm diameter filter paper,

then $C = \frac{0.70 \times 0.80}{128} = 0.00438 \text{ mL/cm}^2$

C = specific gravity of the solvent at 77°F (25°C),

A = 90 % evaporation time, s, and

B = 10 % evaporation time, s.

The multiplicand, 10^8 , is inserted in the equation to avoid decimal fractions.

17. Precision⁸

17.1 On the basis of an interlaboratory study of the test

method in which operators in four laboratories determined the 90 % evaporation point of seven solvents covering a broad range in evaporation rate, the within-laboratory coefficient of variation was found to be 1.78 % relative at 35 degrees of freedom and the between-laboratories coefficient of variation was found to be 3.88 % relative at 28 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

17.1.1 *Repeatability*—Two results, each the mean of two determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 5.1 %.

17.1.2 *Reproducibility*—Two results, each the mean of two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 11.25 %.

18. Index Terms

18.1 These test methods are indexed under the following terms: evaporation rate, Shell thin film evaporometer, and evaporation rates of solvents.

ANNEXES

(Mandatory Information)

A1. CALIBRATION OF THE THIN-FILM EVAPOROMETER, MANUAL RECORDING

A1.1 Remove the filter from its wire support and replace it with a small piece of aluminum foil, about 50 by 50 mm; then again suspend the support from the coil spring. Record the position of the sighting disk. Load the spring by placing a 1-g analytical balance weight onto the aluminum foil and again record the elongation of the spring. Thus, the spring constant, C , in grams per centimetre, is equal to 1.000 g divided by the elongation in centimetres.

A1.2 *Standardization of the Evaporometer*—Since there may be slight variations in manufacture, the evaporometer should be standardized prior to routine usage. The standard solvent, *n*-butyl acetate, (99 % ester), should have a 90 % evaporation time of $470 \pm 10 \text{ s}$ under the prescribed test conditions. If the evaporation time is outside these limits, a slight increase or decrease of the air flow should be sufficient to bring about the desired results.

A2. SAMPLE EVAPORATION RATE REPORT FORM

A2.1 A sample evaporation rate report form is shown in Table A2.1.

TABLE A2.1 Evaporation Rate Report Form

No. _____ Specimen _____ Date _____

Spring No. _____; Specimen Volume, V , _____ m³

Spring constant, C , _____ g/cm Elongation: Specimen Density, D , _____

No-Load Scale Reading, N , _____ cm; Specimen Weight, $S = V \times D$, _____

Zero %-Scale Reading, $Z = (N - (S/C))$ _____; Spring Elongation, $\approx S/C$ _____

Calculation: weight percent Evap. = $\frac{C}{S} \times 100 (S - Z)$.

Evaporometer Temperature _____

Relative Humidity _____

Cabinet Temperature _____

^a Measured at 77 ± 1.0°F (25 ± 0.5°C).

TIME, s	SCALE READING (B)	SCALE READING (B-Z)	SPECIMEN EVAPORA- TION % (W)	TIME, s	SCALE READING (B)	SCALE READING (B-Z)	SPECIMEN EVAPORA- TION % (W)
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A3. CALIBRATION OF THE THIN-FILM EVAPOROMETER, AUTOMATIC RECORDING

A3.1 The total deflection of the recording pen in chart units divided by the weight added corresponds to the sensitivity of the apparatus. For example, if the total pen deflection is 60 chart divisions for a 0.500-g weight, the sensitivity factor is 120 chart divisions per gram. This value can then be used to establish the 100 % specimen load at "zero" evaporation time and to calculate the percent evaporated at various time intervals. For instance, pure *n*-butyl acetate (99 mol %) has a density of 0.878 at 25°C, so a 0.70-mL specimen at the cited calibration would deflect the pen 73.75 chart units (that is, $0.878 \times 0.70 \times 120 = 73.75$).

Each 10 % increment that evaporates corresponds to 73.75 chart divisions.

A3.2 Standardization of the Evaporometer—Since there may be slight unavoidable variations in manufacture, the evaporometer should be standardized prior to routine usage. Normal butyl acetate (99% ester) should have a 90% evaporation time of 470 ± 10 s under the prescribed test conditions. If the evaporation time is outside these limits, a small adjustment should be made to the position of the inlet air (or nitrogen) ports within the inner chamber of the evaporometer. **Caution:** The air should *not* be directed above or onto the filter paper.

A4. EVAPORATION RATES OF VOLATILE MATERIALS

A4.1 The evaporation values presented in Table A4.1 are typical of commercial materials. Deviations from the values shown can be expected due to normal variations in test

conditions and purity, composition, source, etc. of test samples.

TABLE A4.1 Evaporation Rates of Volatile Materials

	Automatic Evaporometer		Manual Evaporometer	
	Seconds to 90 % Evaporation	Relative Rate <i>n</i> -Butyl Acetate = 1.0	Seconds to 90 % Evaporation	Relative Rate ^a <i>n</i> -Butyl Acetate = 1.0
Acetone	82	5.7	75	6.0
Amyl acetate (ex. Fusel oil) (85 to 88 %)	690	0.68	680	0.66
Amyl acetate, primary, (mixed isomers) (95 %)	1 200	0.39	1 110	0.41
Amyl alcohol, primary (mixed isomers)	2 300	0.20	...	0.3
<i>tert</i> -Amyl alcohol	505	0.93	430	1.0
Benzene	133	3.5	...	...
Isobutyl acetate	305	1.5	305	1.5
<i>n</i> -Butyl acetate (90 %)	460	1.0	450	1.0
<i>n</i> -Butyl acetate (99 %)	470	1.0	...	...
<i>sec</i> -Butyl acetate (90 %)	260	1.8	241	1.9
Isobutyl alcohol	740	0.64	640	0.70
<i>n</i> -Butyl alcohol	1 080	0.44	1 010	0.45
<i>sec</i> -Butyl alcohol	565	0.83	490	0.92
Isobutyl isobutyrate	970	0.48	980	0.46
Butyl lactate	14 600	0.03	13 600	0.03
Cyclohexanol	9 200	0.05	...	0.1
Cyclohexanone	1 570	0.30	1 720	0.26
DEGMBE ^B	150 000	0.01	...	0.01
DEGMBE acetate	328 000	0.01	...	0.01
DEGMEE ^B	27 800	0.02	...	...
DEGMME ^B	26 300	0.02	...	0.01
Diacetone alcohol	3 840	0.12	4 320	0.10
Diethyl ketone	205	2.3	179	2.5
Diisobutyl ketone	2 430	0.19	2 460	0.18
Dimethyl formamide	2 280	0.21	2 400	0.19
Ethyl acetate (85 %)	115	4.1	110	4.1
Ethyl acetate (95 %)	117	4.0	108	4.2
Ethyl acetate (99 %)	117	4.0	100	4.5
Ethyl alcohol (95 %)	330	1.4	...	...
Ethyl alcohol (100 %)	280	1.7	...	...
Ethyl amyl ketone	1 770	0.27	2 040	0.22
Ethylbenzene	562	0.84	...	...
Ethyl butyl ketone	1 080	0.44	1 020	0.44
Ethyl ether	40	11.8	...	...
Ethylene glycol	...	0.01	...	...
EGMBE ^B	6 780	0.07	8 100	0.06
EGMBE ^B acetate	14 300	0.03	13 300	0.03
EGMEE ^B	1 210	0.39	1 260	0.36
EGMEE ^B acetate (95 %)	2 700	0.17	2 820	0.16
EGMEE ^B acetate (99 %)	2 520	0.19	2 520	0.18
EGMME ^B	880	0.53	...	...
2-Ethyl hexanol	25 700	0.02	...	0.01
2-Ethyl hexyl acetate (95 %)	13 400	0.04	...	0.03
Ethyl lactate	2 580	0.18	2 490	0.18
Hexyl acetate	2 580	0.18	2 400	0.19
Hexylene glycol	...	0.01	...	...
<i>n</i> -Hexane	60	7.8	...	...
Isophorone	20 000	0.02	27 600	0.02
Mesityl oxide	535	0.88	555	0.81
Methyl acetate (80 %)	93	5.0	93	4.8
Methyl alcohol	220	2.1	220	2.0
Methyl amyl acetate (95 %)	1 000	0.47	900	0.50
Methyl ethyl ketone	121	3.9	112	4.0
Methyl isoamyl ketone	1 020	0.46	1 040	0.43
Methyl isobutyl carbinol	1 710	0.27	1 790	0.25
Methyl isobutyl ketone	280	1.7	277	1.6
Methyl isopropyl ketone	164	2.9	...	...
Methyl <i>n</i> -amyl ketone	1 380	0.34	1 250	0.36
Methyl <i>n</i> -propyl ketone	200	2.4	...	...
4-Methoxy - 4-methyl pentanone-2	295	1.6	...	...
Nitroethane	445	1.1	390	1.2
Nitromethane	360	1.3	330	1.4

TABLE A4.1 Continued

	Automatic Evaporometer		Manual Evaporometer	
	Seconds to 90 % Evaporation	Relative Rate <i>n</i> -Butyl Acetate = 1.0	Seconds to 90 % Evaporation	Relative Rate ^a <i>n</i> -Butyl Acetate = 1.0
1-Nitropropane	645	0.73	600	0.75
<i>n</i> -Octane	295	1.6	...	...
Isopropyl acetate (95 %)	134	3.5	125	3.6
<i>n</i> -Propyl acetate	220	2.1	197	2.3
Isopropyl alcohol	320	1.5	290	1.6
<i>n</i> -Propyl alcohol	530	0.89	450	1.0
Propylene glycol	...	0.01	...	...
Isopropyl ether	57	8.2	...	...
Tetrahydrofuran	97	4.8	90	5.0
Toluene	235	2.0	229	2.0
Water	1 290	0.36	...	...
Xylene	610	0.77	620	0.73

^a These data are based on tests made with commercial grade *n*-butyl acetate containing 90 % ester.

^b Key—EGMME, Ethylene glycol monomethyl ether (2-methoxy ethanol), EGMEE, Ethylene glycol monoethyl ether (2-ethoxy ethanol), EGMBE, Ethylene glycol monobutyl ether (2-butoxy ethanol), DEGMME, Diethylene glycol monomethyl ether, DEGMEE, Diethylene glycol monoethyl ether, DEGMBE, Diethylene glycol monobutyl ether.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Detection of Lead in Paint and Dried Paint Films¹

This standard is issued under the fixed designation D 3618; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This test method is intended as a screening test to determine if the solids in a paint contain more than 0.5 % lead. The test described barely detects the presence of 0.4 % but gives a definite positive result at the 0.5 % level.

NOTE 1—This test method may be used to detect the presence of lead at concentrations higher or lower than 0.5 % by making appropriate changes in the specimen size and reagent quantities specified.

1.2 Paints giving an unexpected positive or questionable result should be analyzed quantitatively for lead, using Test Method D 3335.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²
D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings³
D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy³

3. Summary of Test Method

3.1 The sample of liquid paint or dried film is prepared by dry ashing a weighed specimen at 475 to 500°C. The ash is extracted with hot sodium hydroxide solution and a drop of the extract is transferred to filter paper. Lead present is oxidized to lead peroxide with bromine water, then treated with "tetrabase" to produce a blue quinoidal salt. Known amounts of lead are added to standard paints that are concurrently tested to provide a base for comparison.

4. Significance and Use

4.1 The permissible level of heavy metals in certain

coatings is specified by governmental regulatory agencies. This test method provides a fully documented procedure for determining low concentrations of lead present in both water and solvent-reducible coatings to determine compliance.

5. Apparatus

- 5.1 *Burner*, Meker-type.
5.2 *Crucibles*, porcelain, high-form, 15-mL, with covers.
5.3 *Filter Paper*, ashless, medium texture.
5.4 *Hot Plate*, with variable surface temperature control over the range from 70 to 200°C.
5.5 *Muffle Furnace*, maintained at $475 \pm 25^\circ\text{C}$.
5.6 *Syringe*, glass, 2-mL.
5.7 *Volumetric Flasks*, 50, 100, 1000-mL.
5.8 *Paint Shaker*.
5.9 *Paint Draw-Down Bar*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water conforming to Type II of Specification D 1193.

6.3 *Acetic Acid*, glacial.

6.4 *Ammonium Hydroxide* (1+1)—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH , sp gr 0.90) with 1 volume of water.

6.5 *Bromine Water*, saturated.

6.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

6.7 *Lead Standard Stock Solution, Aqueous* (1 mg/mL)—Dissolve 1.600 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 100 mL of water, add 2 mL of nitric acid (sp gr 1.42), and dilute to 1 L.

6.8 *Lead Standard Working Solution, Aqueous* (0.2 mg/mL)—Pipet 10 mL of the aqueous lead standard stock

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 31 and Sept. 27, 1985. Published November 1985. Originally published as D 3618 - 77. Last previous edition D 3618 - 77 (1984).

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ "Reagent Chemicals, American Chemical Society Specifications." American Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

solution into a 50-mL volumetric flask and dilute to volume with water.

6.9 *Toluene*, technical grade.

6.10 *Lead Standard Stock Solution, Solvent-Based* (1 mg/mL)—Dissolve 4.2 g of lead naphthenate (containing 24 % lead) in 100 mL of toluene and dilute to 1 L.

NOTE 2—Other toluene-soluble lead driers such as lead tallate, lead linoleate, and lead octoate may also be used provided that the lead content is accurately known and the appropriate weight is diluted to 100 mL to provide a toluene solution containing 1 mg/mL of lead.

6.11 *Lead Standard Working Solution, Solvent-Based* (0.2 mg/mL)—Pipet 10 mL of the solvent-based standard stock solution into a 50-mL volumetric flask and dilute to volume with toluene.

6.12 *Sodium Hydroxide Solution* (12 % w/v)—Carefully dissolve 12 g of sodium hydroxide in 100 mL of water.

6.13 *Paint, Standard Solvent-Reducible*, any solvent-reducible paint known to contain less than 100 ppm of lead.

6.14 *Paint, Standard Water-Reducible*, any water-reducible paint known to contain less than 100 ppm of lead.

6.15 *Tetabase Reagent* (4,4'-Methylenebis (N,N-dimethylaniline)), 1 % in 10 % *Acetic Acid*—Dissolve 1 g of tetabase³ in 50 mL of water to which 10 mL of glacial acetic acid has been added. Dilute to 100 mL with water.

7. Hazards

7.1 Glacial acetic and concentrated nitric acids will cause burns of the skin and eyes. Sodium hydroxide is corrosive. Use care in handling these materials. Avoid contact with skin. Refer to suppliers' Material Safety Data Sheets. Bromine water should be prepared and used in a laboratory hood.⁶

7.2 Use only a rubber bulb aspirator for pipetting liquids.

8. Procedure

8.1 If the sample is a liquid coating, mix it until homogeneous, preferably on a mechanical shaker. Determine the nonvolatile content in accordance with Guide D 2832.

NOTE 3—Recover dried paint films from previously coated substrates (being careful not to remove any underlying material from the substrate) or prepare in the laboratory from liquid samples. For the laboratory preparation, flow some of the well-mixed sample onto a clean glass plate. The use of a paint draw-down bar is recommended to obtain a uniform film thickness not exceeding 2 mils (50 μ m). Allow to dry in an oven at 105°C for a minimum of 1 h. Scrape the dried film off the glass plate, preferably with a single edge razor blade.

8.2 Weigh to 0.1 mg, 50 mg of paint solids into a porcelain crucible or 50-mL glass beaker. For a liquid coating, determine the specimen weight to be taken by the following equation:

$$S = \frac{5}{C}$$

where:

S = specimen weight, g, and
 C = nonvolatile content, %

³ Eastman-244 (Eastman Organic Chemicals, Rochester, NY 14650) has been found to be satisfactory; equivalent grades may be used.

⁶ Saturated bromine water may be purchased from chemical supply houses.

NOTE 4—Crucibles or beakers used in this test method should be new or in very good condition. Otherwise, it is possible that some lead may be lost and a false negative obtained.

8.3 Weigh 50 mg of solids from the appropriate standard paint (solvent-reducible or water-reducible) into each of two additional porcelain crucibles or 50-mL glass beakers. Using a 2-mL glass syringe, add 1.0 mL of the appropriate 200-ppm standard working solution (solvent-reducible or water-reducible) to one of the crucibles or beakers, and 1.8 mL to the other.

NOTE 5—The standard paint to which has been added 1.0 mL of standard working solution contains approximately 0.4 % lead based on the paint solids, and the one to which has been added 1.8 mL of standard working solution will contain approximately 0.72 % lead based on the paint solids.

8.4 Place all three crucibles or beakers on a cold hot plate and slowly increase the temperature until the material is dried. With some types of coatings, an initial oven-drying at 105°C may be necessary to remove solvents without incurring losses due to spattering.

8.5 When the specimens appear to be dry, or when starting with a dried film, gradually increase the temperature of the hot plate until the material chars.

8.6 After charring is complete, place the crucibles or beakers in a preheated muffle furnace and ash at 475 to 500°C.

8.7 When the ashing appears to be complete, (approximately 1 to 2 h) remove the crucibles or beakers from the muffle furnace and allow them to cool to room temperature. Add 5 mL of 3 N sodium hydroxide solution to each crucible or beaker and mix thoroughly with a stirring rod. Scrape any adhering residue from the sides or bottom of the container and break up any particles, using a separate stirring rod for each specimen.

8.8 Place covers on the crucibles or watch glasses on the beakers, and boil gently for approximately 5 min. Remove from the hot plate and allow the contents to settle. Do not filter.

8.9 With the tip of the stirring rod, transfer 1 to 2 drops of each extract to the center of a piece of filter paper. Using separate droppers, successively apply directly on each specimen spot a drop of bromine water and a drop of NH_4OH solution. Remove excess ammonia by holding each filter paper over a hot plate until no odor is detectable, but do not take to complete dryness. Place 2 drops of tetabase reagent on each spot and compare the color developed on the specimen spot with those on the standard paint spots. To ensure that maximum color development has been achieved, add 1 or 2 more drops of tetabase reagent on the center of each spot after the first addition has almost-dried (if still wet, the color may diffuse).

NOTE 6—Although the color is stable, it is barely visible in the range from 0.4 to 0.5 %. Comparing results with those obtained on the two standard paints helps to orient the observer to the color intensity to be expected.

NOTE 7—Certain iron-based pigments have high natural manganese levels. The ash extract of products containing high manganese may produce a diffuse blue ring on the filter paper. With experience, this is easily distinguished from the small blue spot produced by lead.

9. Report

9.1 Report a positive test (lead content equal to or in excess of 0.5 % of paint solids) when a blue color forms and persists for several seconds.

10. Precision and Bias⁷

10.1 An interlaboratory test was conducted in which

⁷ Supporting data are available from ASTM Headquarters. Request RR: D01-1007.

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11. Keywords

11.1 lead; lead in paints and dry paint films; spat test for lead



Standard Method for Testing Antifouling Panels in Shallow Submergence¹

This standard is issued under the fixed designation D 3623; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers a procedure for testing antifouling compositions in shallow marine environments and a standard antifouling panel of known performance to serve as a control in antifouling studies.

NOTE—Subcommittee D01.45 has a revised rating procedure now being evaluated by round robin.

2. Referenced Documents

2.1 ASTM Standards:

A 569/A 569M Specification for Steel, Sheet and Strip, Carbon (0.15 Maximum Percent), Hot-Rolled, Commercial Quality²

D 2200 Pictorial Surface Preparation Standards for Painting Steel Surfaces³

2.2 U.S. Military Specifications:

MIL-P-15328D Primer Pretreatment (Formula 117 for Metals)⁴

MIL-P-15929C Primer Coating, Shipboard, Vinyl-Red Lead (Formula 119—For Hot Spray)⁴

MIL-P-15931B Paint, Antifouling, Vinyl, Red (Formula 121/63)⁴

MIL-S-22698A Steel Plate, Carbon, Structural⁴

3. Significance and Use

3.1 This method is designed as a screening test in evaluating antifouling coating systems. Results of the standard system in a specific marine environment are included to assist in interpreting results.

3.2 Antifouling systems providing positive comparisons with the standard system should be considered acceptable for use in protecting underwater marine structures.

3.3 The degree and type of fouling will vary depending on the environment. Hence, differences in geographic location of test sites, in time of year when panels are exposed, and in weather conditions from 1 year to the next can affect results. Therefore, a fouling census on a nontoxic surface is taken. For the exposure to be valid the nontoxic surface should show heavy fouling, and the standard system should show significantly less fouling than the nontoxic surface.

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.45 on Marine Coatings.

Current edition approved Oct. 27, 1978. Published January 1979. Originally published as D 3623 - 78. Last previous edition D 3623 - 78.

² Annual Book of ASTM Standards, Vol 01.03.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

4. Terminology

4.1 Definition:

4.1.1 *shallow submergence*—an immersion to depths between 1 and 10 ft (0.3 and 3.0 m).

4.2 Abbreviations:

4.2.1 The following abbreviations are used in reporting test results:

Al	algae
Barn	barnacles
E.B.	encrusting bryozoans
Hyd	hydroids
Sl	slime
Tun	tunicates
C.F.	completely fouled
F.R.	fouling resistance rating
A.F.	antifouling rating
A.C.	anticorrosive rating
O.P.	overall performance rating
CO	coelenterates
F.B.	filamentous bryozoans
Mol	molluscs
PC	polychaetes

4.2.2 Algal Mups may be delineated by classification notation by phylum as follows:

Al-b	Phaeophyta (brown)
Al-bg	Cyanophyta (blue-green)
Al-g	Chlorophyta (green)
Al-r	Rhodophyta (red)

5. Apparatus

5.1 *Blast Cleaning Apparatus* capable of preparing panels in compliance with 8.1.

5.2 *Application Equipment* consisting of brush, roller, conventional spray, or airless spray. Usually the equipment is dictated by the physical properties of the coating and the film thickness desired.

5.3 *Exposure Rack* to provide firm positioning of the specimen panels such that they are held vertically in place in spite of the current and are electrically insulated from metallic contact with the rack or other panels. The rack should be positioned such that the prevailing tidal currents will move parallel to the panel face, and the panels will be immersed to a depth of a minimum of 1 ft (0.3 m) and a maximum of 10 ft (3 m). In a rack where the panels are stacked front to back, they should be spaced at least 2 1/2 in. (60 mm) apart, with the two end positions filled with blank panels. In a rack where the panels are mounted side by side, the distance between adjacent panels should be a minimum 1/2 in. (1.5 mm).

6. Materials

6.1 *Test Panel*—The substrate for the standard antifouling coating system shall be medium low-carbon steel

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plate per Specification A 569,⁵ 1/8 in. thick by 6 to 10 by 10 to 12 in. (3 mm thick by 150 to 250 mm by 250 to 300 mm) with a minimum area per side of 72 in.² (465 cm²). A 1/4-in. (6-mm) diameter hole, 1/2 to 1 in. from the top and centered shall be drilled for holding the panel while handling and painting. The test coating system shall be applied to that substrate for which it is designed provided there is a minimum area per side of 72 in.² (466 cm²).

6.2 *Standard Coating System*—The standard antifouling coating system shall consist of:

6.2.1 Green pretreatment coating conforming to U.S. Military Specification MIL-P-15328B (Formula 117), B revision only.

6.2.2 Vinyl red lead primer conforming to U.S. Military Specification MIL-P-15929C (Formula 119), C revision only.

6.2.3 Vinyl antifouling coating conforming to U.S. Military Specification MIL-P-15931B (Formula 121/63), B revision only.

6.3 *Test Coating System*—The test antifouling coating may be applied to the standard primer system, or to any other suitable anticorrosive primer system.

7. Safety Precautions

7.1 *Caution*—Antifouling paints contain toxic materials that could cause skin and eye irritation on contact and adverse physiological effects if ingested or inhaled. In the preparation of panels and the application of various types of antifouling paints the use of appropriate protective clothing and equipment is required consistent with local, state, and federal government regulations, and recognized industrial and technical standards. Spills, overspray, and unused material should not be flushed down the drain, but should be disposed of as hazardous waste.

8. Procedure

8.1 Abrasive blast the required number of panels to near-white metal (Sa 2 1/2 of Method D 2200) to obtain a profile of 1 to 1.5 mils (25 to 38 μm).

NOTE 1—A profile of 1 to 1.5 mils (25 to 38 μm) can be obtained with the following parameters: (1) Type and size of grit, No. 46 (sand); (2) Pressure, 90 psi (620 kPa); (3) Angle, 90 deg; (4) Distance from surface, 3 to 5 in. (75 to 125 mm); (5) Nozzle size, 3/8 in. (9 mm).

8.2 On the clean, dry, uncontaminated, blasted surface apply to each standard panel one coat of pretreatment coating MIL-P-15328D to give a nominal dry film thickness of 1/2 mil (13 μm).

NOTE 2—All coating drying times are for a minimum temperature of 21°C and a maximum relative humidity of 70 %.

8.3 Within 24 h of application of the pretreatment coating apply the first coat of red lead vinyl primer MIL-P-15929C. Apply a total of four coats of red lead vinyl primer to give a nominal dry film thickness of 6 mils (150 μm). Allow a minimum of 2 h and a maximum of 24 h drying between coats of red lead vinyl primer.

8.4 Allowing a minimum of 2 h and a maximum of 24 h drying after the last coat of red lead vinyl primer, apply the first coat of vinyl antifouling coating MIL-P-15931B to the

standard panels, and the test coating to the test panels. A 3/4-in. (19-mm) numbered vinyl tape can be applied before the second coat of antifouling coating for identification. Allowing a minimum of 2 h and a maximum of 24 h drying after the first coat, apply a second coat of the respective antifouling coating to give a nominal dry film thickness for both antifouling coats of 4 mils (100 μm).

8.5 Before immersion permit the second coat of antifouling coating to dry a minimum of 4 h and a maximum of 2 weeks, the latter time allowing for shipping the panels to the immersion site.

8.6 Expose for a minimum period of 1 year at an immersion site with a high incidence of fouling as indicated by attachments on a dark nontoxic surface such as slate. A monthly fouling census as well as a yearly accumulation when appropriate is required.

8.7 Evaluate the antifouling panels for surface fouling and physical condition of the film system at least monthly as follows using the report form in Table 1.

8.7.1 *Fouling on Surfaces*—Rate fouling present on that portion of the antifouling test surface which is intact at the time of inspection (see 9.1). Rate both sides of the panel if appropriate. Ignore fouling present on the substrate or on anticorrosive undercoats. Barnacles, polychaetes, coelenterates, etc., that are immature or loosely attached should be so reported in the appropriate space. Report fouling by initial algal germination, low-form algae and diatoms as "algal slime." Report absorbed organic and inorganic chemicals, trapped silt and detritus, and other unidentified slimes as "silt."

8.7.2 *Physical Condition*—Rate the condition of coating films in accordance with 9.2. Record qualitative descriptions of film deterioration and discoloration in this column, and unless otherwise specified, refer only to the antifouling test surface. Indicate deterioration of undercoats, when evident, by the notation "A.C.". For example, "Peeling, A.F. from A.C." or "Chipping, A.C. from steel".

9. Calculations:

9.1 *Fouling Resistance (F.R.)*—Award each test surface free of fouling except for the presence of algal spores and other biological slimes a rating of 100. Reduce the rating to 95 if only incipient fouling is present. If mature forms of fouling are present, obtain the rating by subtracting from 95 the sum of the number of individuals present and percent surface covered by colonial forms. For example, if the "fouling on surface" is:

Barn	6, 3 to 10 mm
E.B.	None
Others	Mol 1,20 mm
	Al-g (green) 10 %

then the F.R. percent rating would be $95 - (6 + 1 + 10) = 78$.

9.2 *Physical Condition:*

9.2.1 *Antifouling Film (A.F.)*—Award an antifouling test surface having no physical defects a rating of 100. Subtract the percent surface affected by film defects from 100 to obtain the rating for imperfect films.

9.2.2 *Anticorrosive Film (A.C.)*—Obtain the rating by the same procedure as 9.2.1.

⁵ See also U.S. Military Specification MIL-S-22698, Type 1, Class A.

TABLE 1 Behavior Report of Experimental Surfaces

Origin:
Series:
Base:
Size:

Place of Immersion:
Depth of Immersion:
Date Immersed:
Date Inspected:
Inspected by:

Test Surface No.	Fouling on Surfaces ^A	Physical Condition	Percent Ratings			
			F.R.	A.F.	A.C.	O.P.
	Barn: E.B.: Others:					
	Barn: E.B.: Others:					
	Barn: E.B.: Others:					
	Barn: E.B.: Others:					
	Barn: E.B.: Others:					
	Barn: E.B.: Others:					
	Barn: E.B.: Others:					

^A Fouling reported as found on the more heavily fouled surface. Solitary forms reported numerically; colonial forms by percent surface covered, Al: algae; Barn: barnacles; E.B.: encrusting bryozoans; Hyd: hydroids; Tun: tunicates; C.F.: completely fouled; CO: coelenterates; F.B.: filamentous bryozoans; Mol: molluscs; Pol: polychaetes.

9.3 Overall Performance (O.P.)—For overall performance, award the panel the lowest percent rating of the three preceding values.

9.4 Normalization—The rating system described above is based on a minimum test on one side area of 72 in.² (465 cm²). Correct the percent ratings for test surfaces of non-standard dimensions for the difference between the minimum standard panel area of 72 in.² (465 cm²) and the area on one side of the test specimen.

10. Report

10.1 Report the results of the immersion test in terms of fouling resistance and overall performance for both the material under test and the standard system.

10.2 Other Data—Report the place, depth, and date of immersion, whether mounted from a dock or a floating raft, date the panels were removed and inspected, panel size, and panel identification number. A census of fouling on a nontoxic surface taken each month for the period of expo-

sure must be included in the report.

11. Precision

11.1 The precision statements are based on an inter-laboratory study in which seven laboratories prepared pairs of panels coated with the standard antifouling system, submerged them, and after 1 year rated the fouling. The within-laboratory standard deviation was found to be 2.08. The between-laboratory standard deviation was found to be 4.67. Based on these standard deviations, the following criteria should be used to judge the acceptability of results at the 95 % confidence level:

11.1.1 Repeatability—Two results each the mean of duplicates obtained by the same operator should be considered suspect if they differ by more than seven units.

11.1.2 Reproducibility—Two results each the mean of duplicates obtained by operators in different laboratories should be considered suspect if they differ by more than 16 units.

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ANNEXES

A1. STANDARD COATING SYSTEM FORMULAS

TABLE A1.1 Green Pretreatment Coating, MIL-P-15328D (Formula 117)

	Pounds per 100 gal of Mixed Material	Gallons per 100 gal of Mixed Material
Ingredients of resin component (80 gal):		
Polyvinyl-butylal resin ^A	56	6.10
Zinc chromate (insoluble type) ^B	54	1.78
Magnesium-silicate (Type A or B of Specification MIL-M-15173)	8	0.34
Lampblack (Specification TT-L-70)	0.6	0.04
Butyl alcohol, normal (Spec. TT-B-846)	125	18.48
Isopropyl alcohol, 99 % ^C	353	53.80
Water	15	1.80
Ingredients of acid component (20 gal):		
Phosphoric acid (Class A of Spec. O-P-313)	28	2.0
Water	25	3.0
Isopropyl alcohol, 99 % ^C	99	15.0

^A The resin shall be a polyvinyl partial butylal resin containing only poly(vinyl butylal), poly(vinyl alcohol), and poly(vinyl acetate) in the molecule. The resin shall contain 18.0 to 20.0 % vinyl alcohol, and not more than 1.0 % of vinyl acetate. A 6 % solution of the resin in methanol shall have a viscosity of 12 to 18 cP at 20°C. The specific gravity of the resin shall be 1.05 to 1.15.

^B The zinc chromate shall be of an insoluble type, showing an analysis of 16 to 19 % CrO₃, and 67 to 72 % ZnO, and not more than 1 % water-soluble salts.

^C Isopropyl alcohol, 99 % shall have a specific gravity of 0.785 to 0.790 at 20/20°C and a distillation range not greater than 1.5°C and this range shall include 82.3°C.

TABLE A1.2 Vinyl Red Lead Primer, MIL-P-15929C (Formula 119)

Ingredients	Specifications	Pounds per 100 gal	
		Composition G	Composition L
Red lead, 98 % Pb ₃ O ₄		247.0	247.0
Organic suspension agent 24 % NV		8.2	8.2
Vinyl resin ^A		162.8	162.8
Tricresyl phosphate	TT-T-656	16.8	16.8
Methyl isobutyl ketone	TT-M-268	284.7	
Methyl n-butyl ketone ^B	TT-M-261		273.2
Methyl ethyl ketone	TT-M-261	47.0	105.5
Toluene	TT-T-548	217.7	75.1
Aliphatic naphtha ^C	TT-N-95(I)		81.1

^A Hydroxyl containing vinyl chloride-acetate copolymer (89.5 to 91.5 % vinyl chloride, 5.3 to 7.0 % vinyl alcohol and 2 to 4.0 % vinyl acetate); white powder, specific gravity not under 1.35, not less than 98 % through sieve 20.

^B To contain not over 5 volume % of branched chain ketones.

^C To contain not over 11 volume % of aromatic hydrocarbons.

TABLE A1.3 Vinyl Antifouling Coating, MIL-P-15931B (Formula 121/63)

Ingredients	Specifications	Pounds ^A
Cuprous oxide	MIL-P-15169	1440
Rosin	LLL-R-626, Class A, Grade WW	215
Vinyl resin ^B		55
Tricresyl phosphate	TT-T-656	50
Methyl isobutyl ketone	TT-M-286	165
Xylene	TT-X-916	115
Antisettling agent ^C		5 to 9

^A The formula given is slightly in excess of 100 gal to allow for normal manufacturing loss, may be proportioned to the size batch desired.

^B The resin shall be a vinyl chloride-vinyl acetate copolymer. It shall contain 85 to 88 % vinyl chloride and 12 to 15 % vinyl acetate. The resin shall have a specific gravity of 1.35 to 1.37. Material shall be furnished as a powdered white solid, not less than 98 % of which shall pass through a No. 20 sieve, conforming to RR-S-366.

^C The antisettling agent shall be sufficient to prevent hard pigment settling and otherwise meet requirements of Military Specification MIL-P-15328D, Section 3.4.6.

A2. RESULTS OF EXPOSURE OF STANDARD ANTIFOULING PANELS 1 YEAR AT MIAMI BEACH, FLA., AT A MINIMUM DEPTH OF 1 FT (0.3 m) UNDER A FLOATING RAFT

NOTE—Physical condition or its extent indeterminate, or both, due to intensive, extensive algae spore growth. Ratings for A.F. and A.C. are as recorded before surface became obscured by spore foulings.

Origin: ASTM Subcommittee 45
Series: #1
Base: Steel
Size: 10 by 12 in. and 6 by 12 in. and 8 by 10 in.

Place of Immersion: Miami Beach
Date Immersed: 11 June 1971
Date Inspected: 11 June 1972
Inspected by: F. Lepicard

Test Surface No.		Fouling on Surfaces ^A	Physical Condition	Percent Ratings			
				F.R.	A.F.	A.C.	O.P.
VAL/1	numbered	Barn: incipient E.B.: 2 % Others: A1 (green and red) 1 % A1 (spores) 85 %	soft pinhead blistering (extent indeterminate), see note	92	98	100	92
VAL/1	unnumbered	Barn: 10,4–7 mm E.B.: 6 % Others: A1 (spores) 85 %	soft pinhead blistering (extent indeterminate), see note	79	99	100	79
NN/1	numbered	Barn: 2,3 to 10 mm E.B.: 14 % Others: A1 (spores) 90 %	soft eroding (extent indeterminate), see note	79	98	100	79
NN/1	unnumbered	Barn: incipient E.B.: 3 % Others: A1 (spores) 95 %	soft (extent indeterminate), see note	92	99	100	92
MMR/1	numbered	Barn: none E.B.: 1 % Others: A1 (spores) 30 %	eroding	94	90	100	90
MMR/1	unnumbered	Barn: incipient E.B.: none Others: A1 (spores) 50 %	eroding	95	95	100	95
VAL/2	numbered	Barn: incipient E.B.: 2 % Others: A1 (spores) 80 %	indeterminate, see note	93	100	100	93
VAL/2	unnumbered	Barn: incipient E.B.: 4 % Others: A1 (spores) 80 %	indeterminate, see note	91	100	100	91
NN/2	numbered	Barn: 4,3 to 6 mm E.B.: 3 % Others: A1 (spores) 85 %	indeterminate, see note	88	100	100	88
NN/2	unnumbered	Barn: incipient E.B.: 10 % Others: A1 (spores) 90 %	indeterminate, see note	85	100	100	85
MMR/2	numbered	Barn: none E.B.: none Others: A1 (spores) 45 %	Indeterminate, see note	100	100	100	100
MMR/2	unnumbered	Barn: none E.B.: 1 % Others: A1 (spores) 40 %	Indeterminate, see note	94	100	100	94
NLC/1	unnumbered	Barn: none E.B.: 2 % Others: A1 (spores) 85 %	Indeterminate, see note	93	100	100	93
NLC/2	numbered	Barn: incipient E.B.: 5 % Others: A1 (spores) 65 %	indeterminate, see note	90	100	100	90
NLC/2	unnumbered	Barn: none E.B.: none Others: A1 (spores) 90 %	indeterminate, see note	100	100	100	100

FIG. A2.1 Behavior Report of Experimental Surfaces

EPA 171

EPA 171

EPA 271

EPA 271

EPA 371

EPA 371

EPA 471

EPA 471

SW/1

SW/1

SW/2

SW/2

^A Fou
Barn: Ba

FLA.,

FIG. A2.1 (Continued)

Test Surface No.	Fouling on Surfaces ^A	Physical Condition	Percent Ratings			
			F.R.	A.F.	A.C.	O.P.
EPA 171	numbered Barn: 9,3 to 11 mm E.B.: 2 % Others: A1 (red) 1 % A1 (spores) 80 %	Indeterminate, see note	83	100	100	83
EPA 171	unnumbered Barn: none E.B.: 4 % Others: A1 (spores) 85 %	Indeterminate, see note	91	100	100	91
EPA 271	numbered Barn: none E.B.: none Others: A1 (red) 1 % A1 (spores) 90 %	Indeterminate, see note	94	100	100	94
EPA 271	unnumbered Barn: incipient E.B.: 12 % Others: A1 (green and red) 2 % A1 (spores) 80 %	Indeterminate, see note	81	100	100	81
EPA 371	numbered Barn: incipient E.B.: 1 % Others: A1 (red) 1 % A1 (spores) 80 %	Indeterminate, see note	93	100	100	93
EPA 371	unnumbered Barn: incipient E.B.: 6 % Others: A1 (spores) 95 %	Indeterminate, see note	89	100	100	89
EPA 471	numbered Barn: incipient E.B.: 3 % Others: A1 (green and red) 2 % A1 (spores) 90 %	Indeterminate, see note	90	100	100	90
EPA 471	unnumbered Barn: incipient E.B.: 1 % Others: A1 (green and red) 1 % A1 (spores) 85 %	Indeterminate, see note	93	100	100	93
SW/1	numbered Barn: none E.B.: 7 % Others: A1 (spores) 65 %	Indeterminate, see note	88	100	100	88
SW/1	unnumbered Barn: incipient E.B.: 5 % Others: A1 (spores) 75 %	Indeterminate, see note	90	100	100	90
SW/2	numbered Barn: incipient E.B.: 2 % Others: A1 (spores) 90 %	Indeterminate, see note	93	100	100	93
SW/2	unnumbered Barn: none E.B.: none Others: A1 (green and red) 3 % A1 (spores) 85 %	Indeterminate, see note	92	100	100	92

^A Fouling reported as found on the more heavily fouled surface. Solitary forms reported numerically; colonial forms by percent surface covered, Al: Algae; An: Anomia; Barn: Barnacles; Bug: Bugula; E.B.: Encrusting Bryozoans; Hyd: Hydroids; Oy: Oyster; Sp: Sponge; T. W.: Tube worms; Tun: Tunicates; C.F.: Completely fouled.
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A3. FOULING CENSUS—JUNE 1971 TO JUNE 1972⁴

Test Surface:
Exposure:

8 by 10 in. Slate (Nontoxic)
From raft, 1 ft (0.3 m) beneath water surface

Date	Barnacle Count	Tube Worm Count	Oyster Count	Tunicates	Hydroids	Algae	Encrusting Bryozoa	Bugola
June 1971	209	44	7	70 %	10 %	3 %	1 %	2 %
July 1971	618	24	9	65 %	8 %	5 %	1 %	2 %
Aug. 1971	225	44	1	95 %	18 %	5 %	1 %	none
Sept. 1971	2206	5	7	55 %	3 %	5 %	none	none
Oct. 1971	1090	none	50	20 %	10 %	15 %	2 %	none
Nov. 1971	1354	2	10	5 %	3 %	7 %	none	none
Dec. 1971	983	57	none	2 %	15 %	8 %	3 %	1 %
Jan. 1972	660	157	3	5 %	60 %	35 %	6 %	2 %
Feb. 1972	176	145	none	5 %	60 %	85 %	3 %	1 %
March 1972	726	560	4	5 %	50 %	85 %	40 %	1 %
Apr. 1972	1120	560	1	10 %	40 %	92 %	25 %	1 %
May 1972	1618	150	7	10 %	30 %	20 %	1 %	none
June 1972	900	350	10	20 %	15 %	10 %	1 %	none

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A4. SUMMARY OF BARNACLE SETTLEMENT FOR EXPOSURE PERIOD OF ANNEX A2.

ANNUAL SUMMARY OF BARNACLE SETTLEMENT

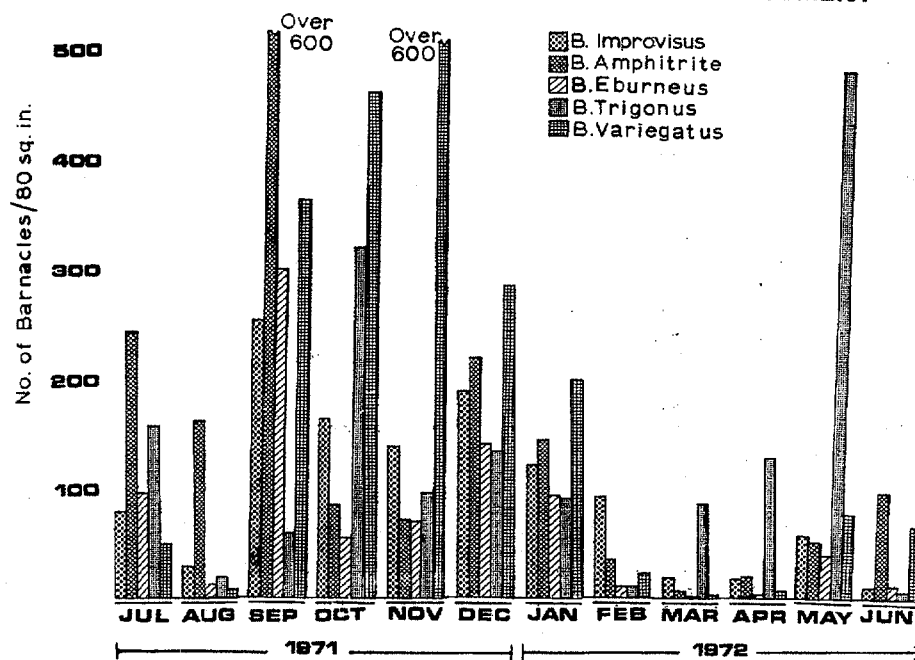


FIG. A4.1 Summary of Barnacle Settlement.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Low Concentrations of Mercury in Paint by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 3624; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This test method covers the determination of the content of mercury in the range between 10 and 1000 ppm (mg/kg) present in liquid coatings, coatings vehicles, or in dried films obtained from previously coated substrates. There is no reason to believe that higher levels could not be determined by this test method, provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7 and 9.1.1.*

2. Referenced Document

- 2.1 *ASTM Standard:*
D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The sample of liquid coating or dried film is weighed into a polytetrafluoroethylene (PTFE)-lined acid decomposition vessel and digested at an elevated temperature using sulfuric and nitric acids. Use of a sealed acid decomposition vessel prevents loss of mercury during the digestion. The digested sample is diluted to a known volume with water and the concentration of mercury is determined using a cold-vapor, atomic absorption technique.

4. Significance and Use

4.1 The permissible level of heavy metals in certain coatings is specified by governmental regulatory agencies. This test method provides a fully documented procedure for determining low concentrations of mercury present in both water and solvent-reducible coatings to determine compliance.

5. Apparatus

- 5.1 *Atomic Absorption Spectrophotometer*—Any commer-

cial instrument having an open sample presentation area in which to mount the absorption cell or an instrument designed specifically for the measurement of mercury using the cold vapor technique.

- 5.2 *Recorder*, 0 to 10 mV.

- 5.3 *Mercury Source Lamp*.

5.4 *Absorption Cell*—Standard spectrophotometer cells 100 mm long, having quartz end windows may be used. Prior to use, the cell must be positioned in the optical path of the spectrophotometer and held in place by suitable clamps or straps. The cell should be carefully aligned both vertically and horizontally to give the maximum transmittance.

5.5 *Reduction Vessel*—Cylindrical gas washing bottle, 250-mL, equipped with a coarse (40 to 60- μ m) fritted glass inlet tube and a standard-taper glass stopper. Polyethylene or poly(vinyl chloride) tubing may be used for connecting the reduction vessel to the absorption cell.

5.6 *Flowmeter*, capable of measuring a gas flow of 1 L/min.

5.7 *Drying Tube*—Approximately 6 by 3/4-in. (150 by 20-mm) glass tube filled with magnesium perchlorate. The tube should be filled each day that it is in use, and the $Mg(ClO_4)_2$ should be replaced whenever it becomes saturated (carefully observe after each analysis).

NOTE 1—Use of an indicator desiccant at the exit end of the tube will make this observation easier.

5.8 *Water Vapor Trap*—A second 250-mL gas washing bottle (the same as used for the reduction vessel). If preferred, a 250-mL Erlenmeyer vacuum flask fitted with a one-hole stopper and 200 mm of 5-mm outside diameter glass tubing, may be substituted.

5.9 *Mercury Trap*—A 250-mL Erlenmeyer vacuum flask containing 75 mL of 10 % sulfuric acid and 75 mL of 0.1 N potassium permanganate solution to absorb the mercury vapor after analysis.

- 5.10 *Circulating Oven*, maintained at $140 \pm 5^\circ\text{C}$.

5.11 *Acid Decomposition Vessel*, with 25-mL PTFE digestion cup.³

- 5.12 *Volumetric Flasks*, 100, 250, and 1000-mL.

- 5.13 *Paint Shaker*.

- 5.14 *Paint Draw-Down Bar*.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved July 17 and Nov. 29, 1985. Published January 1986. Originally published as D 3624 - 77. Last previous edition D 3624 - 84.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ Acid decomposition vessel manufactured by the Parr Instrument Co., 211 Fifty-third St., Moline, IL 61265 (Catalog No. 4745) has been found satisfactory for this purpose.

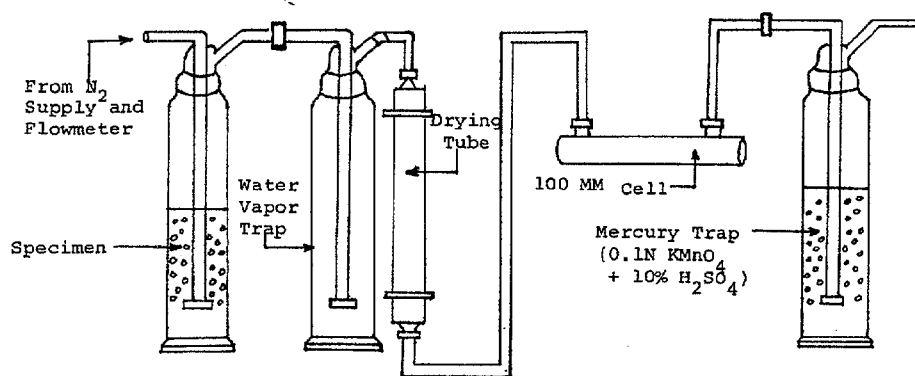


FIG. 1 Apparatus

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

6.3 *Hydroxylamine Hydrochloride Solution* (100 g/L)—Dissolve 10 g of NH₂OH·HCl in 100 mL of water. Transfer a portion of this solution to a small dropping bottle.

6.4 *Mercury Solution, Stock* (1 mg/mL)—Dissolve 0.1354 g of HgCl₂ in 50 mL of water. Carefully add 5 mL of concentrated H₂SO₄ and 3 mL of concentrated HNO₃ and dilute to 100 mL. This solution contains 1000 µg/mL of mercury.

6.5 *Mercury Standard, Working* (0.1 µg/mL)—Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 mg/L (0.1 µg/mL), maintaining a concentration of 5 % H₂SO₄ and 3 % HNO₃ by volume, in the diluted solutions. The working mercury standard and the dilutions of the stock mercury solution should be prepared fresh each day that it is used.

6.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

6.7 *Nitrogen*.

6.8 *Potassium Permanganate Solution* (0.1 N)—Dissolve 15.8 g of KMnO₄ in water and dilute to 1 L.

6.9 *Stannous Chloride Solution* (100 g/L)—Dissolve 25 g of tin (II) chloride (SnCl₂) by adding it to 60 mL of concentrated HCl (sp gr 1.19) and warming on a hotplate. When all of the SnCl₂ has dissolved, transfer to a 250-mL volumetric flask and dilute to volume with water. Mix well.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

This solution should be prepared fresh each week that it is used.

6.10 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

6.11 *Sulfuric Acid* (1+9)—Carefully mix 1 volume of H₂SO₄ (sp gr 1.84) into 9 volumes of water.

7. Hazards

7.1 Concentrated nitric and sulfuric acids are corrosive and may cause severe burns of the skin or eyes. The vapor from concentrated nitric acid is irritating to mucous membranes. Use care in handling these acidic substances. Refer to suppliers' Material Safety Data Sheet.

7.2 Mercury and its compounds are harmful and accumulate in the aquatic environment. Mixtures containing mercury compounds should not be flushed down a drain, but disposed of as hazardous waste.

7.3 Use only a rubber bulb aspirator for pipetting liquids.

8. Calibration and Standardization

8.1 Assemble the various components, as illustrated in Fig. 1 if an atomic absorption spectrophotometer is used, or prepare the instrument for operation if a commercial mercury analyzer is being used.

NOTE 2—Be sure that all glassware has been thoroughly cleaned and rinsed with reagent water prior to use.

8.2 Operational instructions for atomic absorption spectrophotometers and commercial mercury analyzers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

8.3 With the apparatus empty, stabilize the recorder base line while maintaining a flowrate of 250 mL of nitrogen per minute.

8.4 Transfer 0, 5, 10, 15, and 20-mL aliquots of the working mercury standard (containing 0 to 2 µg of mercury) to a series of 250-mL gas washing bottles. Add enough water to each bottle to make a total volume of 100 mL. Add 5 mL of H₂SO₄ and 3 mL of HNO₃ to each bottle and mix thoroughly. Make dropwise additions of 0.1 N KMnO₄ solution to each bottle in turn until a pink color persists for at least 30 s (1 mL is usually sufficient). Add exactly 1 mL of NH₂OH·HCl solution dropwise to each bottle and swirl

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8.5 Add 10 mL of SnCl_2 solution to the bottle containing no added working mercury standard. Immediately insert the fritted glass inlet tube, making sure that the stopper provides a good seal. Adjust the nitrogen flow rate to a previously determined optimum setting (see Note 3) and take a reading. A blank value greater than 5 % of full scale indicates either reagent or apparatus contamination. When the recorder, meter, or other readout device reaches a maximum value, it should be immediately zeroed. Repeat this blank determination until a steady, repeatable zero value is obtained at the maximum response.

NOTE 3—Optimum nitrogen flow rate may be determined by analyzing 1- μg quantities of mercury (10 mL of working mercury standard diluted to 100 mL) by this procedure, varying the flow rate to determine the optimum mercury signal and time required for analysis. Then use this optimum flow rate for all analyses, unless a change is made in the volume or geometry of the apparatus.

8.6 Similarly treat each standard solution made up in 8.4, recording the maximum peak height for each. When the recorder pen begins dropping, remove the fritted glass inlet tube from the gas washing bottle and insert it into another bottle containing only water. Continue nitrogen flow until the absorbance reaches zero, when the apparatus is ready for the next solution.

NOTE 4—Because mercury vapor is very toxic, take precautions to avoid its inhalation. Bubble the exhaust from the absorption cell through an absorbing solution consisting of equal volumes of 0.1 N KMnO_4 solution and 10 % H_2SO_4 (see Fig. 1). Do not discharge the absorbing solution into a sewer system.

8.7 Construct a standard curve by plotting peak heights versus micrograms of mercury. Peak heights used should be the mean of duplicate determinations on each solution.

9. Procedure

9.1 If the sample is a liquid coating, mix it until homogeneous, preferably on a mechanical paint shaker. Prepare at least two replicate specimens by weighing to 0.1 mg by difference from a dropping bottle or syringe, 100 to 200 mg of the mixed paint into the PTFE insert of the acid decomposition vessel. Place the insert in an oven set at 60°C (140°F) for 1 h to remove all volatiles.

9.1.1 **Caution**—If the solvent present in a sample is not removed prior to the digestion step as recommended, potentially dangerous pressures may result when the vessel is heated at 140°C.

9.2 If the sample consists of dried film, coarsely grind it and weigh 50 to 100 mg by difference from a weighing bottle into the digestion vessel.

NOTE 5—Recover dried paint films from previously coated substrates (being careful not to remove any underlying material from the substrate) or prepare in the laboratory from liquid samples. For the laboratory preparation, flow some of the well-mixed sample onto a clean glass plate. The use of a paint draw-down bar is recommended to obtain a uniform wet film thickness not exceeding 2 mils (50 μm). Allow the film to dry in an oven at 60°C for a minimum of 1 h. Scrape the dried film off the glass plate, preferably with a single-edge razor blade.

9.3 Pipet 5 mL of concentrated H_2SO_4 and 3 mL of concentrated HNO_3 into the decomposition vessel insert, using a fume hood for the operation. Seal the vessel tightly and place in the circulating oven at 140°C for 1½ h. Remove

the vessel from the oven and allow it to cool slowly to room temperature.

NOTE 6—Do not assist cooling by putting the vessel in a refrigerator or freezer, or by submerging it in a cooling liquid; otherwise mercury losses may occur due to leakage of pressurized gases resulting from uneven contraction of the bomb parts and PTFE seal.

NOTE 7—Analysis of the digested specimen (9.4 to 9.7) should be completed within 1 h after the digestion step.

9.4 When completely cooled, carefully unseal the vessel in a fume hood, add approximately 5 mL of cold water and swirl gently. Quantitatively transfer the contents to a 100-mL beaker, using several small quantities of water. Dilute to a volume of 30 to 40 mL and filter through filter paper directly into a 100-mL volumetric flask. Wash the residue on the filter paper several times with small volumes of water. Dilute to volume with water and mix thoroughly.

9.5 Transfer an aliquot of each digested specimen containing not over 2 μg of mercury to a 250-mL gas washing bottle. Add enough water to make a total volume of 100 mL. To each bottle, add 5 mL of concentrated H_2SO_4 and 3 mL of concentrated HNO_3 and mix thoroughly.

NOTE 8—If large aliquots are taken for analysis, reduce the amount of added H_2SO_4 and HNO_3 proportionately to compensate for the amounts present in the digested solutions.

9.6 Proceed with the addition of KMnO_4 , $\text{NH}_2\text{OH} \cdot \text{HCl}$, and SnCl_2 solutions as described in 8.4 and 8.5. Record the maximum peak height for each solution.

9.7 If the response obtained is above or below the response obtained for the standards (8.7), repeat 9.5 and 9.6 using an appropriate aliquot size. Determine the concentration of mercury present in microgram per millilitre from the calibration curve.

10. Calculation

10.1 Calculate the mean concentration of mercury in the sample as follows:

$$\text{mercury in sample, ppm (mg/kg)} = (100 \times C) / (F \times S)$$

where:

C = concentration of mercury in aliquot used, $\mu\text{g/mL}$,

F = dilution factor from 9.5 (aliquot volume used), and

S = grams of sample.

11. Report

11.1 Report the mercury content in the material being tested and whether the analysis was conducted on a liquid coating or a dried film.

12. Precision and Bias⁵

12.1 **Precision**—The precision estimates are based on an interlaboratory study in which seven different laboratories analyzed in duplicate, on two different days, four samples of water-reducible paints and four samples of solvent-reducible paints containing from 1 to 1000 ppm mercury. The within laboratory coefficient of variation was found to be 6.4 % relative at 42 degrees of freedom, and the between-laboratory coefficient of variation 10 % relative at 36 degrees of freedom. Based on these coefficients, the following criteria

⁵ Supporting data are available from ASTM Headquarters. Request RR: D01-1006.

laboratories, should be considered suspect if they differ by more than 30 % relative.

12.2 Bias—The true value for the amount of mercury present in a coating should be between 79 and 121 % of the experimental value.

12.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different

13.1 AAS, mercury; low concentration of mercury in paints

1. Scope

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Standard Guide for Determining Constituents Classified as Hazardous Contained in Protective Coatings¹

This standard is issued under the fixed designation D 3630; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide applies to liquid protective coatings, related products, and their dried films. It is a guide for the selection of analytical procedures for the determination of the presence and quantity of selected materials that may present potential physiological hazards.

1.2 Various levels of government have established laws and regulations that limit the quantity or prohibit the presence of certain materials classified as hazardous in protective coatings. Materials subject to such regulations are within the scope of this guide.

1.3 A hazardous material within the scope of this guide is one that exhibits harmful physiological effects through ingestion, inhalation, absorption, or skin or eye contact. Hazards associated with combustion are not within its scope.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 49 Methods of Chemical Analysis of Red Lead²
- D 126 Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green²
- D 215 Methods of Chemical Analysis of White Linseed Oil Paints³
- D 283 Methods of Chemical Analysis of Cuprous Oxide and Copper Pigments²
- D 444 Test Methods for Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow)²
- D 564 Test Methods for Liquid Paint Driers⁴
- D 715 Test Methods for Analysis of Barium Sulfate Pigment²
- D 1301 Test Methods for Chemical Analysis of White Lead Pigments²
- D 1844 Test Methods for Chemical Analysis of Basic Lead Silicochromate²

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.22 on Health and Safety.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 3630 - 77. Last previous edition D 3630 - 77 (1981) ^{ϵ 1}.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 06.03.

- D 1845 Test Methods for Chemical Analysis of Strontium Chromate Pigment²
- D 2348 Test Method for Arsenic in Paint³
- D 2349 Method of Qualitative Determination of Nature of Thinner in Solvent-Reducible Paints³
- D 2350 Test Method for Antimony Oxide in White Pigment Separated From Solvent-Reducible Paints²
- D 2371 Test Method for Pigment Content of Solvent-Reducible Paints³
- D 2372 Method of Separation of Vehicle From Solvent-Reducible Paints³
- D 2374 Test Method for Lead in Paint Driers by EDTA Method⁴
- D 2621 Test Method for Infrared Identification of Vehicle Solids From Solvent-Reducible Paints³
- D 2698 Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging³
- D 2742 Methods for Chemical Analysis of Tribasic Lead Phosphosilicate²
- D 3257 Test Methods for Aromatics in Mineral Spirits by Gas Chromatography⁴
- D 3271 Practice for Direct Injection of Solvent-Reducible Paints Into a Gas Chromatograph for Solvent Analysis³
- D 3272 Practice for Vacuum Distillation of Solvents From Solvent-Reducible Paints For Analysis³
- D 3280 Methods for Analysis of White Zinc Pigments²
- D 3329 Test Method for Purity of Methyl Isobutyl Ketone by Gas Chromatography⁴
- D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy³
- D 3432 Test Method for Free Toluene Diisocyanates in Urethane Prepolymers and Coating Solutions by Gas Chromatography²
- D 3618 Test Method for Detection of Lead in Paint and Dried Paint Films³
- D 3624 Test Method for Low Concentrations of Mercury in Paint by Atomic Absorption Spectroscopy³
- D 3717 Test Method for Low Concentrations of Antimony in Paint by Atomic Absorption Spectroscopy³
- D 3718 Test Method for Low Concentrations of Chromium in Paint by Atomic Absorption Spectroscopy³
- E 202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols⁵
- E 260 Practice for Packed Column Gas Chromatography⁶

⁵ Annual Book of ASTM Standards, Vol 15.05.

⁶ Annual Book of ASTM Standards, Vol 14.01.

2.2 ANSI Standard:

Z66.1 Specification for Paints and Coatings Accessible to Children to Minimize Dry Film Toxicity⁷

2.3 Federal Standards:⁸

U.S. Federal Test Method Standard No. 141:

- 4021.1 Pigment Content (Ordinary Centrifuge)
- 4032 Vehicle Isolation (Super Centrifuge)
- 7041 Analysis of Basic Carbonate White Lead Pigment
- 7051 Analysis of Basic Sulfate White Lead Pigment
- 7071 Red Lead Pigments
- 7106 Analysis of Antimony Oxide Pigment
- 7111 Analysis of Chrome Green Pigment
- 7131 Analysis of Chrome Yellow and Chrome Orange Pigments
- 7135 Analysis of Cadmium Pigment
- 7231 Metal Content of Driers
- 7271 Analysis of Pigments Extracted from Chrome Yellow and Chrome Orange Paints
- 7281 Analysis of Pigment Extracted from Chrome Green Paints

2.4 Canadian Standards Association Standards:⁹

- CAN2-1.500 Method of Test for Toxic Trace Elements in Protective Coatings:
 - 1-GP-500.1 Determination of Lead in Low Concentration
 - 1-GP-500.2 Determination of Leachable Cadmium in Low Concentration
 - 1-GP-500.3 Determination of Leachable Barium in Low Concentration
 - 1-GP-500.4 Determination of Leachable Antimony in Low Concentration
 - 1-GP-500.5 Determination of Leachable Selenium in Low Concentration
 - 1-GP-500.6 Determination of Leachable Mercury in Low Concentration

3. Summary of Guide

3.1 This guide covers each material separately, discussing the methods available, the advantages and drawbacks of each, including the range, the equipment needed, and when information is available, the precision. Methods discussed are:

- 3.1.1 ASTM methods adopted by Committee D-1,
- 3.1.2 Other ASTM methods believed to be applicable to paint and related products,
- 3.1.3 Methods adopted as standard by other scientific and technical or governmental organizations, and
- 3.1.4 Widely used methods reported in the literature.

4. Significance and Use

4.1 Protective coatings and related products containing materials classified as hazardous may be regulated or controlled in various ways as follows:

4.1.1 Precautionary Labeling Required:

4.1.1.1 Under the U.S. Federal Hazardous Substances Act and corresponding state laws, "consumer" paint products must carry precautionary labeling as specified in the laws, or implementing regulations, or both.

NOTE 1—Under the Federal Hazardous Substances Act lead, ethylene and diethylene glycol, petroleum distillates, and turpentine are classed as hazardous through ingestion. Methyl alcohol, benzene, xylene, and toluene are classed as hazardous through ingestion and inhalation. Ethylene diamine, diethylene triamine, and diglycidyl ethers are classed as strong sensitizers.

4.1.1.2 In certain states, coatings and related materials packaged and sold for industrial application are subject to precautionary labeling laws and regulations.

4.1.2 Use Prohibited in Certain Areas:

4.1.2.1 Under the U.S. Federal Hazardous Substances Act and the Lead Paint Poisoning Prevention Act, and the laws of certain states and municipalities, coatings and related products containing more than a specified quantity of lead are banned for use in and around a household or other areas where children might be exposed.

4.1.2.2 Under certain local ordinances, coatings and related products containing more than a specified quantity of "other toxic heavy metals" are also subject to control.

NOTE 2—The amount of lead, antimony, arsenic, cadmium, mercury, selenium, and soluble barium that may be present in coatings for use on children's furniture and toys and areas that might be chewed by children is restricted under ANSI Standard Z66.1 and under various U.S. municipal ordinances.

4.1.3 Subject to Approval for Certain Uses:

4.1.3.1 The U.S. Food and Drug Administration permits only approved ingredients to be used in coatings for food and beverage containers or processing equipment that comes into contact with the product.

4.1.3.2 The U.S. Department of Agriculture authorizes for use on structural surfaces in federally inspected meat and poultry processing plants only coatings accepted as nonhazardous.

4.1.3.3 The U.S. Environmental Protection Agency regulations control the distribution of paints that claim special fungicidal or pesticidal characteristics, and the use in ship-bottom paints of materials that have the potential for harming the aquatic environment.

NOTE 3—Cuprous oxide, bis(tributyltin oxide), arsenic, and mercury are classed as hazardous materials by the U.S. Environmental Protection Agency when used in antifouling paints.

4.1.4 Environmental Controls During Use and Corrective Measures in the Event of Accidents or Misuse Required:

4.1.4.1 Under U.S. Occupational Safety and Health Act regulations, airborne concentrations of certain vapors, fumes, and dusts are limited in the work-place atmosphere. Coatings, when applied, may emit solvent vapors that are subject to these limits.

4.1.4.2 Under Occupational Safety and Health Act regulations, skin or eye contact with liquid paint containing specified potentially hazardous materials must be avoided and protective apparel may be required. In the event of hazardous skin or eye contact, corrective action to ameliorate physiological injury is prescribed.

NOTE 4—Over 400 materials have been classified by the Occupational Safety and Health Administration as being potentially hazardous.

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⁷ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁸ Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

⁹ Available from Canadian Standards Association, 178 Rexdale Blvd., Rexdale, Ontario, Canada M9W 1R3.

Of this number approximately 80 may be found in varying amounts as components of coatings and related products.¹⁰

4.1.4.3 To achieve air quality standards under the Clean Air Act, regulations of various states and regions control emissions of volatile organic compounds resulting from the application of surface coatings.

5. Pigment, Vehicle Separations (Centrifugal)

5.1 The methods listed in this section cover centrifugal separation of pigment from vehicle. The pigment may then be analyzed as described in subsequent sections. More recent methods utilizing analysis of the whole paint by atomic absorption spectroscopy may be preferred due to greater speed and accuracy.

5.2 Separation of the vehicle from the pigment is generally the first step in coatings analysis. There are three ASTM methods covering the use of centrifuges:

5.2.1 Test Method D 2371 describes the use of the normal laboratory centrifuge that develops 2000 g. It should be used where a quantitative determination of the amount of pigment in the coating is required.

5.2.2 Method D 2372 covers use of a high-speed centrifuge that develops 10 000 g and is used when analysis of the vehicle is of main interest.

5.2.3 Method D 2698 covers use of a centrifuge that develops at least 32 000 g. Determination of the pigment content is the main objective of the method although the separated pigment may be solvent-extracted to remove residual vehicle.

NOTE 5—Centrifuge methods do not generally give good separations of pigments that bleed (partially dissolve) or have low densities and fine particle sizes (such as carbon black).

5.3 U.S. Federal Test Method Standard 141 contains two centrifuge methods. Federal Method 4021.1 is similar to Test Method D 2371 except that additional extraction mixtures are listed for difficult pigments and for cellulosic and vinyl lacquers. Federal Method 4032 is similar to Method D 2698 but has the same extraction solvents as in Federal Method 4021.1.

6. Solvent Separation

6.1 Practice D 3272 covers a vacuum distillation procedure that separates the solvents from the nonvolatile portion of paints so they may be analyzed.

6.2 Practice D 3271 covers analysis of the solvents by injection of the liquid coating into a gas chromatograph. Prior separation is not required.

ANALYTICAL METHODS

7. Heavy Metals Analysis, ASTM Methods

7.1 Methods for the analysis of pigments apply to dry pigment or to pigment separated from the vehicle in accordance with Section 5.

7.2 Lead:

7.2.1 *Pigment Analysis*—In all the following methods except for Methods D 2742, the concentration range for which the method is applicable is not given.

7.2.1.1 Methods D 49 cover procedures for the chemical analysis of dry red lead having the approximate formula Pb_3O_4 (probably $PbO_2 \cdot 2PbO$). Total lead is determined gravimetrically as lead sulfate.

7.2.1.2 Test Methods D 126—Total lead is determined gravimetrically as lead sulfate. The comparable Fed. Test Methods Std. 141 methods are 7271 and 7281.

7.2.1.3 Methods D 215—Two methods are described for the gravimetric determination of total lead, one as the sulfate and the other as the chromate. The choice of method may be dictated by potential interferences discussed in the text.

7.2.1.4 Test Methods D 1301 cover procedures for the chemical analysis of basic carbonate white lead and basic sulfate white lead. Total lead is determined gravimetrically as lead chromate. The comparable Fed. Test Method Std. 141 method is 7041.

7.2.1.5 Test Methods D 1844—Total lead is determined gravimetrically as lead chromate.

7.2.1.6 Methods D 2742—Silicon is removed by filtration and total lead determined gravimetrically as lead sulfate. The methods are designed for the analysis of essentially pure pigment.

7.2.1.7 Methods D 3280 cover procedures for the analysis of white zinc pigments, including leaded zinc oxide. Total lead is determined gravimetrically as lead sulfate.

7.2.2 *Analysis of Liquid Material or Dried Film:*

7.2.2.1 Test Methods D 564 cover the determination of percent lead in liquid driers (see Section 9). Total lead is determined gravimetrically as lead sulfate. The comparable Fed. Test Method Std. No. 141 method is 7231.

7.2.2.2 Test Method D 2374 covers a titrimetric determination of lead in paint driers that can be dissolved in a suitable solvent and utilizes excess EDTA (ethylene diamine tetraacetic acid) to react with lead and cupric sulfate to back titrate. The drier is dissolved in glacial acetic acid, diluted with isopropyl alcohol and water, and treated with an excess of standard EDTA solution. The excess is titrated with standard cupric sulfate solution using an indicator. The method includes a precision statement but the applicable concentration range is not given.

7.2.2.3 Test Method D 3335 covers the determination of lead contents between 0.01 and 5 % present in the solids of liquid coatings or in dried films. A specimen of liquid paint or dried film is prepared for analysis by dry ashing. The lead content of an acid extract of the ash is determined by atomic absorption spectroscopy. The precision statement is based on a relative scale.

7.2.2.4 Test Method D 3618 is intended as a screening test to determine if a sample of paint solids contains more than 0.5 % lead. The test will barely detect the presence of 0.4 % and will give a definite test at the 0.5 % level. The specimen is dry ashed and extracted. Lead present is oxidized to lead peroxide and spotted with "tetrabase" to develop a blue coloration. Higher or lower concentrations may be detected by this test method by making appropriate changes in the specimen size and reagent quantities.

7.3 Chromium:

7.3.1 *Pigment Analysis:*

7.3.1.1 *Test Methods D 126*—In these test methods the chromium content of pigments is only calculated as Cr_2O_3 for chromium oxide green. For chrome oranges, yellows, and

¹⁰ Occupational Safety and Health Reporter, Section 1910.1000 Table Z.1.

greens the chromium is calculated as lead chromate.

7.3.1.2 *Test Methods D 444* cover procedures for the chemical analysis of the pigment known commercially as "zinc yellow" or "zinc chromate yellow." Total chromium in the pigment is determined by titration using either the dichromate method or the thiosulfate method and calculated as CrO_3 .

7.3.1.3 *Test Methods D 1844*—Total chromium in the pigment is determined as chromium trioxide by titration using the thiosulfate method.

7.3.1.4 *Test Methods D 1845*—Total chromium in the pigment is determined as chromium trioxide by titration using the thiosulfate method.

7.3.2 *Analysis of Liquid Paint or Dried Film:*

7.3.2.1 *Test Method D 3718* covers the determination of chromium or chromium compounds (including chromium oxide) when present in the solids of liquid coatings or in dried films in the concentration range from 0.005 to 1.0 % (50 to 10 000 ppm) expressed as chromium. A specimen of liquid paint or dried film is prepared for analysis by dry ashing. A weighed quantity of the ash is digested with potassium permanganate and sulfuric acid. Chromium is determined on the filtered digestion mixture by atomic absorption spectroscopy.

7.4 *Barium:*

7.4.1 *Pigment Analysis:*

7.4.1.1 *Methods D 215*—Soluble barium is determined gravimetrically as the sulfate.

7.4.1.2 *Test Methods D 715*—Applicability to mixed pigments when barium is a minor component is not indicated.

7.4.1.3 *Methods D 3280* cover procedures for the analysis of white zinc pigments. Determination of total barium sulfate content is described in Section 20 of *Methods D 3280*.

7.4.2 *Analysis of Liquid Paint or Dried Film*—Current legislation restricts the quantity of "soluble" barium in coatings used in areas accessible to children, but the conditions of solubility are not usually defined. There is no ASTM method for soluble barium; see Section 10.

7.5 *Mercury:*

7.5.1 *Analysis of Liquid Paint or Dried Film*—*Test Method D 3624* covers the determination of mercury contents between 10 and 1000 ppm in liquid coatings and dried films. The specimen of whole paint, paint vehicle, or dried film is digested using sulfuric and nitric acids, and the concentration of mercury determined using cold-vapor atomic absorption.

7.6 *Arsenic:*

7.6.1 *Analysis of Liquid Paint or Dried Film*—*Test Method D 2348* covers the determination of less than 0.5 % arsenic in liquid coatings or dried films. Whole paint or dried film is digested in nitric and sulfuric acid. Sodium chloride is added and the arsenic trichloride distilled. Total arsenic is determined by titration with potassium bromate.

7.7 *Antimony:*

7.7.1 *Pigment Analysis: Test Method D 2350*—Total antimony is calculated from the antimony oxide content determined by titration with potassium permanganate. The concentration range for which the method is applicable is not given. The comparable Federal Method is 7106.

7.7.2 *Analysis of Liquid Paint or Dried Film*—*Test*

Method D 3717 covers the determination of antimony contents between 50 and 200 ppm present in the solids of liquid coatings in dried films. The specimen of liquid paint or dried film is prepared for analysis by dry ashing at 500°C followed by refluxing with hydrochloric acid and stannous chloride. The antimony content of the acid extract is determined by atomic absorption spectroscopy.

7.8 *Cadmium:*

7.8.1 *Analysis of Whole Paint or Dried Film*—*Test Method D 3335* covers the determination of cadmium contents between 50 and 150 ppm present in the solids of liquid coatings or in dried films. The sample of liquid paint or dried film is prepared for analysis by dry ashing. The content of cadmium is determined by atomic absorption spectroscopy.

8. Other Regulated Metallic Components

8.1 *Copper:*

8.1.1 *Pigment Analysis*—Methods for the analysis of the pigment portion of the coating after separation.

8.1.2 *Methods D 283* cover analysis for total copper in copper-based pigments.

9. U.S. Federal Test Method Standard No. 141

9.1 *Lead:*

9.1.1 *Method 7041* is comparable to *Test Methods D 1301*.

9.1.2 *Method 7051* is comparable to *Test Methods D 1301*.

9.1.3 *Method 7071.1* is for the quantitative measure of red lead in paint pigments.

9.1.4 *Method 7111* is comparable to *Test Methods D 126*.

9.1.5 *Method 7131* is comparable to *Test Methods D 126*.

9.1.6 *Method 7231* is comparable to *Test Methods D 564*.

9.1.7 *Method 7271* is comparable to *Test Methods D 126*.

9.1.8 *Method 7281* is comparable to *Test Methods D 126*.

9.2 *Antimony:*

9.2.1 *Method 7106* is comparable to *Test Method D 2350*.

9.3 *Cadmium:*

9.3.1 *Method 7135*.

10. Other Analytical Methods for Materials Classified as Hazardous Within the Scope of This Practice

10.1 National Standard of Canada CAN2-1.500 describes test methods for determining toxic elements in low concentrations in protective coatings, employing wet chemical and atomic absorption and visible-ultraviolet spectrophotometric techniques. Determination of leachable elements are made on a dilute hydrochloric acid extract of the dried film that has first been ground to a specific fineness.

10.1.1 1-GP-500.1 is used for determining total lead in liquid and applied coatings by atomic absorption spectroscopy.

10.1.2 1-GP-500.2 is used for determining soluble cadmium in applied coatings by atomic absorption spectroscopy.

10.1.3 1-GP-500.3 is used for determining soluble barium in applied coatings by atomic absorption spectroscopy.

10.1.4 1-GP-500.4 is used for determining soluble antimony in applied coatings by visible-ultraviolet spectroscopy.

10.1.5 1-GP-500.5 is used for determining soluble selenium in applied coatings by visible-ultraviolet spectroscopy.

10.1.6 1-GP-500.6 is used for the detection of, and the quantitative determination of, soluble mercury in applied coatings by visible-ultraviolet spectroscopy.

11. Nonvolatile Vehicle Components, ASTM Methods

11.1 Test Method D 2621 covers the qualitative characterization or identification of separated paint vehicle solids by infrared spectroscopy within the limitations of this procedure.

12. Volatile Components

12.1 The volatile components of protective coatings must be identified to determine compliance with regulations covering labeling, work place concentrations, and air quality standards.

12.2 Gas chromatography is widely used for this purpose. However, owing to the empirical nature of the technique, chemical or instrumental confirmatory tests may be necessary as supplementary procedures.

12.3 Identification of the components of an unknown solvent blend would in many cases be expedited by means of a combination of gas chromatography and infrared spectroscopy or mass spectrometry.

12.4 Interconnected chromatographic, infrared analyzers are available that separate multicomponent mixtures into their pure components for infrared analysis.

12.5 Analysis of the volatile component of whole paint has been covered in Section 6.

12.6 The following ASTM methods cover the analysis of some of the volatile components of protective coatings currently subject to regulation as potentially hazardous materials:

12.6.1 Method D 2349 covers the determination of the nature of thinner in solvent-reducible house paints containing only hydrocarbon solvents.

12.6.2 Test Method D 3432 covers low molecular weight urethane products containing (a) 0.1 to 1.0 % and (b) 1.0 to 10 % of free toluene diisocyanate (TDI) content.

12.6.3 Test Methods D 3257 cover the determination of ethylbenzene and total eight-carbon (C_8) and heavier aromatics in the concentration range from 0.1 to 30 % in mineral spirits having a distillation range from 300 to 410°F (150 to 210°C). Oxygenated compounds, if present, may interfere and cause erroneous results.

12.6.4 Method D 3329 covers the determination of the purity of methyl isobutyl ketone by gas chromatography.

12.6.5 Test Methods E 202—See Sections 26 to 35 (Gas Chromatographic Techniques).

12.6.6 Practice E 260.

13. Keywords

13.1 antimony; aromatics; barium; cadmium; Canadian methods; chromium; copper; government regulations; hazardous substances; heavy metals; lead; mercury; pigments; selenium; solvents; zinc

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Low Concentrations of Antimony in Paint by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 3717; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This test method covers the determination of the content of antimony in the range between 50 and 200 ppm (mg/kg) present in the solids of liquid coatings or in dried films obtained from previously coated substrates. There is no reason to believe that higher levels could not be determined by this test method, provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings³

3. Summary of Test Method

3.1 The sample of liquid coating or dried film is prepared for analysis by dry ashing at 500°C, followed by refluxing with hydrochloric acid and stannous chloride. The antimony content of the acid extract is determined by atomic absorption spectroscopy.

4. Significance and Use

4.1 The permissible level of heavy metals in certain coatings is specified by governmental regulatory agencies. This test method provides a fully documented procedure for determining low concentrations of antimony present in both water- and solvent-reducible coatings to determine compliance.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of

an atomizer and either a single- or three-slot burner; gas pressure regulating and metering devices for air and acetylene; an antimony source lamp with a regulated constant current supply; a monochromator and associated optics; a photosensitive detector connected to an electronic amplifier, and a readout device.

5.2 *Muffle Furnace*, maintained at $500 \pm 10^\circ\text{C}$.

5.3 *Oven*, maintained at $105 \pm 2^\circ\text{C}$.

5.4 *Hot-Plate*, with variable surface temperature control over the range from 70 to 200°C.

5.5 *Reflux Condenser*, water-cooled, and fitted with a standard-taper joint.

5.6 *Erlenmeyer Flask*, 125-mL, with standard-taper joint to fit condenser.

5.7 *Volumetric Flasks*, 100 and 1000-mL.

5.8 *Dropping Bottles*, $\frac{1}{4}$ or $\frac{1}{2}$ -oz (8 or 15-mL) capacity.

5.9 *Glass or Disposable Syringes*, 10-mL capacity.

5.10 *Pipets*, 1, 5, 10, and 15-mL capacity.

5.11 *Filter Paper*, ashless, medium or slow filtering.

5.12 *Paint Shaker*.

5.13 *Paint Draw-Down Bar*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water conforming to Type II of Specification D 1193.

6.3 *Antimony Standard Stock Solution* (0.1 mg/mL)—Dissolve 0.1000 g of antimony metal in 40 mL of sulfuric acid (H_2SO_4 , sp gr 1.84) by heating. Cool, carefully transfer quantitatively to a 1-L volumetric flask already approximately half full with water, allow to cool to room temperature, and dilute to 1 L.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 31 and Nov. 29, 1985. Published January 1986. Originally published as D 3717 - 78. Last previous edition D 3717 - 84.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

6.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

6.5 *Stannous Chloride Solution* (400 g/L in HCl)—Dissolve 40 g of tin (II) chloride (SnCl_2) in 50 mL of HCl (sp gr 1.19). Transfer to a 100-mL volumetric flask and fill to the mark with concentrated HCl.

7. Hazards

7.1 Use care in handling concentrated HCl because it is corrosive and may cause burns to the skin and eyes and its vapor is irritating to mucous membranes. Refer to suppliers' Material Safety Data Sheet.

7.2 Use only a rubber bulb aspirator for pipetting liquids.

8. Calibration and Standardization

8.1 Prepare 100-mL quantities of at least four standard solutions bracketing the expected antimony concentration in the sample to be tested. To suitable aliquots of the 0.1 mg/mL standard antimony solution, add 40 mL of concentrated HCl and 8 drops of SnCl_2 solution. Dilute to 100 mL with water. Prepare a blank solution by diluting 40 mL of concentrated HCl and 8 drops of SnCl_2 solution to 100 mL with water.

8.2 Operational instructions for atomic absorption spectrophotometers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

8.3 Turn the instrument on and set the wavelength to the 217.6-nm antimony line. Apply the recommended current to the antimony source lamp. Allow the instrument to warm up for about 15 min and set the slit width. Adjust the air and acetylene pressures and ignite the burner in accordance with instructions.

8.4 Aspirate water to rinse the atomizer chamber. Aspirate a standard solution and make any necessary readjustment in instrument parameters to obtain maximum absorption.

8.5 Aspirate the blank solution. When the recorder, meter, or other readout device reaches a constant value, it should be immediately zeroed. Repeat this blank determination until a steady, repeatable zero value is obtained at the maximum response.

8.6 Aspirate each of the appropriate standard solutions in ascending antimony concentrations and record the corresponding instrument readings. Aspirate water between each standard.

NOTE 1—A deuterium background corrector, if available, should be used to correct for background absorption. If not available, it is necessary to reaspirate each standard solution and measure the background at a nearby nonabsorbing region of the spectrum.

NOTE 2—Because of the high concentration and corrosive nature of the acids used for the analysis, it is essential that the burner assembly be disassembled and thoroughly cleaned out immediately after the analyses are completed. Consult the instrument manual for cleaning instructions.

8.7 Construct a calibration curve on linear graph paper by plotting the absorbance (corrected for background) versus concentration (micrograms per millilitre) for each standard solution.

9. Procedure

9.1 If the sample is a liquid coating, mix it until homogeneous, preferably on a mechanical paint shaker. Determine the nonvolatile content in accordance with Guide D 2832.

9.2 Prepare at least two replicate samples by weighing by difference from a dropping bottle or syringe, 5 to 10 g of the mixed liquid coating, or by directly weighing approximately 2 to 6 g of dried film, into 125-mL Erlenmeyer flasks. Weigh to the nearest 0.1 mg.

NOTE 3—The specimen size called for will have a concentration of approximately 4 to 12 $\mu\text{g/mL}$ antimony in the final diluted solution for paints containing approximately 200 ppm (mg/kg) antimony based on the nonvolatile content, and 1 to 3 $\mu\text{g/mL}$ for paints containing approximately 50 ppm antimony, based on the nonvolatile content.

NOTE 4—Recover dried paint films from previously coated substrates (being careful not to remove any underlying material from the substrate) or prepare in the laboratory from liquid samples. For the laboratory preparation, flow some of the well-mixed sample onto a clean glass plate. The use of a paint draw-down bar is recommended to obtain a uniform film thickness not exceeding 2 mils (50 μm). Allow the film to dry in an oven at 105°C for a minimum of 1 h. Scrape the dried film off the glass plate, preferably with a single-edge razor blade.

9.3 Place the flasks containing the liquid coating on the hot plate and slowly increase the temperature until the material is dried.

9.4 When the sample appears to be dry, or when starting with a dried film, gradually increase the temperature of the hot plate until the material chars.

9.5 After charring appears complete, place the flasks in the muffle furnace and ash at 500°C. When the ashing appears to be complete (approximately 1 to 2 h), remove the flasks from the muffle furnace and allow to cool to room temperature.

9.6 Carefully add 40 mL of concentrated HCl and 8 drops of SnCl_2 solution to each flask. Connect the flasks to water-cooled condensers and reflux for 1 h.

9.7 Filter each digested specimen through medium-porosity filter paper into a 100-mL volumetric flask. If the filtrate is not clear, refilter through fine-porosity filter paper. Rinse each flask several times with water, adding the rinsings to the filter paper. Wash the filter paper several times with water. Adjust the volume to 100 mL with water and mix.

9.8 Aspirate each test solution and determine the absorbance in the same manner in which the instrument was calibrated (see Notes 1 and 2). Determine the concentration of antimony in micrograms per millilitre from the calibration curve. If the absorbance is above the range covered by the calibration curve, dilute an aliquot of the test solution to a suitable volume with water containing 40 mL of HCl and 8 drops of SnCl_2 solution per 100 mL. If the absorbance is below the range covered by the calibration curve, repeat the analysis using a larger specimen size.

NOTE 5—Increased sensitivity may be obtained with instruments possessing scale expansion and concentration readout capability.

NOTE 6—The method of standard additions may be used to improve the accuracy of the analysis. This method is particularly recommended for use with unknown samples where matrix effects may be potentially significant. For a detailed description of the procedure and calculations used in the method of standard additions, consult a standard text on atomic absorption spectroscopy or the instruction manual provided by the instrument manufacturer.

10. Calculation

10.1 Calculate the mean concentration of antimony in the nonvolatile portion of the sample as follows:

$$\text{Antimony, ppm (mg/kg) in nonvolatile} = (C \times F \times 10^4) / (NV \times S)$$

where:

- C = concentration of antimony in the aspirated test solution, $\mu\text{g/mL}$,
 F = dilution factor from 9.8 (volume diluted to/volume of aliquot),
 NV = percent nonvolatile of paint sample (use 100 if sample was dried film), and
 S = grams of sample.

11. Report

11.1 Report the antimony content of the nonvolatile content of the sample and whether the analysis was conducted on a liquid coating or a dried film.

⁵ Supporting data are available from ASTM Headquarters. Request RR: D01-1011.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

12. Precision and Bias⁵

12.1 The precision estimates are based on an inter-laboratory study in which eight different laboratories analyzed in duplicate, on two different days, three samples of water-reducible paints and three samples of solvent-reducible paints containing from 50 to 200 ppm (mg/kg) antimony. The within-laboratory coefficient of variation was found to be 4.8 % relative at 31 degrees of freedom and the between-laboratory coefficient of variation was 8.8 % relative at 25 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days, should be considered suspect if they differ by more than 14 % relative.

12.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories, should be considered suspect if they differ by more than 26 % relative.

12.2 *Bias*—The mean of duplicate determinations within any laboratory should be within 18 % relative of true value.

13. Keywords

13.1 AAS, antimony; low concentration of antimony in paints

1. Scope

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Standard Test Method for Low Concentrations of Chromium in Paint by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 3718; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This test method covers the determination of the content of chromium (including chromium oxide) in the range between 0.005 and 1.0 % present in the solids of liquid coatings or in dried films obtained from previously coated substrates. There is no reason to believe that higher levels could not be determined by this test method, provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²
D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings³

3. Summary of Test Method

3.1 The sample of liquid coating or dried film is prepared for analysis by dry ashing at 500°C followed by digestion with potassium permanganate and sulfuric acid in a polytetrafluoroethylene (PTFE)-lined acid decomposition vessel at an elevated temperature. The chromium in the filtered digestion mixture is determined by atomic absorption spectroscopy.

4. Significance and Use

4.1 The permissible level of heavy metals in certain coatings is specified by governmental regulatory agencies. This test method provides a fully documented procedure for determining low concentrations of chromium present in both water- and solvent-reducible coatings to determine compliance.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of an atomizer and either a single- or three-slot burner; gas pressure-regulating and metering devices for nitrous oxide (N_2O) and acetylene; a chromium hollow cathode lamp with a regulated constant current supply; a monochromator and associated optics; a photosensitive detector connected to an electronic amplifier; and a readout device.

5.2 *Muffle Furnace*, maintained at $500 \pm 10^\circ C$.

5.3 *Force-Draft Oven*, maintained at $105 \pm 2^\circ C$.

5.4 *Acid Decomposition Vessel*, with PTFE digestion cup.⁴

5.5 *Hot Plate*, with variable surface temperature control over the range from 70 to 200°C.

5.6 *Volumetric Flasks*, 50, 100 and 1000-mL.

5.7 *Pipets*, 5, 10, 15, and 20-mL capacity.

5.8 *Filter Paper*, ashless, medium filtering 15-cm.

5.9 *Paint Shaker*.

5.10 *Crucibles*, wide form, porcelain, approximately 30-mL capacity.⁵

5.11 *Mortar and Pestle*.

5.12 *Paint Draw-Down Bar*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

6.3 *Chromium Standard Stock Solution* (1 mg/mL)—

⁴ Acid decomposition cup manufactured by the Parr Instrument Co., 211 Fifty-third St., Moline, IL 61265 (Catalog Number 4745) has been found satisfactory for this purpose.

⁵ Coors No. 25007 crucibles, or equivalent, have been found satisfactory for this purpose.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 31 and Nov. 29, 1985. Published January 1986. Originally published as D 3718 - 78. Last previous edition D 3718 - 84.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

Dissolve 3.735 g of potassium chromate (K_2CrO_4) in 100 mL of water and dilute to 1 L.

6.4 *Chromium Standard Working Solution* (0.1 mg/mL)—Pipet 10 mL of the chromium standard stock solution into a 100-mL volumetric flask and dilute to volume with water.

6.5 *Oxidizing Solution*—Dissolve 0.2 g of potassium permanganate ($KMnO_4$) in 100 mL of H_2SO_4 (1+1). Stir until completely dissolved. The color of this solution is dark brown.

6.6 *Reducing Solution*—Dissolve 1 g of hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) in 100 mL of water.

6.7 *Reagent Blank*—Pipet 5 mL of oxidizing solution into 25 mL of water contained in a 50-mL volumetric flask. Add reducing solution dropwise until the permanganate color has been discharged; then dilute to 50 mL with water.

6.8 *Sulfuric Acid* (1+1)—Carefully add 1 volume of concentrated H_2SO_4 (sp gr 1.84) to 1 volume of water.

6.9 *Sulfuric Acid* (5 % volume per volume)—Carefully add 50 mL of concentrated H_2SO_4 (sp gr 1.84) to 500 mL of water and dilute to 1 L.

7. Hazards

7.1 Use care in handling concentrated H_2SO_4 because it is corrosive and may cause severe burns of the skin or eyes. Refer to suppliers' Material Safety Data Sheet.

7.2 The National Institute for Occupational Safety and Health has stated that hexavalent chromium compounds are hazardous to health. Care should be exercised in preparation of a sample for test. The wearing of a respirator and rubber or synthetic gloves is recommended. In case of contact, wash thoroughly with soap and water. Mixtures containing hexavalent chromium compounds should not be flushed down a drain but disposed of as hazardous waste.

7.3 Use only a rubber bulb aspirator for pipetting liquids.

8. Calibration and Standardization

8.1 Prepare 50-mL quantities of at least four standard solutions bracketing the expected chromium concentration in the sample to be tested. To suitable aliquots of the 100-ppm chromium standard working solution, add 10 mL of the oxidizing solution with a pipet, followed by the dropwise addition of reducing solution until the permanganate color has been discharged; then dilute to 50 mL with water.

8.2 Operational instructions for atomic absorption spectrophotometers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

8.3 Turn the instrument on and set the wavelength to the 357.9-nm chromium line. Apply the recommended current to the chromium hollow-cathode lamp. Allow the instrument to warm up for about 15 min and set the slit width. Adjust the nitrous oxide and acetylene pressures and ignite the burner according to instructions.

8.4 Aspirate water to rinse the atomizer chamber. Aspirate a standard solution and make any necessary readjustment in instrument parameters to obtain maximum absorption. Zero the instrument while aspirating reagent blank solution (6.7). Aspirate each of the appropriate standard solutions and record the corresponding instrument readings.

Aspirate water between each standard.

8.5 Transfer a 25-mL aliquot from each of the standard solutions prepared in 8.1 to 50-mL volumetric flasks and dilute to volume with water. Repeat the steps outlined in 8.4 for the diluted aliquot solutions.

8.6 Construct a calibration curve on linear graph paper by plotting the absorbance versus concentration (micrograms per millilitre) for each set of standard solutions.

NOTE 1—To obtain maximum accuracy one should complete calibration and standardization just prior to sample analysis.

9. Procedure

9.1 If the sample is a liquid coating, mix it until homogeneous, preferably on a mechanical paint shaker. Determine the nonvolatile content in accordance with Guide D 2832.

9.2 Determine the ash content of the material under test in duplicate.

9.2.1 Weigh to the nearest 0.1 mg approximately 5 g of liquid coating or 3 g of dried film into each of two tared porcelain crucibles.

NOTE 2—Recover dried paint films from previously coated substrates (being careful not to remove any underlying material from the substrate) or prepare in the laboratory from liquid samples. For the laboratory preparation, flow some of the well-mixed sample onto a clean glass plate. The use of a paint draw-down bar is recommended to obtain a uniform film thickness not exceeding 2 mils (50 μm). Allow the film to dry in an oven at 105°C for a minimum of 1 h. Scrape the dried film off the glass plate, preferably with a single-edge razor blade.

9.2.2 Place the crucibles containing the liquid coating on the hot plate and slowly increase the temperature until the material is dried. With some types of coatings, an initial oven-drying at 105°C may be necessary to remove solvents without incurring losses due to spattering.

9.2.3 When the specimen appears to be dry, or when starting with a dried film, gradually increase the temperature of the hot plate until the material chars.

9.2.4 After charring is complete, place the crucibles in the muffle furnace and ash at 500°C.

9.2.5 When the ashing appears to be complete (approximately 1 to 2 h), remove the crucibles from the muffle furnace and allow to cool to room temperature in a desiccator. Weigh and calculate the mean value for the percent ash.

9.3 Transfer each ash to a clean, dry mortar and grind to a fine, uniform powder. Weigh to the nearest 0.1 mg, 0.02 to 0.2 g of each ash directly into separate decomposition vessel inserts.

NOTE 3—For a material containing 50 ppm (mg/kg) of chromium based on the solids and having 50 % ash and 70 % nonvolatile material, 0.2 g of ash extracted and diluted to 50 mL will contain 0.28 μg of chromium per millilitre, and for one containing 10 000 ppm, 0.02 g of ash extracted and diluted to 100 mL will contain 2.8 μg of chromium per millilitre. To ensure complete recovery of chromium, do not use more than 0.05 g of ash for materials that are known or suspected to contain over 1000 ppm of chromium based on the solids.

9.4 Pipet exactly 10 mL of oxidizing solution into the cup. Swirl gently to facilitate mixing. Place the vessel in a circulating oven at 105°C for 1½ h.

9.5 Remove the vessel from the oven and allow to cool slowly to ambient temperature. Do not cool by placing the vessel in a refrigerator or freezer, or by submerging it in a

cooling liquid. When cooled, carefully unseal the vessel in a fume hood.

NOTE 4—When the vessel is disassembled, if the solution does not show the presence of excess permanganate, repeat the digestion using a smaller weight of ash.

9.6 Quantitatively filter the contents through filter paper directly into a 50 or 100-mL volumetric flask. Rinse the digestion cup several times with water, transferring any insoluble material to the filter paper. Wash the filter paper several times with small volumes of water. Add hydroxylamine hydrochloride solution dropwise to the filtrate until the permanganate color has been discharged, then dilute to volume with water.

9.7 Aspirate each test solution and determine the absorbance in the same manner in which the instrument was calibrated. Determine the concentration of chromium in micrograms per millilitre from the appropriate calibration curve. If the absorbance is above the range covered by the calibration curve, dilute an aliquot of the test solutions to a suitable volume with H_2SO_4 (5 % volume per volume) and repeat the determinations.

NOTE 5—The method of standard additions may be used to improve the accuracy of the analysis. This test method is particularly recommended for use with unknown samples where matrix effects may be potentially significant. For a detailed description of the procedure and calculations used in the method of standard additions, consult a standard text on atomic absorption spectroscopy or the instruction manual provided by the instrument manufacturer.

10. Calculation

10.1 Calculate the mean concentration of chromium in the nonvolatile portion of the sample as follows:

$$\text{Chromium, ppm (mg/kg) in nonvolatile} = (C \times F \times A \times V) / (S \times NV)$$

where:

- C = concentration of chromium in the aspirated test solution, $\mu\text{g/mL}$,
 F = dilution factor from 9.7 (volume diluted to volume of aliquot),

- A = mean percent ash as determined in 9.2.5,
 V = volume diluted to in 9.6 (50 or 100 mL),
 S = weight of ash, and
 NV = percent nonvolatile of paint sample (use 100 if sample was a dried film).

11. Report

11.1 Report the chromium content of the nonvolatile portion of the sample tested and whether the analysis was conducted on a liquid coating or a dried film.

12. Precision and Bias⁷

12.1 The precision estimates are based on an inter-laboratory study in which six different laboratories analyzed in duplicate, on two different days, four samples of water-reducible paints and four samples of solvent-reducible paints containing from 50 to 10 000 ppm (mg/kg) chromium. The within-laboratory coefficient of variation was found to be 4.9 % relative at 39 degrees of freedom and the between-laboratory coefficient of variation was 8.5 % relative at 31 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 14 % relative.

12.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 24 % relative.

12.2 *Bias*—The mean of duplicate determinations within any laboratory should be within 17 % relative of true value.

13. Keywords

13.1 AAS, chromium; low concentration of antimony in paints

⁷ Supporting data are available from ASTM Headquarters. Request RR: D01-1012.

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Standard Test Method for Quantifying Dirt Collection on Coated Exterior Panels¹

This standard is issued under the fixed designation D 3719; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers an instrumental procedure for quantifying the degree of dirt collection on exposed exterior coated panels.

1.2 This test method is limited to those coated exterior panels with lightness values before exposure greater than that of dirt, that is, above a value of 52.

1.3 The instrumental readings do not distinguish between dirt collection and mildew growth.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²

D 3274 Test Method for Evaluating Degree of Surface Disfigurement of Paint Films by Microbial (Fungal or Algal) Growth or Soil and Dirt Accumulation²

3. Definition

3.1 *dirt collection*—the deposit of foreign matter consisting of dirt, soot, or stain present on the surface of exposed exterior coated panels.

3.1.1 This test method measures total dirt collection. Loose dirt may be washed and removed from specimens if only imbedded dirt collection is to be measured.

4. Summary of Test Method

4.1 Lightness readings using a color difference meter are made before and after exposure, and the difference is considered to be due to dirt collection.

5. Significance and Use

5.1 Change in color or appearance of coated exterior surfaces due to such factors as dirt collection or retention is undesirable. If Test Method D 3274 or other test methods

are used to establish that dirt collection is the primary cause of such a change, this test method can be used to quantify the degree of dirt collection or, more properly, the degree of change in visual appearance of the coated surface due to dirt collection.

5.2 When used in this manner and with color change, exposure duration and environment specified, this test method can provide a performance specification with respect to dirt collection.

6. Apparatus and Materials

6.1 *Color Difference Meter*, complying with Method D 2244.

6.2 *Test Panels*, of any convenient size and of substrate suitable for exterior use.

7. Preparation of Test Panels

7.1 Apply the material under test to the test panels in accordance with Test Method D 823, or as agreed upon between the purchaser and the seller. Allow the final coat of air drying systems to cure for 7 days before exposure.

8. Procedure

8.1 Operate the color difference meter in accordance with the manufacturer's instructions. Standardize with either a primary or working standard as defined in Test Method D 2244. Restandardize at sufficient intervals, in accordance with the manufacturer's instructions, to minimize the effects of instrumental drift.

8.2 Determine the metric lightness, L^* , in the CIE 1976 $L^*a^*b^*$ Uniform Color Space (CIE LAB), for each unexposed panel at three marked, random locations. Calculate the arithmetic mean, L^*A .

8.3. Expose the panels at a 45° angle to the horizontal facing the equator at an exterior location for 61 days.

8.3.1 The calendar time, location, angle of exposure, orientation, exposure time, and method of exposure may be modified by mutual agreement.

8.4 Remove the panels and determine, using the procedure outlined above, the percent light reflectance value, L^* , for the exposed panels. Calculate the arithmetic mean of the marked locations, L^*B .

9. Calculations

9.1 Compute the dirt collection index, D_c , as follows:

where:
 L^*A = ar
 L^*B = ar

10. Report

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10.1.1 value, and as well as exposed p value.

10.1.2 other than

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.52 on Factory-Coated Wood Products.

Current edition approved Oct. 30, 1987. Published December 1987. Originally published as D 3719 - 78. Last previous edition D 3719 - 80.

² Annual Book of ASTM Standards, Vol 06.01.

$$D_c = \frac{L^*B}{L^*A} \times 100$$

where:

L^*A = arithmetic mean of unexposed panel values, and
 L^*B = arithmetic mean of exposed panel values.

10. Report

10.1 Report the following information:

10.1.1 Arithmetic mean and range of each panel's L_x value, and whether fungal growth is included in the reading, as well as the calculated dirt collection index. Report if the exposed panel was washed before taking the light reflectance value.

10.1.2 Any noticeable color change which, for reasons other than dirt accumulation, may have occurred. The dirt

collection index is not valid if these color changes or fungal growth are significant.

10.1.3 Description of the method of panel preparation, including substrate identity, coating types, number of coatings used, methods of application, coverage, and drying conditions.

10.1.4 Description of the exposure conditions including location and calendar time of exposure period.

10.1.5 Identification of the color difference meter by manufacturer's name and code number.

10.1.6 Which Adams color scale was used.

11. Precision and Bias

11.1 *Precision*—Precision of the dirt collection index has not been determined but the precision of the lightness values used in its calculation has been defined.

11.2 *Bias*—Bias cannot be determined.

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Standard Test Method for Pigment Content of Water-Emulsion Paints by Low- Temperature Ashing¹

This standard is issued under the fixed designation D 3723; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Section 9 was added editorially in May 1990.

1. Scope

1.1 This test method covers a procedure for the pigment content determination of water-based paints. It is applicable only to pigments that do not decompose or lose weight at temperatures below 500°C. Such pigments include most metal oxides, silicates, and a majority of anhydrous inorganic salts.

1.2 Many water-based paints contain pigments and organic colorants that lose water of hydration or decompose at this temperature. The residual ash should be carefully inspected for changes in color or texture that could indicate a pigment alteration and hence lead to erroneous results. Caution should therefore be exercised when applying this test method to samples containing unknown pigment compositions.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

- 2.1 *ASTM Standard:*
D1193 Specification for Reagent Water²

3. Significance and Use

3.1 This test method is used by paint producers and consumers for product process control and for product acceptance.

4. Apparatus

- 4.1 *Oven*, forced draft, maintained at $105 \pm 2^\circ\text{C}$.
4.2 *Furnace*, muffle, maintained at $450 \pm 25^\circ\text{C}$.
4.3 *Syringe*, 5-mL.
4.4 *Aluminum Foil Dish*, 58 mm in diameter by 18 mm high with a flat bottom. The bottom of the dish should be as nearly flat as possible so that a uniform film is produced.

5. Reagents

- 5.1 *Purity of Reagents*—Reagent grade chemicals shall be

used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type III of Specification D 1193.

5.3 *Ammonium Hydroxide*⁴—Add 1 volume of concentrated NH_4OH (sp gr 0.90) to 3 volumes of water.

6. Procedure

6.1 Mix samples until homogeneous, preferably on a mechanical shaker. If air bubbles become entrapped in a sample, stir it by hand.

6.2 Draw approximately 1.5 g of the test paint into a 5-mL syringe and weigh to 1 mg. Add the paint dropwise (about 30 drops) into a tared-aluminum foil dish that contains 2 mL of water (5.2). Swirl the dish during the addition and continue the swirling until the specimen is completely dispersed. Reweigh the syringe to 1 mg. Transfer between 0.4 and 0.6 g of sample to the dish. If not, adjust the transferred volume and prepare a new specimen. If the specimen agglomerates or forms a lump that cannot be dispersed, a drop or two of ammonia (5.3) may facilitate the dispersement. If the lumping persists, discard the specimen and prepare a new one. Prepare a duplicate specimen in the same manner.

6.3 Dry the specimen and dishes in the $105 \pm 2^\circ\text{C}$ oven for a minimum of 1 h after making certain that the dishes are level. If the coating does not uniformly cover the bottom of a dish, prepare a new specimen and repeat.

NOTE—The percent nonvolatile content, N , at 105°C may be determined by drying dishes to constant weight and calculating as follows:

$$N = \frac{A - B}{S} \times 100 \quad (1)$$

where:

A = weight of dish and specimen after heating,

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Oct. 26, 1984. Published January 1985. Originally published as D 3723 - 78. Last previous edition D 3723 - 78.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

⁴ For guidance in the safe handling of ammonium hydroxide consult the Manufacturing Chemists Association's Chemical Safety Data Sheet.

B = weight of dish alone, and
 S = specimen weight.

6.4 Transfer the dishes to a muffle furnace and heat at $450 \pm 25^\circ\text{C}$ for 1 h. Remove from the furnace, cool in a desiccator and weigh.

7. Calculation

7.1 Calculate the percent pigment content, P , as follows:

$$P = \frac{C - B}{S} \times 100 \quad (2)$$

where:

C = weight of the dish and specimen after ignition in the furnace,

B = weight of the dish alone, and

S = specimen weight.

8. Precision and Bias⁵

8.1 *Precision: Pigment Content*—On the basis of an interlaboratory test of this test method in which eighteen operators in nine laboratories analyzed three materials containing three pigment levels between 24 and 28 %, the within-laboratory standard deviation was found to be 0.24 % absolute and the between-laboratories 0.38 % absolute. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.66 % absolute.

8.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 1.05 % absolute.

8.2 *Bias: Pigment Content*—On the basis of the same study, the mean of two determinations should be within 5 % relative of the formula pigment content of a waterborne paint containing only inorganic pigments.

8.3 *Precision: Nonvolatile Content*—On the basis of an interlaboratory test of this test method in which fourteen operators in seven laboratories analyzed three materials containing three nonvolatile levels between 43 and 48 %, the within-laboratory standard deviation was found to be 0.18 % absolute and the between-laboratories standard deviation was found to be 0.445 % absolute. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.3.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.49 % absolute.

8.3.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 1.23 % absolute.

9. Keywords

9.1 low-temperature ashing; pigment content; water emulsion paints

⁵ Supporting data are available from ASTM Headquarters. Request RR:D01.1014.

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Standard Guide for Testing High-Performance Interior Architectural Wall Coatings¹

This standard is issued under the fixed designation D 3730; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Editorial changes were made throughout, including the title, in October 1988.

1. Scope

1.1 This guide covers the selection and use of test methods for high-performance interior architectural wall coatings (HIPAC) that differ from more conventional coatings in that they are tougher, more stain-resistant, more abrasion-resistant and, ordinarily, designed to be applied to wall surfaces of steel, masonry (poured concrete, concrete block, or cinder block), and plaster or gypsum wallboard. The tests that are listed in Tables 1 and 2 are designed to measure performance properties. These tests may not all be required for each HIPAC system. Selection of the test methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.

1.2 High-performance architectural coatings are tough, extra-durable organic coating systems that are applied as a continuous (seamless) film and that cure to a hard finish. The finish can be high gloss, semigloss, or low gloss as desired. These coatings are resistant to persistent heat, humidity, abrasion, staining, chemicals, and fungus growth. They are used in areas where humidity, wear, or unusual chemical resistance requirements, particularly to soiling, are required and where strong detergents are used to maintain sanitary conditions. Halls and stairways in public buildings, lavatories, stall showers, locker areas, animal pens, and biological laboratories are typical applications. In addition, food processing plants, dairies, restaurants, schools, and transport terminals frequently use HIPAC systems. These are effective in many areas of building interiors compared with tile and are of low materials and maintenance costs. HIPAC systems should be used as a complete system only as recommended by the manufacturer since the individual coats in a system are formulated to be compatible with each other. HIPAC systems should be applied only to properly prepared surfaces such as steel and masonry that includes cinder blocks and cement blocks. They can be applied over plaster and gypsum wallboard. Ordinarily, a prime or fill coat, if required, is part of the system.

1.3 While they are excellent for walls, HIPAC are not usually intended for ceilings and floors. They would not ordinarily be used in homes, although parents with small children might want to use HIPAC coatings on some walls.

1.4 The types or resin ordinarily used are the following: epoxy-polyamide, two-package; polyester-epoxy type, two-package; polyurethane, one-package or two-package. However, other resin types are not excluded provided they can meet the requirements (performance specifications) laid down by the purchaser.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see the Note in 7.5.

2. Referenced Documents

2.1 ASTM Standards:

- C 220 Specification for Flat Asbestos-Cement Sheets²
- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products³
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester⁴
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints⁵
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁶
- D 523 Test Method for Specular Gloss⁶
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁶
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁶
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁶
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁶
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁶
- D 1211 Test Method for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood⁶

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials, and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Dec. 21, 1978. Published January 1979.

² Annual Book of ASTM Standards, Vol 04.05.

³ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

⁴ Annual Book of ASTM Standards, Vols 05.01 and 06.03.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁶ Annual Book of ASTM Standards, Vol 06.01.

- D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings⁶
- D 1296 Test Method for Odor of Volatile Solvents and Diluents⁷
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁶
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁶
- D 1644 Test Methods for Nonvolatile Content of Varnishes⁸
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁸
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁶
- D 2247 Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity⁶
- D 2615 Test Method for Free Toluene Diisocyanate in Urethane Polymers by Distillation⁹
- D 2794 Test Method for Resistance of Organic Coatings to Effects of Rapid Deformation (Impact)⁶
- D 2801 Test Method for Leveling Characteristics of Paint by Draw-Down Method¹⁰
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁶
- D 3023 Practice for Determination of Resistance of Factory-Applied Coatings on Wood Products to Stains and Reagents⁶
- D 3273 Test Method for Resistance to Growth of Mold on the Surface of Interior Coatings in an Environmental Chamber⁶
- E 84 Test Method for Surface Burning Characteristics of Building Materials¹¹
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry¹²
- E 300 Practice for Sampling Industrial Chemicals¹³
- 2.2 U.S. Federal Test Methods Standard 141: ¹⁴
- 2112 Application by Roller
- 2131 Application of Sprayed Films
- 2141 Application of Brushed Films
- 3011 Condition in Container
- 4121 Dry Opacity
- 4122 Hiding Power (Contrast Ratio)
- 4203 Reducibility and Dilution Stability
- 4250 Color of Pigmented Coatings
- 4494 Sag Test (Multinotch Blade)
- 4541 Working Properties and Appearance of Dried Film
- 6141 Washability of Paints
- 6142 Scrub Resistance
- 6192 Abrasion Resistance (Taber Abraser)
- 2.3 U.S. Federal Specification: ¹⁴
- TT-F-1098

⁷ Annual Book of ASTM Standards, Vol 06.03.

⁸ Annual Book of ASTM Standards, Vol 14.02.

⁹ Discontinued, see 1979 Annual Book of ASTM Standards, Part 28.

¹⁰ Discontinued; see 1989 Annual Book of ASTM Standards, Vol 06.01.

¹¹ Annual Book of ASTM Standards, Vol 04.07.

¹² Annual Book of ASTM Standards, Vols 06.01 and 14.02.

¹³ Annual Book of ASTM Standards, Vols 06.03 and 15.05.

¹⁴ Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

2.4 Military Standards: ¹⁴

MIL-C-83286, Determination of Free Aliphatic Diisocyanate Monomer by Infrared Spectroscopy

3. Definitions

3.1 For definitions of terms used in these practices, refer to Definitions D 16.

4. Conditions Affecting Performance of HIPAC Coating Systems

4.1 Practical requirements for high performance coatings may vary with:

4.1.1 Substrate type such as concrete, poured or precast block, lime-gypsum plaster, etc.

4.1.2 Climatic conditions, both generally and specifically, at the time of coating application. ASTM standard conditions for laboratory testing are 73.5 ± 3.5°F (23 ± 2°C) and 50 ± 5 % relative humidity.

5. Sampling

5.1 Prior to sampling, establish the condition of the container since damage to it may cause evaporation, skinning, or other undesirable effects. Excessive storage time and temperature fluctuations may cause settling or changes in viscosity.

5.2 Sample the coating in accordance with the Practice E 300. Determine the weight per gallon or kilogram per litre in accordance with Test Method D 1475 and repeat until two successive readings agree within 0.2 lb (90 g) or as agreed upon between the purchaser and the seller. Samples for testing may then be taken.

5.3 Specify the amount of the sample, the package sizes, and identification codes to assure a representative sample. A 1-gal (3.8-L) sample is usually sufficient for the recommended tests but the practice gives sample size in relation to quantity and size of container.

6. Laboratory Tests

6.1 Preparation of Test Panels:

6.1.1 Unless otherwise specified, test panels shall be 1½ by 7½ by 15½-in. (38 by 190 by 395-mm) masonry units made from either standard lightweight concrete block, having an apparent specific gravity of 1.60 to 1.62 or smooth asbestos cement board complying with Specification C 220.

6.1.2 One face only of the test panel shall be coated with the complete system, in a vertical position. For concrete, the filler shall either comply with U.S. Federal Specification TT-F-1098 or be the material specified and supplied by the manufacturer. The filler coat shall be applied in conformance with the manufacturer's printed directions for surface preparation, mixing, application, coverage, and curing time under standard conditions of temperature and humidity.

7. Liquid Coating Properties

7.1 Condition in Container—Thickening, settling, and separation are undesirable and objectionable if a coating, after storage, cannot be readily reconditioned and made suitable for application with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties after storage. Determine the condition in the container in accordance with Method 3011 of Federal

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
Liquid coating properties:			
Condition in container	7.1	...	3011
Coarse particles and foreign matter	7.2	D 185, Section C	
Density or weight per gallon	7.3	D 1475	
Fineness of dispersion	7.4	D 1210	
Odor	7.5	D 1296	
Consistency	7.6	D 562	
Dilution stability	7.7	...	4203
Flash point	7.8	D 93	
Free diisocyanate content	7.9	D 2615	
Volatile content	7.10	D 1644	
Application and film formation:			
Application by brush	8.1	...	2141
Application by roller	8.2	...	2112
Application by spray	8.3	...	2131
Leveling properties	8.4	D 2801	
Working properties	8.5	...	4541
Sag resistance	8.6	...	4494
Wet film thickness	8.7	D 1212	
Producing films of uniform thickness	8.8	D 823	
Curing time	8.9	...	...
Appearance of dry coating:			
Color difference, visual	9.1	...	4250
Color difference, instrumental	9.1	D 2244	
Directional reflectance	9.2	E 97	
Gloss (60-deg specular)	9.3	D 523	
Hiding power	9.4	D 344	4121-4122
Properties of dry coatings:			
Abrasion resistance	10.1	...	6192
Adhesion	10.2	...	...
Chemical resistance	10.3	D 1308 ^A	...
Coating thickness	10.4	D 1005, D 1186	...
Flame spread test	10.5	E 85	...
Fungus resistance	10.6	D 3273	...
Heat and coal resistance	10.7	D 1211 ^A	...
Heat and humidity resistance	10.8	D 2247 ^A	...
Impact resistance	10.9	D 2794	...
Perspiration resistance	10.10	...	...
Scrubability	10.11	...	6142
Washability	10.12	...	6141 ^A

^A Modified.

Test Method Standard No. 141.

7.2 Coarse Particles and Foreign Matter—Coatings must be free of oversize particles and foreign matter to form a uniform film of good appearance, a typical maximum being 0.5 weight % of total coating. The specified test with a No. 325 (45- μ m) screen gives the percent of these particles in a material. Determine coarse particles and foreign matter in accordance with Test Methods D 185, Section C.

7.3 Density or Weight Per Gallon—The density of a coating as measured by weight per gallon is used to assure product uniformity from batch to batch and provides a check against the theoretical weight calculated from the formula. The referenced test method gives a procedure for measurement of the density of a liquid coating. The density is expressed as weight in pounds per 1 U.S. gal or in kilograms per litre of the coating at a specified temperature. A calibrated weight-per-gallon cup is used. Determine weight per gallon in accordance with Test Method D 1475.

7.4 Fineness of Dispersion—One method for measuring the degree of dispersion is to draw a liquid coating down in a tapered groove in a hardened steel block with a groove varying in depth from 4 to 0 mils (100 to 0 μ m). The point at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the wet film is taken as

the fineness reading. Lower readings in mils or micrometers or higher readings in Hegman units indicate better dispersion. A typical fineness of dispersion requirement is a reading of 1.5 mil (38 μ m) or 5 Hegman for HIPAC. Determine fineness of dispersion in accordance with Test Method D 1210.

7.5 Odor—Adequate ventilation is required as some solvent combinations produce obnoxious odors. Test for odor in accordance with Test Method D 1296.

NOTE: Warning—Even though the odor may be pleasant, the fumes may be dangerously toxic.

7.6 Consistency—Coatings of a given type should fall within a consistency range agreed upon. In the referenced test method, consistency is defined as the load in grams required to produce a specified rate of shear. A typical range is 85 to 135 Krebs' unit (KU) for base coats and 70 to 95 KU for glaze coats. Determine the consistency of the product in accordance with Test Method D 562.

7.7 Dilution Stability—The diluent suggested for reduction should be readily incorporated into the coating without excessive stirring or shaking. This test is a measure of the stability of a coating that has been reduced to a desired viscosity, for example, for spray application. Determine

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TABLE 2 Alphabetical List of Test Methods

Method No. 141	Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
	Abrasion resistance	10.1	...	6192
	Adhesion	10.2	...	...
	Application by brush	8.1	...	2141
	Application by roller	8.2	...	2112
	Application by spray	8.3	...	2131
	Chemical resistance	10.3	D 1308 ^A	...
	Coarse particles and foreign matter	7.2	D 185, Section C	...
	Coating thickness	10.4	D 1005-D 1186	...
	Color difference	9.1	D 2244, D 1729	4250
	Condition in container	7.1	...	3011
	Consistency	7.6	D 562	...
	Curing time	8.9	...	...
	Density or weight per gallon	7.3	D 1475	...
	Dilution stability	7.7	...	4203
	Directional reflectance	9.2	E 97	...
	Fineness of dispersion	7.4	D 1210	...
	Flame spread test	10.5	E 84	...
	Flash point	7.8	D 93	...
	Free diisocyanate content	7.9	D 2615	...
	Fungus resistance	10.6	D 3273	...
	Gloss (60-deg specular)	9.3	D 523	...
	Heat and cold resistance	10.7	D 1211 ^A	...
	Heat and humidity resistance	10.8	D 2247 ^A	...
	Hiding power	9.4	D 344	4121-4122
	Impact resistance	10.9	D 2794	...
	Leveling properties	8.4	D 2801	...
	Odor	7.5	D 1296	...
	Perspiration resistance	10.10	...	...
	Producing films of uniform thickness	8.8	D 823	...
	Sag resistance	8.6	...	4494
	Scrubability	10.11	...	6142
	Volatile content	7.10	D 1644	...
	Washability	10.12	...	6141 ^A
	Wet film thickness	8.7	D 1212	...
	Working properties	8.5	...	4541

^A Modified.

dilution stability in accordance with Method 4203 of Federal Test Method Standard No. 141.

7.8 Flash Point—Organic solvents used in coatings have characteristic temperatures at which they will support combustion. This temperature is known as the flash point. It is often used for danger classification in shipment by common carriers. It is also used to determine conditions of storage to meet fire regulations and also the safety requirements of the Occupational Safety and Health Act (OSHA). This temperature is not directly translatable as the danger level when applied as films to surfaces. Determine the flash point in accordance with Test Methods D 93, when in closed or open cups.

7.9 Free Diisocyanate Content—It is generally recognized that diisocyanate vapors from polyurethane-type HIPAC coatings are potential health hazards. Therefore, the free diisocyanate content of urethane coating systems must be controlled at an acceptable maximum level, the present accepted maximum being 0.5 % based on total coating weight, which is applicable only to toluene diisocyanate (TDI) and hexamethylene diisocyanate (HMDI). It has not been shown that this level is applicable to all diisocyanates. Determine free toluene diisocyanate content in accordance with Test Method D 2615. There is no ASTM test method applicable for the other diisocyanates, but test methods are now being developed by ASTM. The presence of free HMDI can be determined by the method described in Appendix I of U.S. Military Specification MIL-C-83286.

7.10 Volatile Content (Weight Percent)—The percent of volatile matter indicates the thinner loss from the film as it dries. Calculate the volatile content of the coating by difference after determining the nonvolatile content in accordance with Test Methods D 1644.

8. HIPAC Application and Film Formulation

8.1 Application by Brush—Brushed films should be smooth and free of seeds and on vertical surfaces should show no sagging, color streaking, or excessive brush marks. Test application or brushed films in accordance with Method 2141 of Federal Test Method Standard No. 141.

8.2 Application by Roller—Walls are frequently painted with rollers that produce slight stipple effects. Test roller coating properties in accordance with Method 2112 of Federal Test Method Standard No. 141.

8.3 Application by Spray—Interior coatings are sometimes applied by spray. Both air and airless spray are used in commercial work. Test in accordance with Method 2131 of Federal Test Method Standard No. 141.

8.4 Leveling Properties—Leveling is a factor in hiding and appearance of coatings. The referenced test method covers the laboratory determination of the relative leveling characteristics of liquid coatings. Determine leveling property in accordance with Test Method D 2801.

8.5 Working Properties—Working properties of a coating are generally compared to a standard or described by requirement in the product specification. Test working

properties in accordance with Method 4541 of Federal Test Method Standard No. 141.

8.6 *Sag Resistance*—Some coatings sag and form curtains before the film sets. Test for sag resistance in accordance with Method 4494 of Federal Test Method Standard No. 141.

8.7 *Wet Film Thickness*—Measurement of wet film thickness is useful in calculating spreading capacity or adjusting application to an agreed upon square feet per gallon or square metres per litre. Determine wet film thickness in accordance with Test Methods D 1212.

8.8 *Producing Films of Uniform Thickness*—The following test method covers the preparation of enamel films of uniform thickness essential in conducting various tests. Prepare films in accordance with Test Methods D 823.

8.9 *Curing Time*—The cure of a HIPAC system is governed by the composition of the coating and by atmospheric conditions during cure. Insufficient cure may result in poor stain and abrasion performance. Typical cure times are between 3 and 21 days depending upon the system. There are no applicable ASTM or Federal test methods to measure cure of HIPAC systems, but one method used is as follows:

8.9.1 After the final cure for the duration specified by the manufacturer, place 10 drops of acetone on the coated surface and cover with a 25-mm watchglass. After 3 min lift the watchglass and remove the acetone by blotting. Allow a recovery period of 1 h and examine for evidence of softening or wrinkling of the coating.

9. Appearance of Dry Coating

9.1 *Color Difference*—Visual comparison of color is fast and often acceptable, although numerical values are not obtained. However, the appearance of a color is greatly influenced by several factors.

9.1.1 A color next to a yellow wall will look different than the same color next to a blue wall. The same color illuminated by incandescent light, fluorescent light, and natural northern light, will appear to be different because the spectral makeup of the incident lights vary.

9.1.2 Gloss also affects color appearance. The same color with a low gloss and high gloss will usually appear to differ, although instrumentally the colors may be identical. The reason is that the color seen is different when the incident light is unevenly scattered by the rougher surface of the low-gloss finish. Often times, the paint manufacturer is expected to produce color matches for all sorts of incident conditions. Hence, the method given in Method 4250 of Federal Test Method Standard No. 141 provides a reasonably accurate method of visual comparison using natural northern light as the incident source. Where a more accurate method of visual comparison is desired, Practice D 1729 should be employed since it specifies the composition of the incident light.

9.1.3 Determination of color difference by instrument in accordance with Method D 2244 is the more reproducible, but visual perception is the more nearly related to the needs in actual use, where the conditions are unpredictable but may be expected to include many types of lighting.

9.2 *Directional Reflectance*—This is an important property of a white coating as it is a measure of the brightness of the surface. Frequently the reflectance of a white coating

may be reduced to increase hiding power. To maintain brightness, a typical minimum reflectance value of 84 may be specified for white. Determine directional reflectance in accordance with Test Method E 97.

9.3 *Gloss (60-deg Specular)*—Semigloss HIPAC coatings are particularly sensitive to loss of gloss due to differential absorption on some surfaces. This is often controlled by testing the gloss of a system applied over a nonabsorbent substrate such as glass and an absorbent surface like gypsum board. Determine the specular gloss on appropriate substrates in accordance with Test Method D 523.

9.4 Hiding Power:

9.4.1 Hiding power is a measure of the ability of a coating to hide the substrate. However hiding power of a white coating is inversely related to its reflectance so that it decreases with increasing directional reflectance. Hiding power is expressed as area covered per unit volume of coating to produce a reflectance over a black surface that is 98 % of the reflectance over a white surface (contrast ratio of 0.98).

9.4.2 Hiding power by contrast ratio can be determined by application of a coating to black and to white glass panels or to black and white charts. The former is more accurate and is the preferred procedure in Test Method D 2805, which also employs automatic film application to eliminate rheological effects. Method 4122 of Federal Test Method Standard 141 is a less detailed procedure using drawdown application on black and on white glass. Contrast ratio on black and white charts can also be determined in accordance with Procedure B of Method 4121.

9.4.3. Hiding power is determined in Test Method D 344 by comparison of brush application of the material under test and a standard coating.

9.4.4 Determine hiding power by one of the above methods consistent with the degree of importance attached to this parameter.

10. Properties of Dry Coatings

10.1 *Abrasion Resistance*—Wear resistance of HIPAC coatings is one of the outstanding qualities which distinguishes them from ordinary interior wall and trim enamels. Determine abrasion resistance in accordance with Method 6192 of Federal Test Method Standard No. 141.

10.2 *Adhesion*—Adhesion is the property of the film that resists removal from the substrate when scuffed or scraped and is an important property in a HIPAC system. There are no applicable ASTM or Federal test methods to measure adhesion of HIPAC systems, but one method used is as follows:

10.2.1 Attach heavy screw eyes to one end of each of twelve 1-in. (25-mm) diameter wood or metal dowels approximately 1½ in. (38 mm) long. Glue the dowels to the cured coating on a test panel at 3-in. (75-mm) centers, using a cold-curing epoxy adhesive applied to both the coating and the dowel. Make sure that the button of adhesive does not extend beyond the diameter of the dowel or the area will be larger than intended. After conditioning for 3 days at 73.5 ± 3.5°F (23 ± 2°C), slowly apply an axial pull of 150 lbf (680 N) to each of three dowels. The force may be applied by any method having an accuracy of 5 %, such as by a system of weights and pulleys or through a spring scale (spring bal-

ance). To provide a surface having through removal of between the occurs between

10.2.2 If panel coated 73.5 ± 3.5 water in the end of 1bf (90 N) dowels, us

10.3 C/ HIPAC system removal cleaners. method s

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50 lbf (680
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ance). To prevent injury should the dowel come loose, provide a suitable guard by placing over the dowel a length of pipe having a cap at one end and a drilled hole in the cap through which to apply the force. Failure is defined as removal of at least one of the three dowels due to separation between the test panel and the top or glaze coat(s). If failure occurs between the adhesive and the coating, test an additional dowel.

10.2.2 If the above portion of test is passed, place the test panel coated side up in a pan containing water maintained at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$). Keep about 0.5 in. (13 mm) of water in contact with the uncoated portion of the panel. At the end of 7 days, pull three more dowels, using a force of 20 lbf (90 N). Dry the coating for 24 h and pull three more dowels, using a force of 30 lbf (135 N).

10.3 *Chemical Resistance*—An important property of a HIPAC system is its ability to resist spotting, softening, or removal when subjected to household chemicals or strong cleaners. There is no applicable ASTM or Federal test method specifically designated for measuring resistance of HIPAC systems to chemical and cleaning agents, but Test Method D 1308 has been modified as follows: Prepare 5 weight % solutions of sodium hydroxide, sodium hypochlorite, detergent, hydrochloric acid, sodium phosphate, and sulfuric acid. For each solution saturate a small piece of absorbent cotton, place them on the cured surface, and cover with a 25-mm watchglass. After 4 h, remove, blot dry, allow to recover for 24 h, and examine the exposed surface of the coating for evidence of softening or wrinkling.

10.4 *Coating Thickness*—Measure total dry film thickness from the top of the substrate to the top of the applied coating system. When applied to steel, use Test Methods D 1186. When applied to nonmagnetic surfaces, use Test Methods D 1005 if the surface is plain and rigid.

10.5 *Flame Spread Test*—For some uses HIPAC coatings should not increase fire hazard. Where required, determine the flame spread and smoke development rating in accordance with Test Method E 84.

10.6 *Fungus Resistance*—Since HIPAC coatings may be used in food processing plants and locations of high humidity, they should be resistant to mold growth on the surface. For this test, an environmental chamber is needed. Determine the resistance to mold growth in accordance with Test Method D 3273.

10.7 *Heat and Cold Resistance*—Buildings can undergo cycling of temperatures that could, through differential thermal expansion between the coating and the masonry substrate, cause failure. There is no applicable ASTM or Federal test methods specifically designated for measuring resistance of HIPAC systems to cold and heat cycles but Test Method D 1211 has been modified as follows:

10.7.1 Place coated panels of the substrate of interest in a cold chamber maintained at 5°F (-21°C) for 1 h. Remove and immediately place in an oven maintained at 120°F (49°C) for 1 h. Repeat this cycle 10 times and examine the coating for cracks, checks, or other defects.

10.8 *Heat and Humidity Resistance*—HIPAC coatings can be subjected to environmental conditions where high heat and humidity are present. There is no applicable ASTM or Federal test method specifically designated for measuring resistance of HIPAC systems to heat and humidity but

Practice D 2247 has been modified as follows:

10.8.1 Coat panels with the complete system and expose to an environment of 95 % relative humidity minimum, and 140°F (60°C) for 7 days in an enclosed cabinet. Determine the 60-deg gloss before and after by Test Method D 523 and color change by Method D 2244.

10.9 *Impact Resistance*—An important property of HIPAC coatings is their ability to withstand impact when objects are accidentally knocked against them. Determine impact resistance in accordance with Test Method D 2794, except use zinc phosphate-treated steel panels. Some materials, such as urethanes, require a wash primer or an epoxy primer to give the adhesion needed for this test. Apply top or glaze coat(s) at a minimum dry-film thickness of 3 mils (75 mm) and examine the surface of the impacted coated area under $7\times$ magnification for cracking and crazing of the coating immediately after testing and 72 h after the test.

10.10 *Perspiration Resistance*—HIPAC coatings can sometimes come in contact with perspiration that could stain the coating. There are no ASTM or Federal test methods to measure perspiration resistance of HIPAC systems but where considered important the following method can be used:

10.10.1 Prepare synthetic perspiration fluid having the following composition by weight:

Urea	1.30
K_2CO_3	0.11
$\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$	0.65
NaCl	2.71
Acetic acid (5 % solution)	1.08
H_2O	10.85
Anhydrous lanolin	83.30

10.10.1.1 Dissolve the urea and inorganic salts in the acetic acid solution and water. Heat the lanolin to 45°C and add the solution to form an emulsion. Combine with a dye and wetting agent in the following proportions:

Scarlet red	0.1
Methanol	3.7
Wetting agent	3.8
Synthetic perspirant	92.4

10.10.1.2 Dissolve the dye in the methanol, then mix with the wetting agent and synthetic perspirant. Apply an excess of the mixture to a small area of a test panel, using a 1 in. (25 mm) wide artist's brush. Cover with a 25-mm watchglass and place the panel in an oven at $120 \pm 3.5^\circ\text{F}$ ($49 \pm 2^\circ\text{C}$), for 4 h. Remove the panel and allow to cool at room temperature for 1 h. Wipe off the excess with a soft dry cloth. If the stain is not completely removed, subject the panel to not more than 25 cycles of the washing procedure described in Method 6141 of Federal Test Method Standard No. 141. Examine the film under $7\times$ magnification for stain retention.

10.11 *Scrubability*—The ability of an interior finish to resist scrubbing is an important property. The referenced method provides a measure of the wet abrasion resistance of a film. However, wet abrasion resistance is not necessarily a measure of how well soils or stains can be removed since some porous materials have good scrubability but poor stain cleansability. Test the coating for at least 4000 cycles in accordance with Method 6142 of Federal Test Method Standard No. 141 except use smooth cement asbestos board complying with Specification C 220 as substrate. Apply the coating system to be tested in conformance with the manufacturer's printed directions for surface preparation, mixing,

application, coverage, and curing time (usually 21 days) at standard conditions of temperature and humidity.

10.12 *Cleansability*—The ability to remove marks satisfactorily without damaging the film is an important property of interior finishes. A test method to determine how well stain and soils can be removed from a cured film is now in preparation and will be included in the guides when it is approved by ASTM. Test methods used by the paint industry consist of applying various household soils or stains to the cured film and attempting to remove them by a washing or scrubbing operation. A suggested test for washability, based on Method 6141 of Federal Test Method Standard No. 141 is given below:

10.12.1 *Coating Application*—Coat a test panel approximately 12 by 4 in. (305 by 102 mm) with the complete system of the product under test, applying the glaze or top coating to at least 3-mils (75-mm) dry-film thickness is not more than two coats. Allow to cure under standard condi-

tions for the time specified by the manufacturer but not more than 21 days.

10.12.2 *Stain Application*—Typical household stain media are coffee, grape juice, ink, soil, crayon, and lipstick. (See Practice D 3023 for specific materials.) Apply the stain media about 1 in. (25 mm) apart in the center of the panel. Apply crayon and lipstick by normal hand pressure; smear coffee, grape juice, and ink with an artist's brush; apply soil under load by placing a 1-lb (500-g) weight on top of an applicator in such a manner that the soil is uniform in thickness.

10.12.3 *Removal of Stain*—After allowing the stains to set for 24 h under standard conditions, wash the stains for 100 cycles in accordance with Method 6141 of Federal Test Method Standard No. 141. Observe the panel for stain removal during and at the end of 100 cycles. If a stain is removed in 100 cycles or less, report the number of cycles required. If not removed in 100 cycles, report the test as a failure.

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1. Scope

1.1 This test method covers the determination of the resistance of a coating to energy.

2. Reference

- 2.1 ASTM
 - D 658 Test Method for Coating
 - D 968 Test Method for Coating
 - D 1474 Test Method for Coating
 - D 2197 Test Method for Scrape
 - D 2336 Test Method for Through Wood
 - D 2793 Test Method for Coating
 - D 2794 Test Method for the Effect of the Eff
 - D 3023 Test Method for tory-A
 - Reagent
 - D 3281 Test Method for Coating
 - D 3359 Test Method for Test²
 - D 3363

3. Terminology

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¹ This practice is related to Coating D 01.55 on Federal Test Method Standard No. 141. Current edition published as ² Annual

Standard Practice for Reporting Cure Times of Ultraviolet-Cured Coatings¹

This standard is issued under the fixed designation D 3732; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Editorial changes were made throughout in April 1989.

1. Scope

- 1.1 This practice applies to all coatings cured by ultraviolet energy.

2. Referenced Documents

2.1 ASTM Standards:

- D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive²
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive²
- D 1474 Test Methods for Indentation Hardness of Organic Coatings²
- D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion²
- D 2336 Practice for Specifying Properties from Liquid Through Cured State for Coatings Factory Applied to Wood Products²
- D 2793 Test Method for Block Resistance of Organic Coatings on Wood Substrates²
- D 2794 Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)²
- D 3023 Practice for Determination of Resistance of Factory-Applied Coatings on Wood Products to Stains and Reagents²
- D 3281 Test Method for Formability of Attached Organic Coatings with Impact-Wedge Bend Apparatus²
- D 3359 Test Methods for Measuring Adhesion by Tape Test²
- D 3363 Test Method for Film Hardness by Pencil Test²

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

- 3.1.1 *cure*—the condition of a coating after conversion to the final state of cure as measured by tests generally related to end-use performance and mutually agreeable to supplier and purchaser.
- 3.1.2 *ultraviolet curing*—conversion of a coating from its application state to its final use state by means of a mechanism initiated by ultraviolet radiation generated by equipment designed for that purpose.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.
Current edition approved Oct. 29, 1982. Published January 1983. Originally published as D 3732 - 78. Last previous edition D 3732 - 78.

² Annual Book of ASTM Standards, Vol 06.01.

4. Significance and Use

- 4.1 This practice provides a guide whereby all pertinent variables relating to the ultraviolet cure of a coating are described.

5. Procedure

- 5.1 Apply the coating to be cured to the desired substrate at a film thickness typical to that normally used. After approximately the time delay encountered in production pass the coated substrate through the curing equipment and subsequently test for cure, as defined in 3.1.1, using the appropriate methods listed in 5.1.1 through 5.1.11. The most commonly used test methods are listed in 5.1.1 through 5.1.4.

5.1.1 Impact Resistance—Test Method D 2794.

5.1.2 Film Hardness—Test Method D 3363.

- 5.1.3 *Solvent Rub Test*—Hold a pad of cheesecloth or other cloth saturated with an agreed-upon solvent, usually methyl ethyl ketone, over two adjacent fingers using a protective covering. Rub the wet pad back and forth across a 100-mm portion of the cured film using vigorous pressure, one forward and one backward movement constituting one double rub. Take the end point as the number of double rubs required to soften or penetrate the film. Fully cured films are normally required to resist a specified number of rubs and the result of the test is given as exceeding the limit or failing the test.

- 5.1.4 *Sandability*—Ultraviolet cured fillers are usually judged by their sandability with an agreed-upon grit of paper. This test is often made immediately after the material leaves the ultraviolet processor.

5.1.5 Specifying Properties—Practice D 2336.

5.1.6 Abrasion Resistance—Test Methods D 658 and D 968.

5.1.7 Indentation Hardness—Test Methods D 1474.

5.1.8 Adhesion—Test Methods D 2197 and D 3359.

5.1.9 Block Resistance—Test Method D 2793.

5.1.10 Formability—Test Method D 3281.

5.1.11 Stain and Reagent Resistance—Practice D 3023.

- 5.2 Repeat the application, curing, and testing with fresh material on fresh substrates until the shortest time that yields a cured film is obtained.

6. Report

- 6.1 Ultraviolet curing equipment is available with several design variables. Therefore, include the following information with results where applicable:

6.1.1 Number of bulbs,

- 6.1.2 Bulb type (intensity, spectral distribution, composition),
- 6.1.3 Bulb age,
- 6.1.4 Bulb location (height from work and orientation to workpiece),
- 6.1.5 Reflector design,
- 6.1.6 Conveyor speed or exposure time to achieve cure,
- 6.1.7 Substrate,
- 6.1.8 Coating identification and age of sample if known,

- 6.1.9 Wet film thickness,
- 6.1.10 Temperature conditions in the curing unit,
- 6.1.11 Curing environment (air, nitrogen, or other atmosphere),
- 6.1.12 Cure tests used (refer to Section 5), and
- 6.1.13 Age of cured film when tests were run.

7. Keywords

- 7.1 cure time; ultraviolet-cured coatings

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1. Scope

- 1.1 This water content is determined for latex acrylic, acrylate, and water-reducible water-based coatings. The test is performed outside of the coating's pot life.
- 1.2 The test is performed on a safety basis and is the responsibility of the user.

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Standard Test Method for Water Content of Water-Reducible Paints by Direct Injection Into a Gas Chromatograph¹

This standard is issued under the fixed designation D 3792; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is for the determination of the total water content of water-reducible paints. It has been evaluated for latex systems (styrene-butadiene, poly(vinylacetate)-acrylic, acrylic). It has not yet been evaluated for other water-reducible paints but is believed to be applicable. The established working range of this method is from 40 to 55 % water. There is no reason to believe that it will not work outside of this range.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1364 Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)³

E 180 Practice for Determining Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁴

3. Summary of Test Method

3.1 A suitable aliquot of whole paint is internally standardized with anhydrous 2-propanol, diluted with dimethylformamide, and then injected into a gas chromatographic column containing a porous polymer packing that separates water from other volatile components. The water content is determined from area calculations of the materials producing peaks on the chromatogram.

4. Significance and Use

4.1 With the need to calculate volatile organic content (VOC) of water-reducible paints, it is necessary to know the water content. This gas chromatographic test method provides a relatively simple and direct way to determine water content.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 15, 1991. Published July 1991. Originally published as D 3792 - 79. Last previous edition D 3792 - 86.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

TABLE 1 Instrument Conditions

Detector	thermal conductivity
Temperatures, °C:	
Sample inlet	200
Detector	240
Column ^a	
Initial	80
Final	170
Program rate	30/min
Carrier Gas	helium or nitrogen
Flow rate, mL/min	50
Detector current	150 mA
Specimen size	1 μ L

^a For isothermal operation set the column temperature at 140°C. After the 2-propanol has cleared the column adjust the temperature to 170°C until DMF clears the column. Reset the temperature to 140°C for subsequent runs.

5. Apparatus

5.1 *Gas Chromatograph*—Any gas-liquid chromatographic instrument having a thermoconductivity detector may be used. Temperature programming capability is preferable, but isothermal operations may be adequate. See Table 1.

5.2 *Column*—The column should be 4 ft (1.22 m) of 1/8-in. (3.2-mm) outside diameter tubing of stainless steel, or other suitable material, packed with 60/80 mesh (180 to 250 μ m) porous polymer packing material.⁵ A replaceable glass sleeve, glass wool plug, or other suitable material may be placed on the entrance end of the column to retain any nonvolatile materials. This will minimize sludge buildup in the column.

5.3 *Recorder*—A recording potentiometer with a full-scale deflection of 1 to 10 mV, full-scale response time of 2 s or less and sufficient sensitivity and stability to meet the requirements of 5.1.

5.4 *Liquid Charging Devices*—Micro syringes of 10 or 25- μ L capacity.

6. Column Conditioning

6.1 *Procedure*—The packed column is installed in the gas chromatographic unit leaving the exit end disconnected from the detector. This will prevent any contamination of the detector with the column bleed. Set the helium or nitrogen flow rate at 20 to 30 mL/min if a 1/8-in. (3.2-mm) outside diameter column is used. Purge the column 5 or 10 min before heating. Heat the column from room temperature to 200°C at 5°C/min and hold this temperature for at least 12 h (overnight). At the end of this time, heat the column at

⁵ Parapak Q[®], a trademark of Waters Assoc., Inc., Milford, MA, has been found satisfactory. Any other porous polymer packing or other column giving equivalents or superior performance may be used. These products are available from most gas chromatograph suppliers and distributors.

5°C/min to 250°C (the maximum temperature for this packing) and hold for several hours. Cool the column to room temperature and connect the column detector. Reheat the column to 250°C at 5°C/min to observe if there is column bleed. Optimum conditioning of this column may take several cycles of the heating program before a good recorder baseline is achieved.

6.2 Before each calibration and series of determinations (or daily) condition the column at 200°C for 1 h with carrier gas flow.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

7.3 *Carrier Gas*—Helium of 99.995 % or higher purity. High-purity nitrogen may also be used.

NOTE 1—Care should be taken that any moisture that may be present in the carrier gas is eliminated through the use of a suitable carrier gas purifier. Trace levels of water will accumulate on the column at low oven temperatures and may affect the reproducibility as well as the accuracy of the determination.

7.4 *Dimethylformamide (DMF) (Anhydrous)* gas chromatography, spectrophotometric quality (Note 2).

7.5 *2-Propanol (Anhydrous) (Isopropanol)*—See Note 2.

7.6 *Septum Sample Vials*, 10-mL capacity with fluorocarbon-faced septa are preferred.

NOTE 2—Determine the water content of the DMF and 2-propanol by Karl Fischer titration in accordance with Test Method D 1364. Dry the 2-propanol if water is found in it or replace with anhydrous grade.

8. Hazards

8.1 *Dimethylformamide* is hazardous. Check the supplier's Material Safety Data Sheet (MSDS) before use.

9. Preparation of Apparatus

9.1 Install the column in the chromatograph and establish the operating conditions required to give the desired separation (see Table 1). Allow sufficient time for the instrument to reach equilibrium as indicated by a stable base line. Control the detector temperature so that it is constant to within 1°C without thermostat cycling which causes an uneven baseline. Adjust the carrier-gas flow to a constant value.

10. Calibration

10.1 Using the information in Table 1 (as a guide), select the conditions of temperature and carrier gas flow that give

the necessary resolution of the components.

10.2 *Determination of Relative Response Factors*—Anhydrous 2-propanol is used as an internal standard. The response factor to water relative to the standard is determined by means of the following procedure. See Fig. 1 for a typical chromatogram. It is good practice to determine the relative retention time daily or with each series of determinations.

10.2.1 Weigh about 0.2 g of water and 0.2 g of 2-propanol to 0.1 mg into a septum sample vial. If it has been determined that a correction for the water content is necessary, weigh 2 mL of dimethylformamide (DMF) into the vial. If the DMF is anhydrous, simply add 2 mL of it as weighing is not necessary.

10.2.2 Inject a 1-μL aliquot of the above solution into the column and record the chromatogram. The retention order and approximate retention times after the air peak are (1) water, about 0.7 min; (2) 2-propanol, about 2.8 min; and (3) DMF, about 7 min.

10.2.3 The preferred procedure to obtain the water content of the DMF is the Karl Fischer titration (Note 1). If this has been determined, calculate the response factor for water by means of the following equation:

$$R = \frac{W_i A_{H_2O}}{(W_{H_2O} + P W_s) A_i}$$

where:

R = response factor,
 W_i = weight of 2-propanol,
 W_{H_2O} = weight of water added,
 W_s = weight of dimethylformamide,
 A_{H_2O} = area of water peak,
 A_i = area of 2-propanol peak, and
 P = weight % water in DMF

100

10.2.4 If Karl Fischer titration is not available, the following procedure may be used to obtain a reasonable

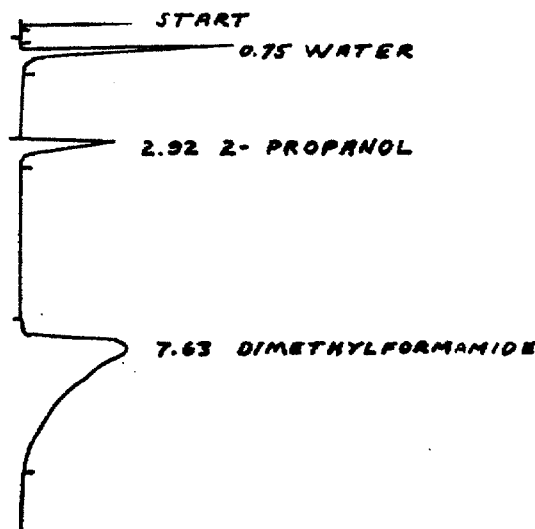


FIG. 1 Typical Chromatogram

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

estimate of the response factor:

10.2.4.1 Inject the same size aliquot of DMF and 2-propanol mixture, but without added water, as a blank. Note the area of the water peak in the blank.

10.2.4.2 The response factor for water is calculated by means of the following equation:

$$R = \frac{W_i(A_{H_2O} - B)}{W_{H_2O}A_i}$$

where:

- R = response factor,
- W_i = weight of 2-propanol,
- W_{H_2O} = weight of the water,
- A_i = area of 2-propanol peak,
- A_{H_2O} = area of the water peak, and
- B = area of the water peak in the blank.

11. Procedure

11.1 Weigh to 0.1 mg, 0.6 g of water-reducible paint (see Note 3) and 0.2 g of 2-propanol into a septum vial. Add 2-mL of DMF into the vial. Seal the vial. Prepare a blank containing the 2-propanol and DMF but no paint.

NOTE 3—Check each paint system to be analyzed for interfering peaks. Coalescing agents do not interfere with this determination.

11.2 Shake the vials on a wrist action shaker or other suitable device for 15 min. To facilitate settling of solids allow the vials to stand for 5 min just prior to injection into the chromatograph. Low-speed centrifugation may also be used.

11.3 Inject a 1-μL sample of the supernatant from the prepared solutions into the chromatographic column. Record the chromatograms using the conditions described in Table 1.

12. Calculations

12.1 Measure the area of the water peak and the 2-propanol internal standard peak and multiply each area by the appropriate attenuation factor to express the peak areas on a common basis. Use of an electronic integrator is recommended to obtain the best accuracy and precision. However, triangulation, planimeter, paper cut out, or ball and disk integrator may be used.

12.2 Calculate the water concentration in the paint by means of the following equation:

$$H_2O, \% = \frac{A_{H_2O} \times W_i \times 100}{A_i \times W_p \times R}$$

where:

- A_{H_2O} = area of water peak,
- A_i = area of 2-propanol peak,
- W_i = weight of 2-propanol added,
- W_p = weight of paint, and

R = response factor determined in 10.2.

12.3 Correction for Water Content of Solvent:

12.3.1 If the blank indicates the presence of a detectable peak for water in the dimethylformamide used as solvent, make a correction in the calculation.

12.3.2 The water content of the dimethylformamide determined by either chromatography (10.2.4) or, preferably, Karl Fischer titration (10.2.3) is used to make the correction. Calculate the water content due to the solvent by using the following equation:

$$H_2O(S), \% = \frac{(W_s)(P)100}{W_p}$$

where:

- W_s = weight of dimethylformamide,
- W_p = weight of paint, and
- P = weight % water in DMF

100

12.3.3 The water content of the paint in this case is the difference between the total percent determined in 12.2 and the correction for the solvent water content as determined in 12.3.2.

13. Precision and Bias⁷

13.1 The precision estimates are based on an inter-laboratory study in which nine different laboratories analyzed in duplicate on two days four samples of water-reducible paints containing from 40 to 55 % H_2O (theoretical). The results obtained were analyzed statistically in accordance with Practice E 180. The within-laboratory coefficient of variation was found to be 1.0 % relative at 34 df and the between-laboratories coefficient of variation 2.6 % relative at 30 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

13.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 2.9 % relative.

13.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 7.5 % relative.

13.2 *Bias*—Bias has not been determined.

14. Keywords

14.1 gas chromatograph; water content of paints by gas chromatograph

⁷ Supporting data are available from ASTM Headquarters. Request RR: D01 - 1022.

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Standard Test Method for Low-Temperature Coalescence of Latex Paint Films¹

This standard is issued under the fixed designation D 3793; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the comparative coalescence of a series of latex paints by determining the porosity of films dried at standard and at low temperature. Porosity is evaluated with a penetrating medium as in Test Method D 3258.

1.2 The texture of the film, which can affect cleanup, will influence the results of the test. Stain applied to a high-hiding paint will not lower the reflectance as much as the same stain applied to a low-hiding paint of equal porosity. These points must be considered in comparing the different paints.

1.3 This method should be used only for comparative testing within one laboratory, as the numerical results obtained by different laboratories do not usually agree.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 3258 Test Method for Porosity of Paint Films²

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry³

3. Summary of Test Method

3.1 The test paints are applied to porous cardboard panels, dried at standard temperature and at 40°F (4.5°C) with 50 % relative humidity in both cases, and their reflectances measured. A special colored penetrating medium is applied to each, the excess removed in a specified manner, and reflectances measured again. The difference between the readings on the stained and unstained films indicates the porosity of that film; the difference between the readings for the two temperatures indicates the coalescence of that film compared to others in the series.

4. Significance and Use

4.1 This test method provides no absolute information, rather ranking only of the selected series of paints.

4.2 This test method can provide a pass-fail situation if known acceptable and unacceptable paints are included in the test.

5. Apparatus and Materials

5.1 *Plate Glass Panel* (base for cardboard panel).

5.2 *Strippable Adhesive*.⁴

5.3 *Test Panel*—Smooth porous cardboard panel such as the back of a white drawdown chart.⁵

5.4 *Film Applicator*, 6 in. (150 mm) wide with a clearance of 6 mils (150 μ m).⁶

5.5 *Film Applicator*, 5½ in. (140 mm) wide with a clearance of 10 mils (255 μ m).⁷

5.6 *Reflectometer*, meeting the requirements of Test Method E 97.

NOTE—Although Test Method E 97 specifies a 45/0 reflectometer, other types of instruments that measure the Y tristimulus value can be used.

5.7 *Cold Cabinet*, maintaining 40 $\pm$ 2°F (4.5 $\pm$ 1°C) and 50 $\pm$ 5 % relative humidity.

5.8 *Camel's Hair Brush*, approximately ½ in. (13 mm) wide.

5.9 *Plastic Wash Bottle*, about 16 oz or 500 mL containing mineral spirits of the odorless type.

5.10 *Penetrating Medium*⁸—A proprietary composition of pigments or dye, or both, dispersed in a liquid vehicle.

5.11 *Filter Paper*.

6. Procedure

6.1 Adhere a porous cardboard test panel to each of as many plate glass panels as needed, using the strippable adhesive and ensuring that the cardboard panels are smooth with no wrinkles.

6.2 For each paint, coat one panel using the 6-mil (150- μ m) clearance drawdown blade. Air dry 48 h under

⁴ Any strippable adhesive such as represented by the following formulas: Vinyl acrylic emulsion such as Flexbond 325, available from Air Products and Chemicals, P.O. Box 538, Allentown, PA 18105 (800 parts by weight) or polyethylene glycol, molecular weight 1500, such as Carbowax 1500, available from Union Carbide Corp., 1300 Lakeside Ave., Cleveland, Ohio 44114 (16 parts by weight).

⁵ Form WA or equivalent white cardboard drawdown chart available from the Leneta Co., P.O. Box 576, Ho-Ho-Kus, NJ 07423, has been found satisfactory for this purpose.

⁶ Bird film applicators or their equivalent available from Byk-Gardner, Inc., Gardner Laboratory, 1110 East West Highway, Silver Spring, MD 20910, have been found satisfactory for this purpose. It should be noted that some applicators are marked with the nominal wet film thickness which is one half of the clearance.

⁷ The Dow latex film caster available from Byk-Gardner, Inc. has been found satisfactory for this purpose.

⁸ K & N S-68 Special Compound available from: K & N Laboratories, Inc. 5331 Dansher Rd., Countryside, IL 60525, has been found suitable for this purpose.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved March 31, 1989. Published May 1989. Originally published as D 3793 - 79. Last previous edition D 3793 - 84.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

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standard conditions of $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity.

6.3 Determine the percent reflectance (Y tristimulus value) of the dry films. Use the green filter when using a reflectometer. When making these readings, back up each panel with a flat rigid opaque object and mark the area on which the reading was made.

6.4 Using the 10-mil (255- μm) blade, apply the penetrating medium to each test paint, covering the area marked as above and drawing the applicator blade in the same direction as before.

6.5 After $5 \text{ min} \pm 15 \text{ s}$, wash off excess penetrating medium. To do this hold the panel vertically and remove the medium using the camel's hair brush wet with odorless mineral spirits. Repeat the process until most of the excess is removed then apply the mineral spirits directly from the wash bottle to the area above the stain and allow to flow down across the stained area. Pause to observe the bead that forms at the bottom of the panel. If the bead is not clear, continue washing with odorless mineral spirits until the bead is essentially clear. Test with filter paper to be sure no dye remains in the bead.

6.6 Hang the panel in a vertical position and air dry for 3 h. As before, read the percent reflectance over the same area on the panels with the same flat rigid opaque object behind them.

6.7 Place one panel for each paint, the 6-mil (150- μm) clearance drawdown blade, and the can of each paint into the cold cabinet maintained at $40 \pm 2^\circ\text{F}$ ($4.5 \pm 1^\circ\text{C}$) and $50 \pm 5\%$ relative humidity.

6.8 At the end of 24 h remove one can of paint and stir thoroughly. Then remove the drawdown blade along with one panel and make the drawdown immediately. Repeat the process for each paint under test. All items should be kept

out of the cold cabinet only long enough to make drawdowns.

6.9 Place all the panels back into the cold cabinet and dry for 48 h.

6.10 Repeat the reflectance readings before and after application of the penetrating medium to these panels in accordance with 6.3, 6.4, 6.5, and 6.6.

7. Calculation

7.1 For each paint calculate the difference between the percent reflectance of the unstained and stained films at both standard and low temperatures. These two differences indicate the porosity of the film formed at the two temperatures. Calculate the low-temperature coalescence of each paint as the ratio of the porosity at low temperature to the porosity at standard temperature which is indicative of relative coalescence.

8. Report

8.1 Report the order of low-temperature coalescence of the test paints in the series.

9. Precision and Bias

9.1 In an interlaboratory study of this test method in which one operator in each of six laboratories tested paint films of three normally different levels of coalescence, all six laboratories ranked the three paints in the same order of coalescence. However, there were considerable differences between the values obtained by the laboratories for any one paint.

10. Keywords

10.1 coalescence; latex paint; porosity

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Testing Coil Coatings¹

This standard is issued under the fixed designation D 3794; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Table 1 was editorially corrected in April 1987.

1. Scope

1.1 This practice covers procedures for testing coil coatings. The test methods included are listed in Table 1. Where more than one test method is listed for the same characteristic, *no attempt is made to indicate superiority of one method over another*. Selection of methods to be followed must be governed by the requirements in each individual case, together with agreement between the producer and user.

1.2 This practice also refers to methods developed specifically for the Coil Industry by the National Coil Coaters Association.²

2. Referenced Documents

2.1 ASTM Standards:

- B 117 Method of Salt Spray (Fog) Testing²
- B 287 Method of Acetic Acid-Salt Spray (Fog) Testing³
- D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings⁴
- D 523 Test Method for Specular Gloss⁴
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces⁴
- D 659 Test Method for Evaluating Degree of Chalking of Exterior Paints⁴
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints⁴
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁴
- D 714 Test Method for Evaluating Degree of Blistering of Paints⁴
- D 822 Practice for Conducting Tests on Paint and Related Coatings and Materials Using Filtered Open-Flame Carbon-Arc Light- and Water-Exposure Apparatus⁴
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion⁴
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁴
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁴

- D 1200 Test Method for Viscosity by Ford Viscosity Cup⁴
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁴
- D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings⁴
- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁴
- D 1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products⁵
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base⁴
- D 1474 Test Methods for Indentation Hardness of Organic Coatings⁴
- D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments⁴
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁶
- D 2092 Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting⁴
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁴
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
- D 2247 Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity⁴
- D 2248 Practice for Detergent Resistance of Organic Finishes⁴
- D 2454 Practice for Determining the Effect of Overbaking on Organic Coatings⁴
- D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings⁴
- D 2794 Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)⁴
- D 2803 Test Method for Filiform Corrosion Resistance of Organic Coatings on Metal⁴
- D 3003 Test Method for Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates⁴
- D 3281 Test Method for Formability of Attached Organic Coatings with Impact-Wedge Bend Apparatus⁴
- D 3359 Test Methods for Measuring Adhesion by Tape Test⁴

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.53 on Factory-Precoated Strip Metal.

Current edition approved May 25, 1979. Published October 1979.

² Annual Book of ASTM Standards, Vols 03.02 and 06.01.

³ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 06.03.

⁶ Annual Book of ASTM Standards, Vol 14.02.

TABLE 1 List of Test Methods and Recommended Practices

	Section	ASTM Method	NCCA Method
Liquid Paint Properties:	6		
Viscosity:	6.1		
Rotational viscometer	6.1.2	D 2196†	
Ford cup	6.1.2	D 1200	
Zahn cup	6.1.3		
Weight solids	6.2	D 1353	
Volume solids	6.3	D 2697	
Fineness of dispersion	6.4	D 1210	
pH	6.5	E 70	
Metal Treatment:	7		
Treatment for zinc-coated steel	7.2	D 2092	
Phosphate weight on steel	7.3.1		NCCA II-1
Phosphate weight on hot-dipped galvanized	7.3.1		NCCA II-3
Chromate weight on aluminum	7.3.2		NCCA II-2
Panel Preparation:	8		
Drawdowns	8.5		
Wet film thickness	8.6	D 1212	
Bake schedule	8.7		
Physical Properties of Cured Coating:	9.0		
Dry film thickness:	9.1		
Nonmagnetic base	9.1	D 1400	
Magnetic base	9.1.1	D 1186	
Micrometre (destructive)	9.1	D 1005	
Color difference	9.2	D 2244 & D 1729	
Gloss	9.3	D 523	
Hardness:	9.4		
Pencil hardness	9.4.1	D 3363	
Indentation hardness	9.4.2	D 1474	
Flexibility:	9.5		
Impact	9.5.2	D 2794	
Wedge bend	9.5.3	D 3281	
Conical mandrel	9.5.4	D 522	
T bends	9.5.5		
Adhesion (tape)	9.6	D 3359	
Pressure marking and blocking	9.7	D 3003	
Overbake	9.8	D 2454	
Detergent resistance	9.9	D 2248	
Stain resistance	9.10	D 1308	
Exterior and Accelerated Durability Tests:	10		
Outdoor exposure	10.1	D 1014	
Accelerated durability:	10.2		
Salt spray	10.2.1	B 117	
Acidified salt spray	10.2.2	B 287	
Humidity	10.2.3	D 2247	
Water immersion	10.2.4	D 870	
Accelerated weathering	10.2.5	D 822	
Accelerated weathering	10.2.5	D 3361	
Filiform corrosion	10.2.6	D 2803	

† Editorially corrected.

D 3361 Practice for Operating Light- and Water-Exposure Apparatus (Unfiltered Open Flame Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products Using the Dew Cycle⁴

D 3363 Test Method for Film Hardness by Pencil Test⁴
E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁷

2.2 *National Coil Coaters Standards:*⁸

No. II-1, Specification for Determination of Phosphate Coating Weights on Steel

No. II-2, Specification for Coating Weight Determination for Chromate Conversion Coatings on Aged or Baked Aluminum Alloy Panels

No. II-3, Specification for Determination of Phosphate Coating Weight on Hot Dip Galvanized and Electro-galvanized Steel

3. General Requirements

3.1 All tests shall be made in diffused light (not direct sunlight) and at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, immediately after baking unless otherwise specified.

4. Definitions

4.1 *coil coating*—application of coatings or films to continuous metal coil stock.

4.2 *metal pretreatment*—chemical treatment normally applied to the metal substrate prior to prime or finish coating. The treatment is designed to react with and modify the metal substrate to produce a surface suitable for coating or adhesive bonding.

⁷ Annual Book of ASTM Standards, Vol 15.05.

⁸ Copies of National Coil Coaters Assn. methods are available through NCCA Headquarters, 1900 Arch St., Philadelphia, PA 19103.

4.3 *reverse roller coat*—coating with the applicator or coating roll revolving in a direction opposite to that of the strip.

4.4 *direct roller coat*—coating with the applicator or coating roll revolving in the same direction as the strip.

5. Sampling

5.1 The number of samples per unit of production shall be agreed upon between producer and user.

6. Liquid Coatings Properties

6.1 Viscosity:

6.1.1 Coatings in the coil industry cover a wide range of generic qualities with many of them having non-Newtonian characteristics. It is therefore important to consider the behavior of these coatings under different shear conditions as well as measuring efflux viscosity by the Ford cup.

6.1.2 Determine viscosity in accordance with Test Methods D 2196 and D 1200.

6.1.3 Viscosity by Zahn cup.⁹

6.1.3.1 *Apparatus*—Standard Zahn cup with specified orifice, a No. 4 or 2 orifice is most commonly used. Stop watch.

6.1.3.2 *Procedure*—Clean the Zahn cup thoroughly. The sample must have a volume of at least 1 pt and the Zahn cup and sample must be $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$). Thoroughly mix the sample. Dip the Zahn cup in a vertical position into the sample being tested, making certain the cup is completely immersed in the liquid but do not allow the cup to touch the bottom of the container. Rapidly lift the cup out of the sample and as the top edge of the Zahn cup breaks the surface start the stop watch. Stop the watch when the first break in the flowing stream of liquid is noted at the orifice exit. Record the seconds elapsed, temperature and number of the Zahn cup used.

6.2 *Weight Solids (NVM)*—Determine nonvolatile matter in accordance with Test Method D 1353.

6.3 *Volume Solids*—Determine volume solids in accordance with Test Method D 2697.

6.4 *Fineness of Dispersion*—Determine fineness of grind in accordance with Test Method D 1210.

6.5 pH:

6.5.1 Control of pH in the pretreatment section of the coil line as well as of waterborne coatings is important in production.

6.5.2 Determine pH in accordance with Method E 70.

7. Metal Pretreatment (Conversion Coating)

7.1 The successful performance of any coil-coated material is dependent not only on the organic coating used but also on the metal preparation. Metal preparation in the coil coating industry usually consists of one of the following:

A		B	
(a)	Clean	(a)	Clean
(b)	Rinse	(b)	Rinse
(c)	Formation of conversion coating	(c)	Formation of conversion coating
(d)	Rinse	(d)	Drying

⁹ Zahn Cups are available from Paul N. Gardner Co. Inc., 316 N.E. First St., Pompano Beach, FL 33061.

A		B	
(e)	Posttreatment of conversion coating		
(f)	Drying		

7.1.1 The metal pretreatment promotes maximum formability and improves adhesion and environmental exposure resistance of the coil coating material. Cleaners, conversion coating treatments and posttreatments used vary with the performance desired, the paint and the metal. Because there is an interdependency between the cleaning, conversion coating, and posttreatment steps, in order to obtain relevant test data, it is necessary that the chemicals, times, concentrations, temperatures, and application methods be as close as possible to those encountered under production conditions.

7.2 In the case of zinc coated steel surfaces, Practices D 2092 Methods A, B, C, and D illustrate the variety of pretreatments available.

7.3 *Coating Weight of Metal Pretreatment*—In many cases, one quality control measurement is the coating weight of the metal pretreatment formed. The following methods are recommended:

7.3.1 *Zinc or Iron Phosphate Conversion Coatings on Steel*—Determine the weight of the iron phosphate conversion coating on steel in accordance with NCCA II-1 and the weight of the phosphate conversion coating on hot dip galvanized steel in accordance with NCCA II-3.

7.3.2 *Chromate-Conversion Coatings on Aluminum*—Determine the weight of the chromate conversion coating on aluminum in accordance with NCCA II-2.

8. Panel Preparation

8.1 *Summary of Method*—This method includes substrate and pretreatment selection for application of coatings by wire wound draw-down bars¹⁰ on laboratory panels.

8.2 *Choice of Substrate*—The substrate to be coated, substrate size, gage, temper, alloy, pretreatment to be used shall be agreed upon between the producer and user. Avoid using treated substrates that have been contaminated by handling or cleaned with solvents or cleaners.

8.3 *Degassing of Substrate*—Some galvanized substrates tend to absorb gasses on aging. To avoid blistering when the substrate is coated and baked it may be necessary to degas the substrate by heating and cooling to room temperature prior to application of the coating. The time and temperature of the degassing cycle shall be agreed upon between the producer and user.

8.4 Drawdowns:

8.4.1 *Apparatus*—Wire-wound draw-down bars, stainless steel bars preferably $\frac{1}{2}$ in. (12.7 mm) in diameter to prevent bowing during application. The choice of the specific draw-down bar is dependent on the dry film thickness required and the volume solids of the coating being tested.

8.4.2 *Procedure*—Secure the substrate to be coated on a hard uniform, even surface. Place an excess amount of the coating to be used near the top of the substrate. Place the draw-down bar in the puddle of the coating to be applied. Rotate or turn the draw-down bar slightly in the coating puddle to ensure complete distribution of the coating in the

¹⁰ Wire-Wound Draw Down Bars available from R. D. Specialties, P.O. Box 206, Webster, N Y 14580, or equivalent may be used.

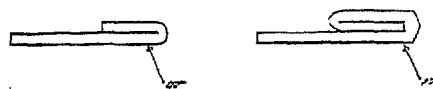


FIG. 1 OT, 1T Bend

ends of the bar. Without rotating the bar, draw it down the length of the substrate at a uniform speed, so that a uniform film is produced. Draw-down speed is dependent on the technology and quality of the coating being tested.

9.5 Wet Film Thickness—Determine the wet thickness of applied coating in accordance with Method D 1212.

9.6 Bake Schedule—Bake the panel at a time and temperature to meet a metal temperature range agreed upon between the producer and user.

Physical Properties of Cured Coating

9.1 Dry Film Thickness—There are several methods currently being used for determining dry film thickness. Depending on the substrates being checked, the following methods should be considered: Test Methods D 1400, D 1186, and D 1005.

9.2 Color:

9.2.1 The color difference between two homogeneously colored opaque films may be determined by visual evaluation or by instrumental means. The color standard used shall be agreed upon between the producer and user.

9.2.2 Color Differences of Opaque Materials by Visual Evaluation—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric and colorimetric characteristics of light source, illuminating and viewing conditions, size of specimens and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color difference in accordance with Practice D 1729.

9.2.3 Color Difference of Opaque Material by Instrumental Evaluation—Color difference between a product and standard can be measured by instrument. The tolerance agreed upon between the producer and user. The referenced method covers the instrumental determination of color differences observable in daylight illumination between nonfluorescent, nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (9.2.2) should be used to verify the results. Make instrumental measurement of color difference in accordance with Test Method D 2244.

9.3 Gloss:

9.3.1 Gloss in the coil industry is generally determined by readings at angles of 20, 60, or 85 deg and in some applications by 60/85-deg or 60/20-deg relationships.

9.3.2 Determine gloss of a coil-coated material in accordance with Test Method D 523.

9.4 Hardness:

9.4.1 Pencil Hardness—Determine the hardness of a coil-coated material by the pencil hardness method in accordance with Test Method D 3363.

9.4.2 Indentation Hardness—Determine the hardness of a coil-coated material in accordance with Test Methods D 1474, using either Method A (Knoop indentation hardness) or Method B (Pfund indentation hardness).

9.5 Flexibility:

9.5.1 There are several flexibility tests used in the coil industry, all giving indications of what the fabrication properties of the coatings are going to be. The most common tests used are impact, wedge bend, T bends, and conical mandrel flexibility.

9.5.2 Impact—Determine the impact resistance of a coil-coated material in accordance with Test Method D 2794.

9.5.3 Wedge Bend—Determine the wedge bend flexibility in accordance with Test Method D 3281.

9.5.4 Conical Mandrel—Determine the conical mandrel flexibility in accordance with Test Methods D 522.

9.5.5 T Bends:

9.5.5.1 Summary of Method—T bend flexibility is a means of evaluating film flexibility and correlating T bends to fabrication properties, by examining the individual T bends for fracturing and tape adhesion of the coating on each bend. The bends are classified OT, 1T, 2T, 3T, etc., with a OT bend being defined as a 180 deg bend, (paint on the outside of the bend) pressed flat so there is no space between the metal and the bend (Note 1). The 1T bend is made by wrapping or bending the metal around the first bent (OT) so that one thickness of metal is between the new wrap, this bend is pressed flat so there is no space between the metal and the bend. This process is repeated to produce the number of T bends required. (See Fig. 1.) The type of metal substrate to be used, whether or not the bends are to be made with or against the grain of the metal, the designation for identifying T bends and the temperature at which the bends will be made should be agreed upon between the producer and user.

NOTE 1—This is the preferred identification of T bends, however, another method is used based on the inside radius of the bend and is $\frac{1}{2}$ of the preferred designation.

9.5.5.2 Apparatus—Brake form machine.¹¹

9.5.5.3 Procedure—Place the panel to be tested into the bed of the brake face, coated side down. Bend panel 180 deg at a uniform rate and press panels flat so there is no space between the metal and the bend, this is the OT bend. Repeat this process to achieve the desired number of T bends. Examine the T bends under 10-power magnification and report the degree of fracturing on each bend based on a rating system agreed upon between the producer and user. Tape each bend with a tape¹² agreed upon between the producer and user.

9.5.5.4 Report—Report the degree of pick off on each bend in a manner agreed upon between the producer and user.

9.6 Adhesion—Determine the tape adhesion of coil materials in accordance with Test Method D 3359.

9.7 Pressure Marking and Blocking—Determine pressure marking and blocking resistance of coil-coated materials in accordance with Test Method D 3003.

9.8 Overbake—Determine the effect of overbaking a coil-coating material in accordance with Practice D 2454.

¹¹ Di Arco Brake form available from Di Arco Manufacturing, Lake City, MN.

¹² Scotch Brand Acetate Fiber Tape No. 710 manufactured by Minnesota Mining and Manufacturing Co., St. Paul, MN., has been found suitable for this purpose.

9.9 *Detergent Resistance*—Determine the detergent resistance of an organic coating for the appliance industry in accordance with Practice D 2248.

9.10 *Stain Resistance*—Determine the stain resistance and effect of household chemicals on a coil-coated finish in accordance with Method D 1308.

10. Exterior Durability and Accelerated Tests

10.1 *Outdoor Exposure*:

10.1.1 While the accelerated tests given elsewhere in this practice are intended to enable prediction of probable performance, actual outdoor exposures should be made on coatings intended for exterior use. The usage of paint systems in the coil area and the liability connected with these systems is so varied that no one set of conditions (length, location, and manner of exposure) can be given in this practice to cover all situations. These conditions, as well as the type of substrate, pretreatment, bake schedule, etc., should be agreed upon between the producer and the user.

10.1.2 Throughout the outdoor weathering tests, several properties should be evaluated periodically or at time intervals agreed upon between the producer and user in accordance with the following methods:

10.1.2.1 *Blistering*—Test Method D 714.

10.1.2.2 *Chalking*—Test Method D 659.

10.1.2.3 *Checking*—Test Method D 660.

10.1.2.4 *Cracking*—Method D 661.

10.1.2.5 *Rusting*—Method D 610.

10.1.2.6 *Corrosion*—Method D 1654.

10.2 *Accelerated Durability*:

10.2.1 *Salt Spray Resistance*—Salt spray testing of coatings may be helpful in determining their resistance to failure in service under conditions of high humidity and salt concentrations. Under the accelerated conditions of the laboratory test, the temperature, pH, concentration of the salt solution and other physical parameters can be controlled. The selection of substrate, pretreatment, the coating system, the manner in which the coating is scribed, the location or position of the panels within the cabinet, the length of the test, the inspection of panels and method of reporting results must be agreed upon between the producer

and user. Test for salt spray resistance in accordance with Method B 117.

10.2.2 *Acidified Salt Spray*—The acidified salt spray is used in the coil industry as a more accelerated test (compared to regular salt spray) for evaluating corrosion resistance of coatings over aluminum. Determine the acidified salt spray resistance of a coating in accordance with Method B 287.

10.2.3 *Humidity*—Determine the humidity resistance of coil-coated material in accordance with Practice D 2247.

10.2.4 *Water Immersion*—Determine the resistance of a coil-coated material to water immersion in accordance with Practice D 870.

10.2.5 *Accelerated Weathering*—The intention of the accelerated weathering test is to cause the degradation of coating films to occur much faster than in natural weathering in various field conditions. The apparatus produces more rapid, but not necessarily the same type of failure of films as does natural sunlight because of its higher level and larger peaks of UV irradiation. Materials which absorb at wavelengths where the peaks in UV occur will degrade more rapidly than those that are transparent to those wavelengths. Most, but not all, organic binders are especially sensitive to radiation below 300 nm, and in some cases, no realistic evaluation of weathering properties can be expected. For these reasons, correlation between natural and accelerated weathering is not always obtained. Whenever possible comparisons should be made with materials having similar vehicles and known durability. Conduct accelerated weathering testing on coil-coated finishes in accordance with Practices D 822 or D 3361 (Note 2), or method agreed upon between producer and user.

NOTE 2—Practice D 3361 does not use filters and the specimens are exposed to UV below 300 nm.

10.2.6 *Filiform Corrosion Resistance*—Filiform corrosion is a type of corrosion that occurs under single-coat systems on a metal substrate and is characterized by definite thread-like structure and directional growth. When filiform corrosion occurs on residential siding the esthetic value of the siding is reduced since the corrosion cannot be washed off. Determine the susceptibility of a coil coated material to this type of corrosion by Test Method D 2803.

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Standard Test Method of Small-Scale Evaluation of Fire-Retardant Paints (2-Foot Tunnel Method)¹

This standard is issued under the fixed designation D 3806; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

This test method determines the protection a coating affords its substrate and the comparative burning characteristics of coatings by evaluating the flame spread over the surface when ignited under controlled conditions in a small tunnel. This establishes a basis for comparing surface-burning characteristics of different coatings without specific consideration of all the end-use parameters that might affect surface-burning characteristics under actual fire conditions.

In addition to the experimental flamespread rate, the weight of panel consumed, time of afterflaming and afterglow, char dimensions and index, and height of intumescent may be measured in this test. However, a relationship should not be presumed among these measurements.

This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Referenced Documents

ASTM Standards:

- D 220 Specification for Flat Asbestos-Cement Sheets²
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts³
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products³
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer³
- E 84 Test Method for Surface Burning Characteristics of Building Materials⁴

This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.22 on Health and Safety.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as D 3806 - 79. Last previous edition D 3806 - 90.

Annual Book of ASTM Standards, Vol 04.05.

Annual Book of ASTM Standards, Vol 06.01.

Annual Book of ASTM Standards, Vol 04.07.

2.2 Federal Standard:

Fed. Spec. TT-V-119 Varnish, Spar, Phenolic Resin⁵

3. Significance and Use

3.1 A number of laboratory procedures are used to evaluate the effectiveness of fire-retardant and fire-resistant treatments and coatings. In general, these methods measure the three stages of fire development: (1) ignition; (2) flame spread (rate of growth of the fire); and (3) conflagration extent. While all three are of extreme importance, flame spread has been recognized as the main factor associated with testing fire-retardant coatings.

3.2 Flame spread ratings based upon Test Method E 84 have acquired common acceptance by regulatory agencies, but such large-scale tests are seldom practical during the development or modification of a fire-retardant coating.

3.3 This test method provides the relative flame spread of experimental coatings using small test specimens. By calibrating the 2-foot tunnel with Test Method E 84-rated fire-retardant paint, results obtained by this test method should be indicative of those obtained with a large specimen in the Test Method E 84 tunnel.

4. Apparatus

4.1 *Flame Tunnel*⁶ (see Fig. 1A)—two-foot (610-mm) flame tunnel consisting of asbestos-cement board mounted on an iron framework and supplied with natural gas fuel of uniform quality.

4.1.1 The framework is constructed of 1/4 by 1 by 1-in. (3 by 25 by 25-mm) angle iron, the top of which provides a 24 by 4-in. (610 by 100-mm) specimen holder inclined at 28° to the horizontal. The framework is mounted on a 10 by 24 by 1/2-in. (250 by 610 by 13-mm) steel base. The specimen holder is elevated 7 3/8 in. (185 mm) above the base at the lower (fire) end and 18 3/8 in. (480 mm) at the upper (flue) end.

4.1.2 The fire end and sides of the tunnel are covered to the extent shown in Fig. 1, with 1/4-in. (6-mm) asbestos-cement board⁷ or 1/4-in. (6-mm) inorganic reinforced-cement

⁵ Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

⁶ Flame tunnels manufactured to similar specifications are available from Custom Scientific Instruments, 13 Wing Drive, Cedar Knolls, NJ 07927 and Monsanto Co., Functional Products, Special Chemicals Div., 800 N. Lindbergh Blvd., St. Louis, MO 63167.

⁷ Transite, manufactured by Manville Corp., Denver CO 80217 has been found satisfactory for this purpose.

Materials of Construction and Equipment List

Item No	Item	Description	Item No	Item	Description
A	Base Plate	1/2 x 10 x 24 Steel plate	F	Glare Shield	1/16 x 2 1/4 x 22 Metal sheeting
B	Framework	1/2 x 1 x Angle iron throughout	G	Ignition Transformer	115 V, 60 Hz, 5000 V, 20 A secondary
C	Side and Front Panels	1/4 Transite or Chem-Fil 100S or 100 H ⁸ cut to specifications	H	Spark Switch	Push button type
D	Slide Glass	1/2 x 2 x 24 Polished Vycor cut as shown	I	Ceramic Insulator	
E	Burner	Artificial Gas Model	J	Spark Wire	1/16 OD Nichrome
	Gas Control Valve (not shown)	1/8 Orifice, 1" Pipe size, outlet 3 oz/in ²	K	Thermocouple	Megopak Type J, totally shielded
	Gage, Gas (not shown)	0-30 oz/in ² Dial		Recording Potentiometer	115 V, 60 Hz, 0-300°C, Type J cond

Note: All Dimensions Expressed in Inches Unless Otherwise Noted.

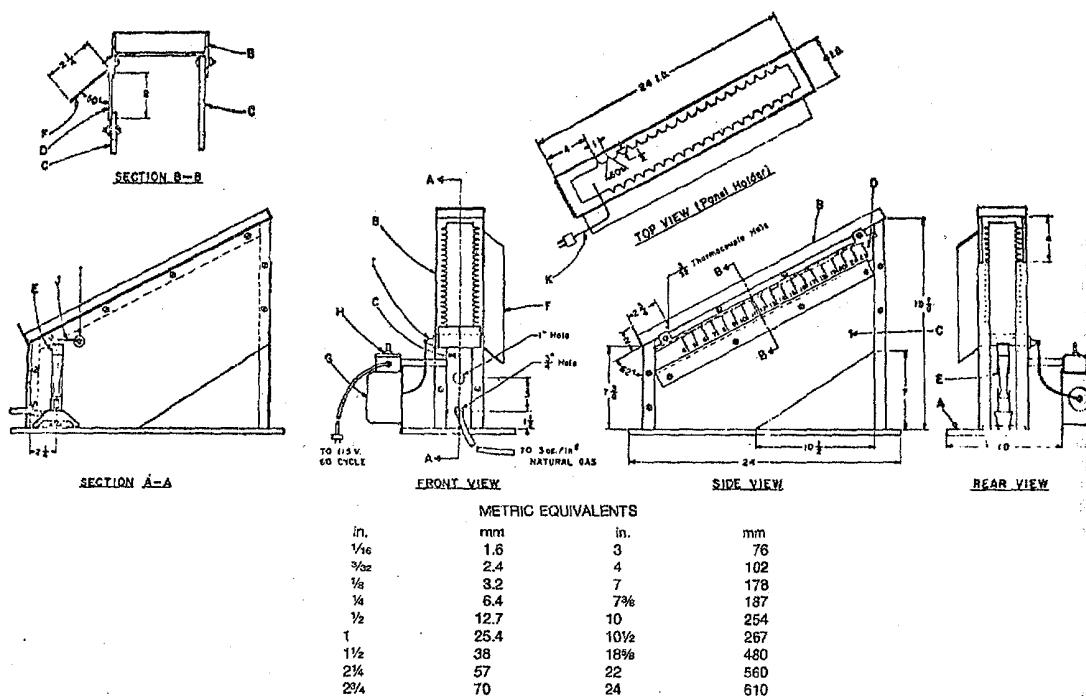


FIG. 1 Flame Tunnel

board⁸ attached to the inside of the angle iron framework. The open flue end and the cut-out sides allow a natural draft through the tunnel. Additional air access is provided by a 1-in. (25-mm) hole drilled in the cover at the fire end, centered and 4 1/2 in. (115 mm) above the base.

4.1.3 An observation window made of a 2-in. (50-mm) wide strip of 1/8-in. (3-mm) polished heat-resistant sheet glass⁹ is located just below the specimen holder, extending the full length of the tunnel, 24 in. (610 mm). This glass is

marked by any appropriate method at 1-in. intervals from 4 to 22 in. A 22 by 2 1/4 by 1/16-in. (560 by 55 by 2-mm) sheet metal shield is attached above the window, slanting downwards at an angle of 60° to shield the observation window. The angle iron panel holder is notched along the bottom or supporting lip of the angle at 1-in. (25-mm) intervals to assist in the measurements of the flame advance.

NOTE 1—A rule may also be mounted outside the observation window to help measure the flame advancement.

4.1.4 A 3/32-in. (2.5-mm) hole is drilled in the side of the specimen holder immediately above the center of the burner to allow insertion of a thermocouple.

4.1.5 The tunnel should be placed in an area as free from

⁸ Chem-Fil 100S and 100H, nominal 100 to 110 lb/ft² (1600 to 1760 kg/m³), Chem-Fil Corp., 100 Spence Lane, Nashville, TN 37210 have been found suitable for this purpose.

⁹ Quartz or high-silica glass has been found satisfactory for this purpose.

TABLE 1 Densities and Panel Weights of Various Woods (Conditioned at Relative Humidity $50 \pm 5\%$ and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) for 14 days)

Type of Wood	Density (9 to 10 % Moisture), lb/ft ³ (kg/m ³)	Weight of 3 $\frac{7}{8}$ by 23 $\frac{7}{8}$ by $\frac{1}{4}$ in. Panel, g
Red cedar	21.4 to 22.4 (345 to 360)	130 to 140
Douglas fir	29 to 35 (465 to 560)	180 to 215
White pine-Ponderosa pine	22.4 to 26.4 (360 to 420)	140 to 160
Southern yellow pine	31 to 37 (495 to 590)	190 to 225
Redwood	27.2 to 28.2 (435 to 450)	165 to 175

as possible with facilities for removal or escape of combustion products. (Standard laboratory hood with forced draft off during test.)

4.2 *Burner*,¹⁰ 8.5 in. (215 mm) high with 40-mm diameter, 10-mm thick grid for use with natural gas. The burner is placed $2\frac{1}{4}$ in. (57 mm) in from the fire end of the panel and 1 in. (25 mm) below the bottom side of the sample holder, that is, $1\frac{1}{2}$ in. (30 mm) from the bottom of the specimen. See Fig. 1.

NOTE 2—Artificial gas or liquid petroleum (LP) gas may be used, but subjects designed for these types of gases must be used.

4.3 *Ignition Transformer*, or other suitable ignition source, to generate a spark through a $\frac{1}{16}$ -in. (2-mm) nichrome wire to the burner barrel. The transformer is activated by a push-button type spark switch.

4.4 *Gas Supply*, of uniform quality controlled by a positive-flow pressure regulatory valve (0 to 6 in. (0 to 152 mm) water) and a gas flowmeter (0.6 to 5.0 SCFH).

4.5 *Insulating and Fire Resistant Backing for Test Panels* consisting of 3 $\frac{7}{8}$ by 23 $\frac{7}{8}$ -in. (100 by 605-mm) pieces of $\frac{1}{2}$ -in. (13-mm) asbestos-cement board or inorganic reinforced cement board and $\frac{1}{2}$ -in. steel plate applied one above the other with asbestos cement or the inorganic reinforced cement board placed directly on top of the test panel.

4.6 *Thermocouple*¹¹ totally shielded (see Note 3) installed through the hole in the side of the specimen holder so that its tip rests on the back, cool side, of the test panel.

NOTE 3—Some commercial tunnels⁶ are supplied with more than one thermocouple.

4.7 *Recording Potentiometer*, 0 to 300°C range.

4.8 *Audible Timer*, set to sound at 15-s intervals to indicate the time for flame-front measurements.

4.9 *Constant Temperature and Humidity Room or Cabinet*, maintained at $50 \pm 5\%$ relative humidity and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$).

5. Test Panels

5.1 *Test Panels*—See Table 1 for a list of various woods and their densities. Wood, $\frac{1}{4}$ by 3 $\frac{7}{8}$ by 23 $\frac{7}{8}$ -in. (6 by 100 by 605-mm) close grained, as nearly edge grain as possible and free from knots and imperfections. The panels should be solid wood with surfaces planed and sanded (see Note 4). Kill-dry test panels to contain not more than 10 weight % of moisture on an oven dry basis.

¹⁰ Fisher Burner Catalog No. 3-902 from Fisher Scientific Co. has been found satisfactory for artificial gas. Fisher Burners No. 3-900 and 3-902 may be used for natural gas and LP gas, respectively.

¹¹ Megopak Type J thermocouple has been found satisfactory for this purpose.

NOTE 4—Douglas fir, $\frac{1}{4}$ -in. (6.3-mm) 3-ply, marine-grade plywood may be used upon agreement between manufacturer and user. Asbestos-cement board, steel, or concrete may be used upon agreement between manufacturer and user.

5.1.1 Prior to coating, condition test panels for 14 days in the controlled atmosphere of $50 \pm 5\%$ relative humidity and $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) to a 9 to 10 % moisture content. Refer to Table 1 for the density and weight per panel of each listed wood.

5.1.2 Seal the ends of the panels with two coats of varnish conforming to Fed. Spec. TT-V-119 after conditioning. Allow each coat of varnish to air-dry 18 to 24 h.

6. Calibration Standards

6.1 *Zero-Flame Spread*—Asbestos-cement board, Type F conforming to Specification C 220, $\frac{1}{2}$ by 3 $\frac{7}{8}$ by 23 $\frac{7}{8}$ in. (13 by 100 by 605 mm), conditioned as in 5.1.1.

NOTE 5—Inorganic reinforced cement board, $\frac{1}{4}$ -in. (6.3-mm) thick may be substituted for asbestos cement.

6.2 *Fire-Rated Standard*—A test panel, similar to that used with test coatings, coated with Test Method E 84 rated paint at the manufacturer's recommended spreading rate and conditioned as in Section 7.

NOTE 6—Do not use paint beyond manufacturer's stated shelf life.

7. Preparation of Test Panels

7.1 Thoroughly mix the coating under test by a suitable means until it is uniform in composition and consistency. Test Method D 2196 describes a suitable preparation procedure.

7.2 Determine the density of the coating in accordance with Test Method D 1475 in pounds per gallon (or grams per millilitre) for calculating the weight of the coating to be applied to the panel to comply with the specified spreading rate.

7.3 Brush apply the coating to conditioned test panels in as uniform a manner as possible to obtain the specified spreading rate. A suitable procedure appears in Test Method D 344. Application may be by other conventional means and in as many coats as required to obtain the necessary weight of coating or desired wet- or dry-film thickness.

7.4 Calculate the weight of coating to be applied using the following equation:

$$W = \frac{(3\frac{7}{8} \times 23\frac{7}{8})D}{144 S_R} \times 454$$

where:

W = weight of applied wet coating, g,

D = coating density, lb/gal, and

S_R = spreading rate, ft²/gal.

7.4.1 Use the following equation when metric units are employed:

$$W = \frac{60.5 D}{S_R}$$

where:

W = weight of applied wet coating, g,
 D = coating density, g/mL, and
 S_R = spreading rate in m²/L.

7.5 Dry the coated panels under standard conditions (4.9) until equilibrium weight is obtained, allowing sufficient time for the complete evaporation of solvents, any curing that might be required by the material, and for the attainment of equilibrium moisture content. Forty-eight hours are normally sufficient except for certain coatings that may require longer periods because they trap solvent or contain solvents that evaporate very slowly.

7.6 Apply and cure non-air-drying coatings as recommended by the supplier and condition as specified in 7.5.

8. Calibration of the Tunnel

8.1 Calibrate the tunnel prior to each day's operation with the calibration standards described in Section 6.

8.1.1 Place the zero flame-spread standard in the holder, smooth side down, and back it with the backing plate (4.5).

8.1.2 Open the gas valve and adjust to a pressure of 3 oz/in.² (1.3 kPa) and a constant flow of 4.8 ft³/h (38 mL/s), and actuate the interval timer.

NOTE 7—These conditions are specified for a natural gas supply providing 1055 BTU/ft³ (40 MJ/m³). For any other gas supply, adjust flow to provide 5085 BTU/h (1490 J/s).

8.1.2.1 When the timer sounds, ignite the burner using the ignition transformer. Observe the flame front, measure in inches and record at 15-s intervals the flame position by aligning the notches in the bottom of the specimen holder with the markings on the observation window.

8.1.3 Record the extreme tip of the flame advance (flame front) on the panel surface, disregarding flame extending up the tunnel but completely on the angle iron support. After a total test period of 4 min, extinguish the burner.

8.1.4 Calculate the mean of the three highest consecutive readings of the flame advance of the zero-flame spread standard L_a .

8.2 Repeat the calibration procedure using a fire-rated standard (6.2). Designate the mean of the three highest consecutive readings of the flame advanced as L_R . The difference between the mean readings for the zero flame-spread standard and the fire-rated standard ($L_R - L_a$) is directly related to the Test Method E 84 flame-spread rating of the rated standard (F_{SR}).

9. Procedure for Determination of Flame-Spread Rating

9.1 Remove a test panel (Section 7) from the controlled atmosphere, weigh immediately, and then mount in the specimen holder, coated-side down. Insert the thermocouple through the hole in the holder (4.6) so that its tip is directly above the center of the burner and resting on the back, or uncoated side, of the test panel. Back the panel with the asbestos-cement board and steel backing.

9.2 Apply the flame and determine the advance of the

flame front in the same manner as the calibration procedure (Section 8), except allow the burner to operate for an additional 60 s after making the last of the 15-s readings before extinguishing the burner. Designate the mean of the three highest consecutive readings of the flame advance of the specimen during the 4-min test period as L_s .

9.3 Repeat the determination with at least five panels.

10. Calculation

10.1 Calculate the experimental flame-spread rating using the following equation:

$$F_{SE} = \frac{(L_s - L_a)}{L_R - L_a} \times F_{SR}$$

where:

F_{SE} = flame spread of specimen,
 L_s = mean of three flame advance readings of specimen, in. (mm),
 L_a = mean of three flame advance readings of zero flame-spread standard, in. (mm),
 L_R = mean of three flame advance readings of rated standard, in. (mm), and
 F_{SR} = flame-spread rating of rated standard.

10.2 Calculate the mean of the replicate panels, rounding off to the nearest five units.

11. Optional Measurements

11.1 *Afterflaming Time*—Observe and record the time in seconds that flaming continues on the face of the test specimen after the burner has been extinguished. Report as afterflaming time.

11.2 *Afterglow Time*—Observe in subdued light and record the additional time in seconds that the surface of the test specimen continues to emit light (glows) after the flame has gone out. Report as afterglow time.

11.3 *Panel Consumption*—After the completion of the flame-spread test (Section 9), replace the specimen in the constant atmosphere (see 5.1.1) for not less than 72 h. Reweigh the panel and determine the weight loss in grams. Report loss as panel consumption.

11.4 *Degree of Intumescence*—Measure the height of intumescence (foam formation) in millimetres at the point on the specimen that was directly above the burner. Report the height as intumescence.

11.5 *Insulation Value*—Measure the initial panel temperature in degrees Celsius or Fahrenheit and subtract it from the maximum temperature observed during the flame-spread test (Section 9). Report the difference as ΔT .

11.6 *Char Dimensions and Index*—Cut the panel into four rectangular pieces using a fine-toothed saw so that the cuts intersect at the point of flame impingement (directly over the burner). Measure in centimetres the maximum width of charring of the wood found below the paint film on the lateral cut. Likewise, measure the maximum length of charring found on the longitudinal cut. Measure the maximum depth to which charring has penetrated as evidenced on either cut. Determine the char index by multiplying the maximum char width, length, and depth.

12. Report

12.1 *Experimental Flame Spread*—Report the following information:

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- 2.1.1 The calibration values of L_R and L_a (Section 8),
- 2.1.2 Type of substrate used,
- 2.1.3 Number of test panels,
- 2.1.4 Mean spreading rate,
- 2.1.5 Pressure, flow rate, and type of gas fuel,
- 2.1.6 Mean L_a value for each panel (Section 9), and
- 2.1.7 Mean flame-spread value (F_{SE}) for each material under test (Section 9).
- 2.2 *Optional Measurements*—Report may contain the following information:
 - 2.2.1 *Panel Consumption*—Mean initial and final weight of coated specimens and mean weight loss in grams (11.3),
 - 2.2.2 Afterflaming time in seconds (11.1),
 - 2.2.3 Afterglow time in seconds (11.2),
 - 2.2.4 Height of intumescence in millimetres (11.4),
 - 2.2.5 *Insulation Value*—Initial and maximum temperatures, and temperature difference (ΔT) (11.5), and
 - 2.2.6 *Char Depth and Volume*—Maximum width, length, and depth of charring of wood in centimetres and the char index in cubic centimetres (11.6).

3. Precision and Bias

3.1 Precision:

- 3.1.1 Nonuniformity of test panels (both in the wood and in the coating), variations in fuel supply and rate, and

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TABLE 2 Repeatability^a

Flame-Spread Ratings	Standard Deviation	Coefficient of Variation
0 to 25	1.2	9.6
25 to 75	3.1	6.2
75 to 135	3.65	3.5
0 to 135	2.85	4.6

^a This information was derived from *Journal of Paint Technology*, Vol. 39, No. 511, 1967, p. 495.

operator error are the chief sources of error in operation of the 2-foot (610-mm) tunnel.

13.1.2 The degree of repeatability is dependent on the level of flame-spread ratings. Table 2 shows that while the standard deviation of duplicate results increases with an increase in ratings, the coefficient of variation decreases by a factor of more than 2.0.

13.1.3 The standard deviation in Table 2 relates to the maximum difference that would be expected between duplicate panels; that is, a second result in the flame-spread range from 0 to 25 should fall within 4.8 units ($\pm 2.8 \times 1.2$) at the 95 % confidence level.

13.2 *Bias*—The procedure in this test method for measuring flame spread has no bias because the value of flame spread can be defined only in terms of a test method.

14. Keywords

- 14.1 fire-retardant; flame spread; paints; tunnel method



Standard Guide for Selection of Test Methods for Coatings for Use in Light-Water Nuclear Power Plants¹

This standard is issued under the fixed designation D 3842; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide is intended to provide a common basis in the selection of test methods that may be required to evaluate and qualify protective coatings (paints) for use in a light-water nuclear power plant, and all the listed items are for Coating Service Level I application. It lists and describes tests to be conducted on all coatings specified for this service.

1.2 This guide also lists the specifications and their scopes that describe evaluation tests for these coating systems.

1.3 Coating Service Level I pertains to those coating systems whose failure could impair the operation of the engineered safety features that are essential for preventing the possible consequences of postulated accidents that could cause undue risk to the health and safety of the general public. Coating Service Level I applies principally to the interior surface of the primary containment for both Pressurized Water Reactors (PWR) and Boiling Water Reactors (BWR) and to the structures and equipment contained therein. Coated surfaces include, but are not limited to, carbon steel, stainless steel, galvanized steel, aluminum, and masonry.

1.4 Requirements for coatings applied to other areas shall be assigned at the option of the owner or his designated representative. It is intended that this standard be incorporated in specifications, by reference, on the basis of owner requirements.

1.5 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters.

Current edition approved Sept. 29, 1986. Published November 1986. Originally published as D 3842 - 80. Last previous edition D 3842 - 80.

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2. Referenced Documents

2.1 ASTM Standards:

D 3911 Test Method for Evaluating Coatings Used in Light-Water Nuclear Power Plants at Simulated Design Basis Accident (DBA) Conditions²

D 3912 Test Method for Chemical Resistance of Coatings Used in Light-Water Nuclear Power Plants²

D 4082 Test Method for Effects of Radiation on Coatings Used in Light-Water Nuclear Power Plants²

D 4256 Test Method for Determination of the Decontaminability of Coatings Used in Light-Water Nuclear Power Plants²

3. Significance and Use

3.1 This guide covers methods of testing coatings for use in light-water nuclear power plants.

4. Test Methods

4.1 *Test Method D 3911*—This test method establishes procedures for evaluating protective coating systems test specimens under simulated Design Basis Accident (DBA) Conditions. Included is a description of conditions and apparatus for temperature-pressure testing, condition for radiation testing, and procedures for preparing, examining, and evaluating the samples after testing.

4.2 *Test Method D 3912*—This test method establishes procedures for the evaluation of the chemical resistance of coatings used in light-water nuclear power plants.

4.3 *Test Method D 4082*—This test method establishes a standard procedure for evaluating the lifetime radiation tolerance of coatings to be used in nuclear power plants.

4.4 *Test Method D 4256*—This test method establishes procedures for determining and ranking the ease of the decontaminability of coatings used in nuclear power plants.

² Annual Book of ASTM Standards, Vol 06.01.



Standard Practice for Quality Assurance for Protective Coatings Applied to Nuclear Facilities¹

This standard is issued under the fixed designation D 3843; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice provides a common basis for, and specifically comprises quality assurance requirements applicable to, safety-related protective coating work in Coating Service Level I areas of nuclear facilities.

1.2 Applicable portions of this practice may be used as the basis for limited quality assurance for protective coating work in Coating Service Level II areas of nuclear facilities.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 3842 Guide for Selection of Test Methods for Coatings for Use in Light Water Nuclear Power Plants²

D 4227 Practice for Qualification of Journeyman Painters for Application of Coatings to Concrete Surfaces of Safety Related Areas in Nuclear Facilities²

D 4228 Practice for Qualification of Journeyman Painters for Application of Coatings to Steel Surfaces of Safety Related Areas in Nuclear Facilities²

D 4537 Guide for Establishing Procedures to Qualify and Certify Inspection Personnel for Coating Work in Nuclear Facilities³

2.2 ANSI Standards:

ANSI Z39.2 Quality Assurance Program Requirements for Nuclear Power Plants⁴

2.3 Code of Federal Regulations:⁵

10 CFR 50, Appendix B: Title 10, Chapter 1, Energy, Part 50, Licensing of Production and Utilization Facilities, Appendix B, Quality Assurance Criteria for Nuclear Power Plants and Fuel Reprocessing Plants

10 CFR 21 Reporting of Defects and Noncompliances

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of D33.03 on Quality Systems.

Current edition approved Feb. 24, 1989. Published April 1989. Originally published as D 3843 - 80. Last previous edition D 3843 - 80.

² Annual Book of ASTM Standards, Vols D6.01 and D12.01.

³ Annual Book of ASTM Standards, Vol D6.01.

⁴ Available from American National Standards Institute, 13th Floor, 11 W. 42nd St., New York, NY 10036.

⁵ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *certification*—the written documentation of the qualification of personnel or material.

3.1.2 *coating applicator*—an organization or individual responsible for applying a protective or decorative coating system.

3.1.3 *coating (paint)*—a liquid, liquifiable, or mastic composition that is converted to a solid protective or functional adherent film after application as a thin layer.

3.1.4 *coating manufacturer*—an organization responsible for manufacturing coating materials.

3.1.5 *coating system*—a polymeric protective film consisting of one or more coats, applied in a predetermined order by prescribed methods.

3.1.6 *coating work*—an all-inclusive term to define all operations required to accomplish a complete coating job. The term shall be construed to include materials, equipment, labor, preparation of surfaces, control of ambient conditions, application and repair of coating systems, and inspection.

3.1.7 *Code of Federal Regulations (CFR)*—the rules and regulations of the United States Federal Government. The code is subdivided into titles, with Title 10 (10 CFR) applying to energy.

3.1.8 *deviation*—a departure of a characteristic from established procedures or specified requirements.

3.1.9 *documentation*—any written or pictorial information describing, defining, specifying, reporting, or certifying activities, requirements, procedures, or results.

3.1.10 *inspection*—a phase of quality control which by means of examination, observation, or measurement determines the conformance of materials, supplies, components, parts, appurtenances, systems, processes, or structures to predetermined quality requirements.

3.1.11 *inspection agency*—a person or persons authorized by the owner or owner's designee to verify conformance of the coating work.

3.1.12 *nonconformance*—a deficiency in characteristic, documentation, or procedure that renders the quality of an item unacceptable or indeterminate. Examples of nonconformances include: physical defects, test failures, incorrect or inadequate documentation, or deviation from prescribed processing, inspection, or test procedures.

3.1.13 *owner*—the person, group, company, or corporation who has or will have the license for the facility or installation.

3.1.14 *owner's designee*—a person or persons authorized by the owner to act in his behalf.

3.1.15 *vendor*—any individual or organization who furnishes items or service to a procurement document.

4. Significance and Use

4.1 Quality assurance, as covered by this practice, comprises all those planned and systematic actions necessary to provide adequate confidence that safety-related coating work in nuclear facilities will perform satisfactorily in service.

4.2 Coating Service Level I applies to areas where coatings failure could adversely affect the operation of post-accident fluid systems. With some exceptions, Coating Service Level I applies to coatings inside primary containment.

4.3 Coating Service Level II applies to areas where coatings failure could impair, but not prevent, normal operating performance. The primary function of Service Level II coatings is to provide corrosion protection and decontaminability in those areas outside primary containment subject to radiation exposure and radionuclide contamination.

4.4 It is not practical to impose all the requirements of this practice on certain specific items that require only a small quantity of coating material. The owner, consistent with his formal Quality Assurance Program, may accept affidavits of compliance or certification attesting to the quality of a shop or field coating for such items. If required by licensing commitments, uncontrolled (unqualified, non-conforming, or unidentified) coatings shall be identified, quantified, and documented.

4.5 This practice may be incorporated in a project specification by direct reference or may be used to provide guidelines for the quality assurance program for coatings, on the basis of the owner's requirements. Effective use of this practice may also require the incorporation of applicable sections in project specifications for coatings on concrete, steel, equipment, and other related items.

5. General Quality Assurance Requirements

5.1 This section defines the general quality assurance requirements necessary for compliance with this practice. These requirements shall apply to all other sections of this practice.

5.2 The owner or the owner's designee shall be responsible for determining whether source inspection or a certificate of compliance attesting to the quality of coating work activity is required.

5.3 The general quality assurance requirements necessary to meet the purpose of this practice provide an acceptable basis for establishing a protective coating quality assurance program. All deviations from or exceptions to these requirements shall be reviewed by and shall be subject to approval by the owner or his designee before implementation. If unapproved deviations are discovered during any phase of the coating work activity, a nonconformance report shall be completed either by the owner, vendor, or owner's designee. The owner or the owner's designee shall approve the disposition of the nonconformance report.

NOTE—Notification of the Nuclear Regulatory Commission (NRC) is required by 10 CFR 21 for certain types of defects and noncompliances.

5.4 There are five major activities that shall be controlled and documented as a minimum for Service Level I protective coatings applied to nuclear facilities as follows:

- 5.4.1 Qualification and selection of coating materials,
- 5.4.2 Coating manufacturing,
- 5.4.3 Surface preparation of substrates,
- 5.4.4 Control of coating application, and
- 5.4.5 Coating inspection.

5.5 The five major activities specified in 5.4 are designed to meet the applicable quality assurance requirements of Appendix B to 10 CFR 50, and of ANSI N45.2. All five of these controlled activities shall have appropriate documentation including approved procedures and records to meet the Appendix B and N45.2 requirements.

5.6 The qualification and selection of coating materials shall be evaluated by the owner on the basis of criteria defined in Section 6 of this practice.

5.7 The coating manufacturer shall furnish a quality assurance program describing his methods for quality control of the specified coatings on the basis of criteria defined in Section 7, which is approved by the owner.

5.8 Should the owner designate an outside coating applicator to apply the specified coatings, the coating applicator shall provide a quality assurance program based on the criteria defined in Sections 8 and 9, which is approved by the owner.

5.9 Should the owner designate an inspection agency to inspect the application of the specified coatings, the inspection agency shall provide a quality assurance program based on the criteria defined in Section 10, which is approved by the owner.

5.10 If application or inspection, or both, are performed under the owner's quality assurance program, procedures shall be established to control these activities.

6. Control of Selection and Qualification of Coating Materials

6.1 All qualifications of coating materials shall meet the applicable standards referenced in Guide D 3842 to the extent defined by the owner or the owner's designee in design criteria, safety analysis reports, quality assurance program, or other controlling documents.

6.2 The coating manufacturer shall furnish recommended surface preparation and application procedures for each coating system on each substrate as covered by the project specification including previously coated surfaces as applicable. The coating manufacturer shall also furnish recommended storage conditions for each coating material specified.

7. Control of Coating Manufacturing

7.1 The coating manufacturer shall maintain a Quality Assurance Program and shall provide adequate documentation to show that the quality of a given coating system as supplied is the same composition (within owner approved manufacturing tolerances) the coating system previously tested for qualification. The coating system shall be requalified if there are significant changes in formulation or end-product properties. The manufacturer shall provide the owner or the owner's designee documentation based on the criteria stated in 7.1.1 through 7.1.5.

7.1.1 The coating manufacturer shall provide the purchaser with a product identity certification record for each batch of coating material shipped. As a minimum, the

als, product identity certification record shall contain the following information:

- 1.1 Established acceptance criteria ranges and batch characteristics for weight per gallon and viscosity,
- 1.2 Batch number,
- 1.3 Date manufactured,
- 1.4 Shelf life expiration date,
- 1.5 Certification (signed by a responsible technical manager) that the product shipped is the same composition (within owner-approved manufacturing tolerances) as the product tested for qualification.

1.2 Separate acceptance criteria may be established for evaluation at time of manufacture and for evaluation during the shelf life period, but these shall be separately identified.

1.3 Data which is generic to the product and not batch specific (for example, nonvolatile, cure time, pot life, etc.) shall be furnished to the owner as requested, but need not be duplicated on each product identity certification record.

1.4 Material which has exceeded shelf life may be evaluated by testing and the shelf life extended in writing by the manufacturer's technical department. Testing for extension of shelf life shall be performed on an unopened container removed from the job site storage area, and not from a retained laboratory sample.

1.5 The owner may elect to perform verification testing on products received to ensure compliance with this practice.

2 Each container shall be labeled with the product designation. The label or container shall bear a batch number or other factory marking, permanently affixed, showing the individual lot or batch designation. The date of manufacture, and the shelf life expiration date shall appear separately on the label or container.

3 Retained batch samples from products furnished for the project shall be kept by the manufacturer for the stated shelf life.

Control of Surface Preparation of Substrates

3.1 This section defines the quality assurance requirements for the surface preparation of bare substrates or of previously coated surfaces.

3.2 The surface preparation of substrates or of previously coated surfaces shall conform to the requirements of the project specification. Treatment of nonqualified or incompatible concrete primers/sealers and concrete form-release agents shall be addressed.

3.3 The project specification shall include inspection methods to ensure the requirements of the specification are met.

3.4 The coating applicator shall submit written surface preparation procedures addressing each substrate to be coated including previously coated surfaces.

3.5 The responsible organization shall report daily on

surface preparation during each shift for each area of work.

9. Control of Application of Coating Systems

9.1 This section defines the quality-assurance requirements necessary to comply with this practice to ensure that the applied coating system is basically the same as that which was tested for qualification.

9.2 The coating applicator shall follow written owner accepted application procedures for applying each coating system on each substrate and on each previously coated surface. The coating manufacturer's latest written application instructions shall be incorporated as part of the procedures.

9.3 All application personnel shall be qualified and their qualification documented in accordance with the applicable quality assurance program and Practices D 4227 or D 4228.

9.4 Receiving, storing, and dispensing of coating materials shall be appropriately documented as per established requirements.

9.5 The responsible organization shall report daily on the application work for each shift for each area of work.

10. Control of Coating Inspection

10.1 It is the responsibility of the owner as identified in 5.3 to specify and verify control measures to assure that the inspection of the coating work is adequate to achieve the required quality. Inspection hold points shall be established to ensure in-process inspection results are adequate. The owner or the owner's designee shall be responsible for the inspection activity. In all cases the inspection function shall be properly documented as required by Appendix B of 10 CFR 50 and ANSI N45.2 for record purposes.

10.2 All inspection personnel shall be qualified and certified in accordance with Guide D 4537.

10.3 Inspection shall conform to all the applicable requirements of the project specification and all other applicable requirements of this document.

10.4 A written daily coating inspection record shall be maintained by the owner.

10.5 In the case of any significant deviation or defective work, the corrective action shall be documented.

10.6 Final acceptance of completed work shall be documented.

11. Documentation

11.1 Sufficient quality-assurance records and documents shall be maintained to furnish objective evidence of compliance with the specifications and the specified quality assurance procedures.

11.2 Documentation shall be maintained by the owner or the owner's designee as defined in the owner's quality assurance program.

11.3 Distribution of documentation shall be as defined in the quality assurance procedures.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Preparation of Glass Panels for Testing Paint, Varnish, Lacquer, and Related Products¹

This standard is issued under the fixed designation D 3891; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice covers the preparation of glass panels for subsequent testing of paint, varnish, lacquer, and related products.²

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 364 Specification for Industrial Grade Xylene³
- D 1152 Specification for Methanol (Methyl Alcohol)⁴
- D 1193 Specification for Reagent Water⁵

3. Significance and Use

3.1 The procedures described in this practice are designed to provide uniform glass panels for testing of paint, varnish, lacquer, conversion coatings and related products.

4. Reagents and Materials

4.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

4.2 *Solvents*—V M & P naphtha, xylene, 2-methoxypropanol, and methanol complying with the appropriate specifications (see Specifications D 364 and D 1152).

4.3 Glass of the following types may be specified in the applicable test methods:

4.3.1 *Window Glass*, (float glass) the surface of which is plain and free of irregularities.

4.3.2 *Clear Plate Glass*, not less than 5 mm thick.

4.3.3 *Plate Glass* that has been ground uniformly on one side with 1F carborundum.

4.3.4 *Black Structural Glass*,⁶ one side of which has been polished to a smooth, high-gloss surface.

4.3.5 *White Structural Glass*, one side of which has been polished to a smooth, high gloss.

5. Method of Preparation

5.1 *New Panels*—Using a clean lintless cloth, wipe the panel with a solvent mixture consisting of equal volumes of V M & P naphtha and xylene or of 3 volumes of V M & P naphtha and 1 volume of 2-methoxypropanol or with water. When all soluble and loosely adhering soil has been washed off, flush with clean solvent. Allow panels to dry at room temperature or force dry at a temperature no higher than 300°F (150°C). After cooling to room temperature, rub with a clean lintless cloth if necessary.

5.1.1 The surface after cleaning shall be water-break free. This is determined by momentarily immersing in reagent water one panel of each twenty prepared. If a continuous water film is not retained (that is, the water film breaks), immerse the panel in methyl alcohol to obtain a water-break-free surface. After removal, dry at a temperature of 125 to 200°F (52 to 93°C) and allow to cool before coating or storing.

5.1.2 Other combinations or organic reagents that will produce a surface equal in cleanliness to the solvent mixtures mentioned in 5.1 may be used if agreed upon between the purchaser and the supplier.

5.2 *Used Panels*—Remove the old paint, using a paint and varnish remover of the organic solvent type or a suitable solvent mixture. Do not use a spatula, sandpaper, or other instrument of treatment that might scratch or etch the surface. After washing off the residues clean the panels as described in 5.1.

5.3 *Protection After Preparation*—Use panels immediately after preparation or store wrapped in clean, hard surfaced paper to minimize lint on the glass surface. To avoid contamination by fingerprints, do not touch the surface of the panels after preparation. Handle only by the edges.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Tests for Protective Coatings.

Current edition approved Aug. 30, 1990. Published November 1990. Originally published as D 3891 - 80. Last previous edition D 3891 - 80.

² This practice is intended to be equivalent to Method 2021 of U.S. Federal Test Method Standard 141.

³ Discontinued, see 1982 *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.03.

⁵ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁶ Black Vitrolite (Libby-Owens-Ford Glass Co.) and Carrara (PPG Industries) glass have been found satisfactory for this purpose and are obtainable from some local glaziers.

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Standard Test Method for Evaluating Coatings Used in Light-Water Nuclear Power Plants at Simulated Design Basis Accident (DBA) Conditions¹

This standard is issued under the fixed designation D 3911; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

INTRODUCTION

During a design basis accident (DBA) in nuclear power plants, conditions in the reactor containment will be characterized by elevated temperature and pressure, as well as the presence of a radiation environment. Water sprays, with or without chemical additives, may be used in the primary containment to suppress the consequences of the incident, to scavenge radioactive products, and to return the containment to near-ambient conditions.

1. Scope

1.1 This test method establishes procedures for evaluating protective coating systems test specimens under simulated DBA conditions. Included are a description of conditions and apparatus for temperature-pressure testing, conditions for radiation testing, and procedures for preparing, examining, and evaluating the samples.

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- D 714 Test Method of Evaluating Degree of Blistering of Paints²
- D 1193 Specification for Reagent Water³
- D 4082 Test Method for Effects of Radiation on Coatings Used in Light-Water Nuclear Power Plants²

2.2 ANSI Standard:⁴

- N512-1974 Protective Coatings (Paints) for the Nuclear Industry.

3. Terminology

3.1 Definitions:

3.1.1 *blistering*—the formation of bubbles in a coating (paint) film.

3.1.2 *boiling water reactor (BWR)*—a reactor in which the water moderator-coolant is boiled directly within the reactor

core. The pressure in the reactor vessel is only slightly greater than the steam turbine pressure.

3.1.3 *chemical spray*—a solution of chemicals, such as those contained in Table 1, which could be used during a loss of coolant accident (LOCA) to suppress the incident, to scavenge fission products, and to return the facility to near-ambient conditions.

3.1.4 *coating (paint) system*—a polymeric protective film consisting of one or more coats applied in a predetermined order by prescribed methods to a defined substrate.

3.1.5 *cracking*—a break or a split in the coating (paint) system extending through the film or to the substrate.

3.1.6 *curing*—the transformation of a coating or other material into a solid phase or film.

3.1.7 *DBA*—a generic term for any one of a family of accident conditions which can result from postulated events. These conditions are generally associated with the rupture of high energy piping. The more commonly recognized accident conditions used to evaluate coating systems for primary containment are the LOCA or main steam pipe break.

3.1.8 *deionized water*—water prepared by an ion exchange process meeting the requirements of Specification D 1193, Types II and III.

3.1.9 *delamination*—separation of one coat or layer from another coat or layer, or from the substrate.

3.1.10 *engineered safety system*—a system designed to mitigate the effects of a design basis accident.

3.1.11 *irradiation*—exposure to ionizing radiation.

3.1.12 *light-water nuclear reactor*—an apparatus, using light water as a moderator, in which fissionable material is arrayed so that controlled nuclear fission may be sustained in a self-supporting chain reaction.

3.1.13 *LOCA*—the specific conditions anticipated fol-

TABLE 1 Typical Spray Solutions

Composition	Chemical Compound	Concentration (in Deionized Water)
A	Sodium borate	2000 to 4000 ppm boron
	Sodium hydroxide	adjust solution to pH 9.0 to 10.0
B	Boric acid	2000 to 4000 ppm boron
	Hydrazine	50 ppm unreacted excess
	Sodium phosphate, dibasic	adjust solution pH to 6.8 to 10.0

¹ This method is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters. Current edition approved Feb. 24, 1989. Published April 1989. Originally published as D 3911 - 80. Last previous edition D 3911 - 80¹.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁴ Available from American National Standards Institute, 13th Floor, 11 W. 42nd St., New York, NY 10036.

Following a loss of coolant accident that would expose the heated surface of the containments of a light-water nuclear power facility to the temperature-pressure environmental parameters described.

3.1.14 *peeling*—separation of one or more coats or layers of a coating from the substrate.

3.1.15 *pressurized-water reactor (PWR)*—a nuclear power reactor design utilizing liquid water under high pressure as moderator-coolant.

3.1.16 *quality assurance*—the verification of the conformance of materials and methods of application to the governing specifications, in order to achieve the desired result.

3.1.17 *reactor containment (containment)*—the enclosure provided to protect the environment from the consequences of a nuclear incident.

Significance and Use

4.1 This test method is designed to provide a uniform test to determine the suitability of coatings used inside primary containment of light-water nuclear facilities under simulated DBA conditions. Variations in actual surface preparation and in application and curing of the coating materials may require additional testing as deemed necessary by the specifying or qualifying agency, or both, if it is anticipated that the variations may adversely affect the performance of the coating system during a DBA. This test method is intended only to demonstrate that under DBA conditions, the coatings will remain intact and not become debris which could compromise engineered safety systems.

Apparatus

5.1 *Environmental Test Chamber*, constructed of materials that are corrosion-resistant to the test solutions.

5.2 The equipment shall be capable of reproducing and continuously recording the temperature and pressure profiles of the DBA conditions.

5.3 A sufficient number of thermocouples shall be located in the test chamber to assure conformity to the test curve,

and so that both the temperature of the vapor phase and of the liquid phase (if present) can be recorded.

6. Preparation of Test Specimens

6.1 Determine the appearance of the test panels prior to testing by photo documentation or equivalent methods in order to provide a basis for post-test comparison. The testing requirements should indicate if this assessment will be done prior to shipping to the test facility.

6.2 Unless otherwise specified, a minimum of four samples shall be required to establish conformance of a given coating system on a given substrate, with two of the four samples being irradiated prior to testing in accordance with Test Method D 4082. Typical laboratory test panels are 2 by 4 by 1/8 in. for steel panels and 2 by 2 by 4 in. for concrete panels.

6.2.1 *Steel Panels*—Prepare in accordance with ANSI N512 or as necessary to duplicate actual conditions.

6.2.2 *Concrete Blocks*—Prepare in accordance with ANSI N512 or as necessary to duplicate actual conditions.

7. Procedure

7.1 Test Parameters:

7.1.1 Test coatings using the applicable curves from the latest Safety Analysis Report (SAR) identified by the owner for the specific containment. Illustrations of time-temperature-pressure test curves that simulate primary containment atmospheres during a DBA are shown in Figs. 1 and 2.

7.1.2 The curves depicted in Figs. 1 or 2 may be used if they represent conditions equal to or more severe than those DBA conditions anticipated.

7.1.3 The parameters of the curves may be simulated during testing as continuous functions or as an enveloping stepwise function.

7.1.4 Steam is used initially to achieve the desired thermal shock and to raise the test chamber and its environment to the prescribed test conditions. After equilibrium is achieved, the temperature of the test chamber is maintained by means

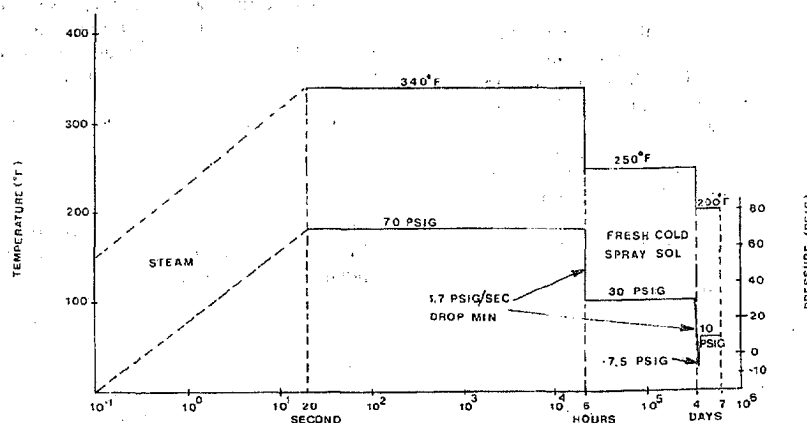


FIG. 1 Typical Design Basis Accident (DBA) Testing Parameters (Temperature-Time-Pressure)—BWR Drywell

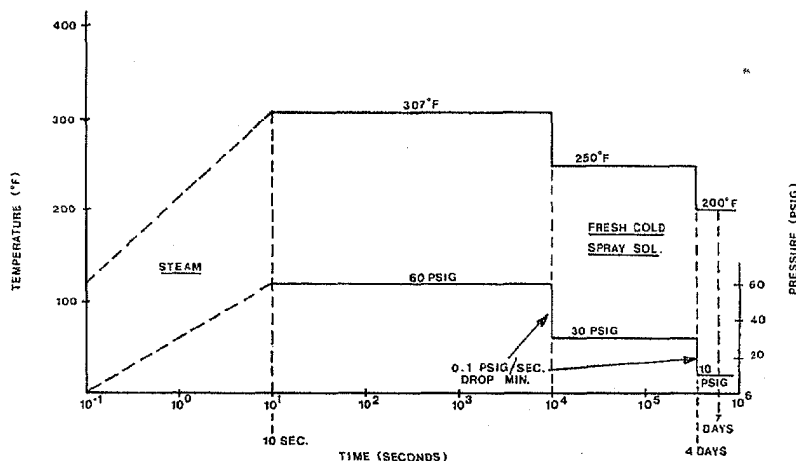


FIG. 2 Typical Design Basis (DBA) Testing Parameters (Temperature-Time-Pressure)—PWR Containment

of internal or external resistance, or both, heating elements, or other suitable means. The duration of steam injection should be minimized, as much as feasible, and the duration shall be recorded. Where inlet steam temperatures exceed 370°F, initial steam injection shall be no longer than 15 min.

7.2 Spray Solution:

7.2.1 Unless otherwise specified, use deionized water when testing under simulated DBA conditions.

7.2.2 Record the chemical composition of the spray solution before each test.

8. Examination and Report

8.1 Examination:

8.1.1 Examine and evaluate test specimens within 4 h and again after 14 days following removal from the test chamber for the following coating defects:

8.1.1.1 Delamination and peeling.

8.1.1.2 Cracking.

8.1.1.3 Blistering in accordance with Test Method D 714.

8.1.2 Unless otherwise instructed, disregard the condition of the edges and plane areas within 1/4 in. (6.4 mm) from the edges of the steel or concrete test surfaces, and the top and bottom ends of the concrete surfaces.

8.2 Report—Report the following information:

8.2.1 The results of the evaluation of each test specimen. Report for all sides of concrete blocks and front and back of steel panels.

8.2.2 The extent of each defect from 8.1. Report "none" if no defects are present.

8.2.3 Any observations of unusual appearances.

9. Acceptance Criteria

9.1 Peeling shall not be permitted.

9.2 Delamination shall not be permitted.

9.3 Cracking is not considered a failure unless accompanied by delamination or loss of adhesion.

9.4 Blisters shall be limited to intact blisters which are

completely surrounded by sound coating bonded to the surface.

9.5 An owner may establish acceptance criteria more stringent than above. The above criteria are meant to establish minimum standards only.

10. Documentation

10.1 Testing Procedures—Document each of the following:

10.1.1 A description of the test apparatus, temperature and pressure profiles, spray solution composition including pH, duration, frequency, and rate of spray solutions, and any other pertinent test conditions.

10.2 Test Agency:

10.2.1 The testing agency shall be responsible for the documenting, reporting, and certifying of all tests.

10.2.2 The testing agency shall be responsible for meeting applicable quality assurance requirements.

10.2.3 The testing agency shall be responsible for providing color photographic documentation of the test surfaces as required.

10.2.3.1 Photographs shall reflect the actual size as close as possible of the test specimens.

11. Repairability

11.1 Test repair coatings applied to significant areas within Service Level I in accordance with the requirements for radiation and DBA conditions.

11.2 The test shall include evaluation of the repair coating applied in accordance with the repair procedure over the intended surface preparation or the original qualified coating system, or both.

11.3 Significant areas shall be determined by the specifying or qualifying agency. Nonsignificant areas have been determined as being less than 2 ft² in an approximate 2000 ft² area; larger areas require a decision regarding significance.

Precision

2.1 Test equipment must be demonstrated to have the capability to reproduce the design time/temperature parameters within ± 10 s and $\pm 5^\circ$ F and the design pressure within

± 3 psig. In any test where the test equipment imposes variances in the pressure/temperature parameters that are outside this range, an analysis of the validity of these test results should accompany the test data.

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Standard Test Method for Chemical Resistance of Coatings Used in Light-Water Nuclear Power Plants¹

This standard is issued under the fixed designation D 3912; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method establishes procedures for the evaluation of the chemical resistance of coatings used in light-water nuclear power plants.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 714 Test Method for Evaluating Degree of Blistering of Paints²

D 1193 Specification for Reagent Water³

2.2 *NACE Standard:*

NACE TM-01-74 Laboratory Methods for the Evaluation of Protective Coatings Used as Lining Materials in Immersion Service⁴

2.3 *ANSI Standard:*

N512-1974 Protective Coatings (Paints) for the Nuclear Industry, Section 7⁵

3. Significance and Use

3.1 The specific chemical resistance tests to be performed are dependent upon the relative severity of the service conditions. Figure 1 should be used as a guide to chemical solutions commonly used in various types of facilities. These listed solutions should not be construed as all-inclusive. Specialized conditions may require chemicals or concentrations not listed. In such cases, applicable solutions may be used.

3.2 The lining test may be used to evaluate the resistance characteristics of coating systems for lining surfaces of tanks, vessels and similar facilities used in a light-water nuclear power plant.

4. Preparation of Test Specimens

4.1 Steel Panels:

4.1.1 Prepare steel panels in accordance with ANSI N512.⁶

4.1.2 Use carbon steel panels in lining tests at least 7 by 7 by 1/4 in. (178 by 178 by 6.4 mm) thick for Procedure A only.

4.2 *Concrete Blocks*—Prepare blocks in accordance with ANSI N512 with the exception that the top and bottom ends of the block shall be coated.

5. Sampling

5.1 Prepare individual specimens for testing in each of the pertinent solutions.

5.2 Prepare one additional specimen as a control. Multiple tests may be performed.

6. Procedure

6.1 Test in accordance with NACE TM-01-74, Procedure B, Immersion Testing, except use Procedure A, One-Side Testing (cold wall) when a significant temperature differential is anticipated across the coating film as for tank linings.

6.2 Lining Test:

6.2.1 Immerse lining test specimens in the appropriate test solutions listed in Fig. 1 or as otherwise specified for a minimum of 180 days.

6.2.2 Maintain a temperature of $72 \pm 5^\circ\text{F}$ ($22 \pm 3^\circ\text{C}$) unless otherwise specified by the purchaser.

6.2.3 Use deionized water with an initial resistivity of not less than $1 \text{ M}\Omega/\text{cm}$ at 77°F and with a total solids of more than 0.5 ppm, more than 0.15 ppm of chloride or more than 0.15 ppm of fluoride. Maintain the pH range from 6.0 to 8.0.

6.2.4 Change the deionized water daily during the first week and weekly thereafter. Measure any constituents leached out before the water is changed.

6.3 Chemical Exposure Tests:

6.3.1 Test the chemical resistance properties of the coatings by immersion in the appropriate test solutions listed in Fig. 1 or as otherwise specified for a time interval that shall vary as follows.

6.3.1.1 For simulated exposure to occasional splash and spillage on areas where the solution may evaporate in place, immerse the specimens for a period of 5 days in the listed reagent solutions.

6.3.1.2 For simulated exposure to decontamination solutions, immerse the specimens for a period of 24 h in the listed solutions.

6.3.1.3 For simulated exposures to corrosive fumes of the listed reagents, immerse the specimens for a period of 8 h.

¹ This test method is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters. Current edition approved May 30, 1980. Published July 1980.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁴ Available from the National Association of Corrosion Engineers, P.O. Box 218340, Houston, TX 77218.

⁵ Available from American National Standards Institute, 13th Floor, 11 W. 42nd St., New York, NY 10036.

⁶ An ASTM standard will be published in the future and will replace the reference to ANSI N512 in this standard.

CHEMICAL-EXPOSURE TEST RESULTS

Specimen No. _____ Date _____

Coating System: Primer _____ DFT _____

Intermediate _____ DFT _____

Finish _____ DFT _____

Batch Number(s) _____ Method of Application _____

Temperature _____ °F, Relative Humidity _____ % Curing Time _____ at _____ °F.

Surface Preparation _____

Substrate _____

Chemical	Concentration, weight %	8 h	24 h	5 days
General Service:				
Hydrazine (NH_2NH_2)	5			
Sodium borate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$)	5			
Distilled water				
Sodium hydroxide (NaOH)	5			
Contamination Solutions:				
Citric acid ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$)	5			
Sodium phosphate dibasic (Na_2HPO_4)	5			
Hydrogen peroxide (H_2O_2)	1.03			
Potassium permanganate (KMnO_4)	4.6			
Sodium fluoride (NaF)	2			
Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$)	2			
Sodium phosphate ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$)	11.5			

FIG. 1 Sample Form

6.3.2 Maintain a temperature of $72 \pm 5^\circ\text{F}$ (22 to 3°C) unless the service conditions require a higher temperature.

6.3.3 Use reagent grade chemicals and distilled water conforming to Specification D 1193, Type III, for all solutions.

Examination and Evaluation

7.1 Lining Test:

- 7.1.1 Examine the test specimens weekly for the first month and then monthly thereafter.
- 7.1.2 Inspect specimens immediately upon completion of the test period. Make a final inspection not later than 24 h after removal from the test solution.
- 7.1.3 Evaluate the test specimens at each inspection for peeling, delamination, blistering (Test Method D 714), discoloration, and softening.

7.2 Chemical Exposure Tests:

- 7.2.1 Examine the test specimens at the end of the test period or every 24 h, whichever is the shorter time.
- 7.2.2 Evaluate the condition of the specimens within 1 h after removal from the test solutions.
- 7.2.3 Evaluate the test specimens at each inspection as in 7.1.3.

8. Documentation and Report

- 8.1 Report all procedures and conditions relating to the test specimen preparation.
- 8.2 Document the testing procedure and test results. A suggested format is illustrated by Fig. 1.

9. Precision and Bias

- 9.1 These tests are qualitative in nature. Precision and bias are not definable.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials¹

This standard is issued under the fixed designation D 3924; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Keywords were added editorially in January 1991.

1. Scope

1.1 This specification defines the standard atmospheres for normal conditioning and testing of paint, varnish, lacquer, and related materials, at approximately ambient conditions.

NOTE 1—See Definitions E 41, Specification E 171 and Method E 337.

2. Referenced Documents

2.1 ASTM Standards:

E 41 Definitions of Terms Relating to Conditioning²

E 171 Specification for Standard Atmospheres for Conditioning and Testing Materials³

E 337 Test Method for Measuring Humidity with a Psychrometer (The Measurement of Wet- and Dry-Bulb Temperatures)⁴

3. Standard Atmosphere

3.1 Unless otherwise specified, conditioning and testing of coating materials known to be sensitive to variations in temperature or relative humidity shall be carried out in an atmosphere having a temperature of $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and a relative humidity of $50 \pm 5\%$. If closer tolerances are

required, $\pm 2^\circ\text{F}$ ($\pm 1^\circ\text{C}$) or even narrower limits, and $\pm 2\%$ relative humidity may be specified.

3.2 The standard room or cabinet shall be well-ventilated but free of drafts, dust, products of combustion, and laboratory fumes. Lighting conditions, unless otherwise specified, shall be that normally maintained in a room used for paint test purposes.

4. Standard Temperature

4.1 Unless otherwise specified, the standard test temperature shall be as specified in Section 3.

5. Room Temperature

5.1 A temperature in the range from 65 to 85°F (18 to 29.5°C) shall be called room temperature.

6. Conditioning and Testing

6.1 Unless otherwise specified in the test method, condition the organic coating materials or dried test films thereof and the test equipment for not less than 3 h in the appropriate atmosphere and verify the temperature of the material before conducting the test in the same atmosphere.

6.2 For routine testing where close temperature control is not critical, conduct all physical tests on organic coating materials or dried test films thereof at the room temperature of 65 to 85°F (18 to 29.5°C).

6.3 For referee testing, unless otherwise specified in the test method, conduct all physical tests on organic coating materials or dried test films thereof in the standard atmosphere as specified in Section 3.

7. Keyword

7.1 conditioning environment

¹ This specification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.20 on Quality Assurance and Statistics.

Current edition approved Sept. 2, 1980. Published November 1980.

² Annual Book of ASTM Standards, Vol 14.02.

³ Annual Book of ASTM Standards, Vol 15.09.

⁴ Annual Book of ASTM Standards, Vol 11.03 and 15.09.

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Standard Practice for Sampling Liquid Paints and Related Pigmented Coatings¹

This standard is issued under the fixed designation D 3925; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes methods of taking representative samples of fluid paint or pigmented coating products from containers of any type.

1.2 The sampling of dry powder paints, clear coatings, mixed solvents, and nonpigmented materials of any type is not covered in this procedure.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products²

3. Terminology

3.1 *Description of Term Specific to This Standard:*

3.1.1 *batch*—the quantity of liquid paint or coating produced in the final mixing operation after all production processes are complete. For example, when a number of pigment dispersions are reduced with additional vehicle together in a large tank, the resulting final mixture is one batch.

4. Significance and Use

4.1 Samples are taken from batches, lots, and shipments of paint in order to determine their uniformity and compliance with specification requirements. It is very important that these samples be of convenient and economical size and that they be representative of the batch of paint at the time it was filled into shipping or storage containers.

4.2 The time and effort necessary to ensure that the sample is representative of the original material will be repaid in reduction of laboratory work and elimination of possible rejections of acceptable material.

5. Sampling Considerations

5.1 The use of common sense and good judgment is important even in the apparently simple task of taking samples.

5.2 Use care to assure that all containers, agitating equipment, and sampling apparatus are *clean* and that they can in no way contaminate the sample being taken. Slight contamination of the paint sample may lead to false test results.

5.3 The sample container should be dry and not cooler than the temperature of the area in which the sample is to be taken.

5.4 Because pigmented coatings are dispersions and not solutions, finely divided pigment particles dispersed in the coating vehicle may settle upon standing. Consequently, thorough and careful agitation before sampling is necessary to restore the paint to its original, uniform condition. The method of agitating or stirring is therefore of prime importance.

5.5 As soon as samples of paint are taken from the shipping or storage container, place them in clean, nonreactive, dry, air-tight containers to prevent evaporation. Do not store samples in plastic bottles because volatile solvents may diffuse through the walls. Loss of volatile solvents may introduce errors in such tests as viscosity, weight per gallon, and nonvolatile content as well as other properties. If cap liners are used, they should also be nonreactive with the material. If the sample is self reactive or highly volatile, appropriate sample container precautions should be taken to prevent over-pressurization of the container.

5.6 When representative samples have been obtained and packaged in clean, closed containers, deliver them promptly to the testing laboratory. During the period between sampling and delivery to the testing laboratory it is important that samples be kept at temperatures from 40 to 100°F (5 to 40°C) because extremes of temperature may change properties of some paint products.

6. Procedure

6.1 Because of differences in physical properties, somewhat different procedures are required for agitating and sampling those paints containing water as the volatile component in comparison to those containing organic solvents. For coatings with no volatile ingredients, use the method applicable to materials containing organic solvents.

6.2 *Pigmented Coatings Containing Organic Solvents*—Materials in this group are of many different types but all contain organic solvents, not water, as their volatile portion and they are fluids, not dry powders.

6.2.1 *Small Containers:*

6.2.1.1 When the batch to be sampled is filled in small containers and batch numbers are marked on the container, put all containers from the same batch together. From *each batch* select at random 1 % of, but not more than five containers, using the next larger whole number if a fraction

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.20 on Quality Assurance and Statistics.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 3925 - 80. Last previous edition D 3925 - 81 (1985) ϵ 1.

² *Annual Book of ASTM Standards*, Vol 06.01.

results. For example, if there are 275 containers in a batch, select three for test.

6.2.1.2 After selection of the filled, unopened containers, thoroughly agitate or stir the contents by the best means available. Acceptable methods of mixing are mechanical shaking or stirring or hand stirring with a paddle, followed by "boxing," that is pouring back and forth between the original and a clean empty container. Mechanical shakers are desirable for most materials since there is thorough agitation in a closed container. To prevent evaporation, agitate in a closed container lacquers and other coatings containing a highly volatile solvent.

6.2.1.3 Before mechanical shaking, open the container and check to be sure that the pigment has not caked on the bottom of the container. If this condition exists, stir manually to break up the hard settling and then put the containers on the mechanical shaker. Agitate paints having a weight per gallon of 11 lb (1.3 g/mL) or less on the shaker for 10 min and those with a weight per gallon of more than 11 lb for 20 min.

6.2.1.4 Once the contents have been thoroughly agitated, pour half of the material into an empty container and take a 1 qt (1 L) (or smaller if taken from a container of 1 qt or less) sample from each half. Determine the weight per gallon on each sample in accordance with Test Method D 1475. The two determinations should not differ more than 0.5 %. If the results differ by more than this the paint is not thoroughly mixed. Return the material to the original container, stir again, and repeat the test.

6.2.2 Containers Larger than 5 Gal:

6.2.2.1 30 and 55-Gal Drums—From each batch select at random 5 % of but not more than three containers. Drums may be stirred satisfactorily by several means. With open-head types, mechanical or manual stirring may be used. Some drums contain their own agitators; drum shakers or rollers may also be used. After thorough agitation, take samples from the top and bottom of the drum and compare weights per gallon as described in 6.2.1.4.

6.2.2.2 250 to 500-Gal Containers (Tote Tanks)—Select

at random 25 % of all containers for test. Take samples from top and bottom of the container and compare weight per gallon determinations as described in 6.2.1.4.

6.2.2.3 Tank Wagons and Tank Cars—Sample each compartment of the wagon or car. Pigmented paints and coatings packaged in large containers are generally formulated to be essentially nonsettling. Therefore, take samples from the top, middle, and bottom of the container and make weight-per-gallon determinations before any vigorous stirring is done. If the resulting tests fall within the limits described earlier, no further agitation is necessary. Samples may be obtained with a Bacon-bomb sampler or a "thief" apparatus.

6.3 Pigmented Coatings Containing Water:

6.3.1 Handle pigmented coatings containing water (latex paints, etc.) in a slightly different manner from solvent-thinned coatings. Water-thinned paints, if stirred too vigorously, have a tendency to incorporate air bubbles, which sometimes result in changing the physical properties of the paint.

6.3.2 With the above consideration, take the samples in accordance with the same general procedure outlined in 6.2 for paints containing organic solvents. If it has been necessary to shake, stir, or agitate a water-thinned paint vigorously, deaerate the samples before the weight-per-gallon tests are run.

6.4 Sampling from Tanks at the Factory—Mix the material in the tank thoroughly before completely filling two 1-qt (1-L) containers. If the containers are to be filled from a valve on the bottom or side of the tank, mix the material, draw off at least 5 gal (20 L) through the valve and return to the tank before taking the sample.

6.5 Sampling During Filling of Containers at the Factory—After the material is thoroughly mixed in the tank and filling of containers has commenced, take a 1-qt (1-L) sample when about 25 gal (100 L) have been filled and another when about 25 gal remain to be filled.

7. Keyword

7.1 sampling

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Standard Guide for State and Institutional Purchasing of Paint¹

This standard is issued under the fixed designation D 3927; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

The committee responsible for this standard has voted its withdrawal. In the absence of substantial reasons that it should be continued, the Society will approve withdrawal from publication in February 1993.

Scope

1.1 This guide aids state and local governments to make logical decisions in purchasing architectural paints on the basis of both quality and cost.

1.2 This guide concerns itself primarily with architectural grade sales paints as listed in Annex A2.2 especially whites and tints. Emphasis on this group of products is justified by their preeminence in usage and the difficulty in describing in simple terms requirements applicable to them.

NOTE 1—With experience much of the logic and many of the techniques described herein can be found applicable to a broad range of architectural coatings.

1.3 Many different methods can be used to select coatings. Three methods are presented in this guide and the user may choose the method or combination of methods best suited to his needs. The three methods are:

	Sections
Vehicle Ingredients/Cost Ratio Method	4
Performance/Cost Ratio Method	5
Application Method	6

Referenced Documents

2.1 ASTM Standards:

- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts²
- D 523 Test Method for Specular Gloss²
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer²
- D 659 Method of Evaluating Degree of Chalking of Exterior Paints²
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints²
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints²
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products²
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature²

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.41 on Government Procurement Criteria for Commercial Paint and Coatings. Current edition approved Aug. 28, 1987. Published October 1987. Originally published as D 3927 - 80. Last previous edition D 3927 - 80.

² Annual Book of ASTM Standards, Vol 06.01.

- D 2197 Test Method for Adhesion of Organic Coatings by Scrape Adhesion²
- D 2243 Test Method for Freeze-Thaw Resistance of Water-Borne Paints²
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²
- D 2366 Test Method for Accelerated Testing of Moisture Blister Resistance of Exterior House Paints on Wood³
- D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints²
- D 2805 Test Method for Hiding Power of Paints by Reflectometry²
- D 3258 Test Method for Porosity of Paint Films²
- D 3359 Test Methods for Measuring Adhesion by Tape Test²
- D 3719 Test Method for Quantifying Dirt Collection on Coated Exterior Panels²
- D 3928 Test Method for Evaluation of Gloss or Sheen Uniformity²
- D 4062 Test Method for Leveling of Paints by Draw-Down Method²
- D 4400 Test Method for Sag Resistance of Paints Using a Multinotch Applicator²

3. Products

3.1 It is recommended that standard commercial products offered to the general public for a period of 2 years or produced and sold in accordance with applicable government specifications be considered for purchase by the methods described in Sections 4, 5, or 6.

4. Active Ingredients/Cost Ratio Method

4.1 This method utilizes, as an indication of quality, the coating composition provided by the manufacturer and is limited to interior and exterior wall paints listed in Annex A2.2.

4.2 Only those ingredients considered to contribute directly to quality are considered. The major active ingredients in most paints are vehicle solids or binder and primary hiding pigments.

4.2.1 The weight percent of vehicle solids may help to predict the scrubability or cleanability of a paint, but the quantity of pigment must also be considered. In general, the greater the ratio of vehicle solids to pigment, the better the scrubability and cleanability will be. The ratio of vehicle solids volume to pigment volume is a more useful parameter

³ Discontinued; see 1988 Annual Book of ASTM Standards, Vol 06.01.

but is not readily available. Moreover, each type of vehicle solids, such as alkyd in solvent-borne paints and vinyl or acrylic latex in waterborne paints, has its own properties and there are many other additives not shown that can affect scrubability, cleanability, and other properties.

4.2.2 The amount of primary hiding pigment, for example, titanium dioxide in a white or pastel gloss finish, is an indication of hiding power. Paints of equal depth of color (light reflectance) should be compared, since lightness affects visual hiding.

4.3 An advantage of the method is that no special equipment or experience is required. Even if extensive testing is possible, the active ingredients/cost ratio method is valuable for initial screening. The active ingredients/cost ratio for several types of paint can be combined into a single figure for each supplier with each paint's "Use Factor." (see Section 7 and Annex A5).

4.4 The active ingredients/cost ratio method is not as conclusive for flat paints that often have a high "Use Factor." The hiding (opacity) of flat paints, particularly latex paints, cannot be predicted from the amount of hiding pigments since these paints often obtain much of their hiding from air in a porous paint film (conventional dry hiding) or air incorporated in the components of the film (micro-void techniques). Consequently, performance tests should be made on latex flat paints whenever possible. The cost per gallon of those latex flat paints with acceptable performance can be used in conjunction with the active ingredients/cost ratio for this type of paint.

4.5 *Calculation of Ingredients/Cost Ratio*—Use the procedure in Annex A1 to calculate the ratio of active ingredients to unit cost of the paint. The greater the ratio, the more active ingredients a paint has per unit cost. In general, this will help determine if a paint is a good buy for the money, but may not determine the quality of a paint.

NOTE 2—A lower quality paint at a lower cost may be a good buy, but may not be of the desired quality.

5. Performance/Cost Ratio Method

5.1 Paint can be tested using standard test methods such as those suggested in Annex A2. Although there are many other test methods used in paint formulating and manufacture, the methods listed have been found to be most useful to the consumer in determining the quality of a product.

5.2 Since different products have different properties, specific test methods have been suggested for each type of product shown in Annex A2. The purchaser may eliminate some of these tests, if the property that the test measures is not important to him, or may add other tests.

5.3 Paint testing by the purchaser requires purchase of test equipment and some skill in performing and interpreting test results. However, a few basic tests such as hiding (opacity), porosity, or scrubability can be run with relatively inexpensive equipment and will help considerably in determining the quality of a paint.

5.4 The quality of a paint can be determined by analysis of test results. Some results are descriptive, such as "good" or "poor" while others are numerical but with large numbers from some test methods, such as 450 for scrubability, or small numbers from others, such as 0.990 in measuring hiding. Also with some tests the higher the numerical values,

the better the quality, but with others lower values indicate better quality. For these reasons the original results from the different tests cannot be directly combined into an equation to provide a numerical ranking.

5.5 If the purchaser wants a numerical value for overall quality to use in ranking various products, it is suggested that a scale from 0 to 10 be used for all test methods. Descriptive words can be converted to a numerical rating scale such as:

10	Excellent
8	Very good
6	Good
4	Fair
2	Poor
1	Very poor

Many ASTM test methods already use the 0 to 10 scale, but numerical results can be converted to this scale as shown in Annex A3.

NOTE 3—ASTM does not intend to define quality for the purchaser. The scales shown in Annex A3 are given as examples that can be modified by the purchaser, if desired.

5.6 Many of the important properties of a paint that can be tested and evaluated by a professional painter are listed in Annex A2. The purchaser can request that the painter note unusual behavior during the application of the coating; such judgemental rating of properties not normally tested can be used as an aid in the paint selection process.

5.7 Calculation of Performance/Cost Ratio

5.7.1 Use the procedure in Annex A4 to calculate the performance/cost ratio from the results obtained from laboratory tests or those supplied by painters, or both.

5.7.2 The calculated ratio will indicate if a paint has high performance relative to cost. It is possible to have a high relative ratio even though the overall quality may be low if the paint is low in cost. The overall quality of products should, therefore, enter into cost comparisons to ensure that they provide the quality desired by the purchaser. It is also possible to have a high overall quality rating for a paint and still have some property that is not acceptable to the purchaser. It is therefore suggested that minimum standards be established by the purchaser for those properties he considers important.

5.8 *Information Provided by the Supplier*—If the purchaser is not equipped to perform tests, he can request the manufacturer to supply test results that can be used for performance/cost evaluations. If the purchaser is equipped to run some simple tests or if a professional painter is available for evaluation, he need ask the manufacturer to supply data only for those properties requiring considerable skill or expensive equipment to measure.

6. Specification Method

6.1 Specifications can provide a useful, mutually satisfactory mechanism for listing essential quality requirements of a product. A specification is a document that attempts to describe precisely the requirements that a product must meet to be considered suitable for a particular use. Requirements may be based on composition of the product or its performance. Composition requirements are easier to monitor while performance requirements, which are preferred, are more difficult to establish and evaluate. Consequently, most coating specifications are a combination of both types.

6.2 Specifications establish the minimum level for a product, but if requirements are set too low it becomes difficult to purchase superior coatings that may be a better value. Specifications may encourage manufacturers to formulate special products that meet the specification but are different from those sold to the general public.

6.3 State and local government purchasers may write a specification for each product, but considerable knowledge of paint properties and how they are related to practical application and performance of the product is required. Government purchasers may use Federal specifications already prepared for many products.

7. Use Factor

7.1 A use factor can be determined by dividing the total

annual cost or quantity of each type of paint by the total cost or quantity of all paints used by the purchaser. These factors permit each type of paint to influence the evaluation of bids in direct proportion to the quantity used.

7.2 If a purchaser wishes to consider the purchase of a complete line of paint from one supplier, then all of the ingredient/cost or performance/cost ratios for each of the supplier's products can be averaged to give an overall value for each supplier's line of products. Since different products in a line may vary in quality, more emphasis should be given to those that the purchaser intends to use the most. This can be done by multiplying the ratio for each paint by the use factor as shown in Annex A5 to determine the weighted average.

ANNEXES

A1. CALCULATION OF INGREDIENT/COST RATIO

A1.1 Obtain from the supplier the weight per gallon of the paint or take the weight per gallon in accordance with Test Method D 1475.

A1.2 Obtain the weight percent of active ingredients from the supplier.

A1.3 The relative hiding efficiency of the most common white hiding pigments are as follows:

	Efficiency Factor
Rutile titanium dioxide	1.00
Anatase titanium dioxide	0.75

	Efficiency Factor
Lithopone	0.18
Zinc oxide	0.14

Consider all grades of high content rutile titanium dioxide as 100 % active pigment even though the actual percent of titanium dioxide in the pigment is less than 100 %, since the total pigment contributes to hiding efficiency.

A1.4 Example of Calculations:

Three latex paints A, B, and C are being considered for purchase

Ingredients ⁴	Composition Weight, %			×	Weight/gal			=	Active Ingredient, lb/gal		
	A	B	C		A	B	C		A	B	C
Titanium dioxide (83 % TiO ₂)	17		13		11.54	11.50	11.00		1.96		1.43
Titanium dioxide (90 % TiO ₂)		16				(for all ingredients)					
Lithopone		5								1.84	
Silicates	13	10	20							0.58	
Silica	9		9								
Calcium carbonate		8									
Vinyl-acrylic resin solids	13	13	9						1.50	1.50	0.99
Additives	2	2	2								
Water	46	46	47								

Paint	Hiding Pigment	Efficiency Factor	Active Hiding Pigments	Vehicle Solids	Total Active Ingredients/Gal	Cost/gal	Ingredients/Cost Ratio
A	1.96	×	1.00	1.50	3.46	5.00	0.69
B	1.84	×	1.00	1.50	3.44	7.00	0.49
C	0.58	×	0.18		0.10		
	1.43	×	1.00	0.99	2.42	3.40	0.71

Paints A and B both have about the same amount of active ingredients, but the cost of B is higher. Paint B may have acceptable quality but it could be eliminated because of cost.

Paints A and C both appear to be good buys, but C with its lower amount of active ingredients may have a lower quality than A and some performance tests would be recommended.

⁴ Chemical tests may be run to confirm the accuracy of composition presented by the manufacturer.

A2. RECOMMENDED TEST METHODS

Property	Test Method	Recommended for Paint Type	D 2805
Adhesion of organic coatings	D 2197	BCD	10
Adhesion by tape test	D 3359	DE	9
Brush application characteristics	painter	all	8
Blister resistance—accelerated testing of moisture blister resistance of exterior house paints on wood	D 2366	DE	7
Chalking of exterior paints	D 659	DE	6
Checking of exterior paints	D 660	DE	5
Color compatibility	in preparation	alt	4
Color difference of opaque materials	D 2244	all	3
Condition in container	painter	all	2
Consistency of paints using the stormer viscometer	D 562	all	1
Cracking of exterior paints	D 661	DE	0
Dirt pick-up	D 3719	DE	
Drying, curing, or film formation of organic coatings at room temperature	D 1640	CE	
Erosion of exterior paints	D 662	DE	
Flaking (scaling) of exterior paints	D 772	DE	10
Flow properties	painter	all	
Freeze-thaw resistance of latex and emulsion paints	D 2243	ABD	
Gloss—specular gloss	D 523	DE	
Hiding power of paints	D 2805	all	
Hiding power, practical	painter	all	A4.1
Hiding power—relative dry hiding power of paints	D 344	all	inside
Leveling characteristics of paint by draw-down method	D 4062	BC	Test
Porosity of paint films	D 3258	A	Test
Scrub resistance of interior latex flat wall paints	D 2486	A	Test
Sag resistance of paints using a multinoth applicator	D 4400	all	Test
Stain removal	in preparation	A	Test
Uniformity of gloss or sheen	D 3928	all	Test

A2.1 These tests may be performed at the discretion of the purchaser to ensure satisfactory performance.

A2.2 Types of Paint:

- A Interior latex flat
- B Interior latex gloss and semigloss
- C Interior solvent gloss and semigloss
- D Exterior latex gloss and flat
- E Exterior solvent-borne gloss and flat

A3. SUGGESTED 0 TO 10 SCALES FOR METHODS NOT ALREADY USING THIS SCALE⁴

D 2805 Hiding Power (Contrast Ratio)	D 3258 Porosity (Percent Reflectance Drop)	Flat ^a	Gloss or Semigloss (Wall Paints)
10 1.000	10 0 to 4	9 360 to 399	1350 to 1499
9 0.995 to 0.999	9 5 to 9	8 320 to 359	1200 to 1349
8 0.990 to 0.994	8 10 to 14	7 280 to 319	1050 to 1199
7 0.985 to 0.994	7 15 to 19	6 240 to 279	900 to 1049
6 0.980 to 0.984	6 20 to 24	5 200 to 239	750 to 899
5 0.975 to 0.979	5 25 to 29	4 160 to 199	600 to 749
4 0.970 to 0.974	4 30 to 34	3 120 to 159	450 to 599
3 0.965 to 0.969	3 35 to 39	2 80 to 119	300 to 449
2 0.960 to 0.964	2 40 to 44	1 79 to 40	150 to 299
1 0.955 to 0.959	1 45 to 49	0 0 to 39	0 to 149
0 0.950 to 0.954	0 50 to over		
D 2486 Scrubbability			
Flat ^a	Gloss or Semigloss (Wall Paints)		
10 400 or over	1500 or over		

⁴ The purchaser may wish to include a control material when paints are evaluated or a series of paints could be ranked to allow for variations in test methods or subjective evaluations.

^a See Footnote 5.

A4. PERFORMANCE/COST RATIO

A4.1 Three latex flat wall paints A, B and C being considered for purchase are tested for the four listed properties with the following results:
 Test 19—Hiding, Test Method D 2805
 Test 3—Brush Application, Painter
 Test 23—Porosity, Test Method D 3258
 Test 24—Scrubbability, Test Method D 2486

Method (from Annex A2)	19	3	23	24
Paint A	7	5	8	4
Rating (0 to 10)				
Overall rating (mean)				6.0
Cost per gallon				\$8.50
Performance/cost ratio				0.71
Paint B	8	4	7	5

Overall rating (mean)	6.0
Cost per gallon	\$7.50
Performance/cost ratio	0.80
Paint C	
Rating	9 6 2 5
Overall rating (mean)	5.5
Cost per gallon	\$6.85
Performance/cost ratio	0.80

A4.2 Paints A and B have the same overall performance rating, but paint A could be rejected because of its higher cost. Paints B and C have the same performance/cost ratio so they are both equal buys for the money. The purchaser, however, could decide that the value of 2 for the porosity of paint C is below his acceptable requirements for this property and reject this paint.

A5. USE FACTOR

A5.1 Paints from two different suppliers being considered for purchase are evaluated according to the active ingredients/cost ratio method with the following results:

Supplier 1					
Paint Type	Ingredients/Cost Ratio		Use Factor		Weighted Ratio
A	0.71	x	42.7 %	=	0.30
B	0.69	x	24.0 %	=	0.17
C	0.70	x	15.0 %	=	0.10
D	0.65	x	10.0 %	=	0.07
E	0.49	x	8.3 %	=	0.04
mean	0.648			Total	0.68

Supplier 2					
Paint Type	Ingredients/Cost Ratio		Use Factor		Weighted Ratio
A	0.65	x	42.7 %	=	0.28
B	0.49	x	24.0 %	=	0.12
C	0.69	x	15.0 %	=	0.10
D	0.70	x	10.0 %	=	0.07
E	0.71	x	8.3 %	=	0.06
mean	0.648			Total	0.63

^A Calculated from Annex A1.

A5.2 The ingredients/cost ratios of each supplier's line of paints averaged the same (0.648), but when these ratios are weighted according to a use factor, then supplier No. 1 is the better choice since paints with the better ingredient/cost ratios are the ones most used by the purchaser. However,

testing is the key to assurance of obtaining an acceptable product.

A5.3 A use factor can also be used to weigh paint lines evaluated by the performance/cost method.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Evaluation of Gloss or Sheen Uniformity¹

This standard is issued under the fixed designation D 3928; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the uniformity of gloss or sheen of a paint applied by brush to a test panel simulating an interior wall section. Variations in gloss or sheen that may be caused by short wet edge time, poor leveling, and pigment orientation or flotation during and after application are evaluated visually.

1.2 Because both the application and the panel evaluation are very subjective, this test method should be used only for comparative testing within one laboratory as the ratings assigned by different laboratories do not usually agree (see Note 1).

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 Paint is brush applied to a test panel using procedures and conditions that are most likely to cause nonuniformity of gloss or sheen. The dry film is then viewed under conditions that accentuate variations in gloss or sheen and the overall appearance is evaluated.

3. Significance and Use

3.1 When coatings are applied to large flat surfaces such as walls, the film thickness may vary or there may be small areas missed entirely (holidays) when the color of the fresh material and the previous film are very similar. It is much less costly if these areas can be touched up instead of having to repaint the entire surface.

3.2 Thickness is also affected in the locations where an area coated a short time before is joined with the area currently being coated (laps). An application technique that may affect the final appearance of the film is brushing or rolling the material in different directions in adjacent areas.

3.3 Both buyers and sellers are interested in determining whether a coating can be applied without exhibiting areas that differ in appearance because of laps and brush or roller marks, and whether it can be readily touched up to provide a uniform appearance in regard to gloss or sheen and all other aspects.

4. Apparatus

4.1 *Paint Brush*,² 2 in. (50 mm) wide with polyester bristles 2 3/4 in. (70 mm) long, chisel tip 7/16 in. (10 mm) thick. Previously used brushes are preferred.

4.2 *Paint Brush*,³ 1 in. (25 mm) wide with polyester bristles 2 1/4 in. (57 mm) long, chisel tip 9/16 in. (15 mm) thick. Previously used brushes are preferred.

5. Reagents and Materials

5.1 *Laminated Fiber Board*,⁴ 24 by 12 in. (610 by 300 mm) with at least one smooth side.

5.2 *Latex-Base Primer*.⁵

5.3 *Control Paint* (optional)—Since there are no standard panels, photographs, or paints for this test method, an agreed-upon paint of known uniformity of appearance should be included with the test paints as a control, unless the paints are only ranked.

6. Procedure

6.1 Apply the latex primer by spray to the entire face of a smooth side of the test panel so as to coat it uniformly at a spreading rate of about 450 ft²/gal (11 m²/L).

6.2 Air dry at least 18 h under standard conditions of 73.5 $\pm$ 3.5°F (23 $\pm$ 2°C) and 50 $\pm$ 5 % relative humidity and examine the panel for uniformity of primer application. If this appears satisfactory, divide the coated area into sections A and B as shown in Fig. 1.

6.3 Mount the panel vertically and position it as shown in Fig. 1 with section A to the right and section B to the left of the operator. Before painting, wet the brush with water if latex paints are to be tested or VM & P naphtha if the test paints are solvent reducible and shake out as much liquid as possible before using. Apply the paint under test to section A with the 2-in. (50-mm) brush (Note) at a spreading rate of 450 $\pm$ 25 ft²/gal (11 $\pm$ 0.5 m²/L). About 13 g are required to cover this area but the exact amount can be determined from the following equation:

$$g = [(A \times W)/S] \times 3.15$$

where:

A = area, in.²,

W = weight per gallon, lb/gal, and

S = spreading rate, ft²/gal.

² A polyester brush No. B-20CS from The Leneta Co., Ho-Ho-Kus, NJ, has been found suitable for this purpose.

³ A polyester brush No. B-10 from The Leneta Co. has been found suitable for this purpose.

⁴ Upson board, available from General Distributing Co., Lockport, NY, or The Leneta Co., has been found suitable for this purpose.

⁵ Similar to U.S. Federal Specification TT-P-650c Primer Coating Latex Base, White (for Gypsum Wallboard).

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved April 14, 1989. Published June 1989. Originally published as D 3928 - 80. Last previous edition D 3928 - 80 (1987).

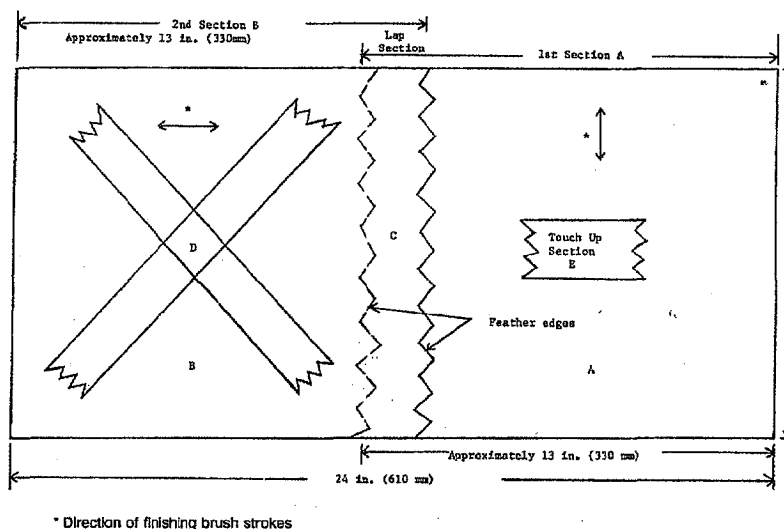


FIG. 1 Test Panel

or the following metric equation:

$$g = [(A_m \times D)/S_m] \times 1000$$

where:

A_m = area, m^2 ,

D = density, g/mL or kg/L , and

S_m = spreading rate, m^2/L .

Make the initial brush strokes in the direction of the length of the panel and finishing strokes across the width of the panel with the wet edge feathered. Complete the painting of this section in about 45 s.

NOTE—This test method can be modified for roller application by using a small 3-in. (75-mm) roller instead of the 2-in. (50-mm) brush to rank the uniformity of a series of paints when applied by roller. The 1-in. (25-mm) brush should still be used for touch up.

6.4 One minute after completing the painting of section A, apply paint to section B with the same brush still wet with the test paint. Use the same amount of paint and time to coat section B as for section A but make initial brush strokes across the width of the panel and finishing strokes along the length of the panel. Brush the finishing strokes into section A overlapping this section by about 2 in. (50 mm) (see Fig. 1, area C).

6.5 Thirty seconds after completing the painting of section B, use the still-wet brush but without additional test material to cross brush a figure X on this section as shown in Fig. 1, area D. Air dry for at least 18 h under standard conditions.

6.6 Using the 1-in. (25-mm) brush and the spreading rate as in 6.2, apply the test paint to an area of about 3 by 5 in. (75 by 125 mm) of section A (Fig. 1, area E). This is to simulate a wall section that has been touched up after the previous coat has dried. Allow the completed test panel to dry a minimum of 18 h under standard conditions.

6.7 View low gloss finishes at a 5° angle to the plane of the panel (85° to the normal) looking toward a strong light source. View gloss or semigloss coatings at 30° to the panel (60° to the normal). Rotate the panel 360° in the same plane while viewing in order to determine the overall appearance of the panel as viewed from different sides. Rate the test panel for gloss or sheen uniformity between sections using the following scale:

Rating	Score
Excellent	5
Very good	4
Good	3
Fair	2
Poor	1
Very poor	0

A rating of excellent means that there is no discernible variation in gloss or sheen between different areas of the panel. A rating of very poor means that there is a great variation in gloss or sheen between different areas.

6.7.1 Variations in gloss or sheen may be caused by various paint and substrate properties and application techniques so that lapped, cross-brushed, and touched up areas will not necessarily have the same degree of sheen or gloss uniformity and it may be advantageous to rate each of these areas separately.

7. Interpretation of Results

7.1 The uniformity in appearance of a wall can be estimated by evaluating the variation in gloss or sheen between areas A and C of the test panel.

7.2 The uniformity in appearance of a paint that has been applied by brush in different directions can be estimated by evaluating the variation in gloss or sheen between areas B and D.

7.3 The uniformity in appearance of touched-up areas can

estimated by evaluating the variation in gloss or sheen between areas A and E.

Precision

8.1 In an interlaboratory study of this test method in which one operator in each of six laboratories tested three paints of different levels of uniformity of appearance, all six

laboratories ranked the three paints in the same order of appearance but there were considerable differences between the actual ratings assigned by the laboratories to any one paint. The test method should, therefore, be used only for the comparative evaluation of a test paint against a mutually agreed upon control paint or for ranking a series of test paints.

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Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings¹

This standard is issued under the fixed designation D 3960; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice provides measures of the volatile organic compound (VOC) content of solventborne and waterborne paints and related coatings as determined from the quantity of material released from a sample under specified bake conditions.

NOTE 1—Other terms used as synonyms for volatile organic compound are photochemically reactive organic compound (PROC) and volatile organic substance (VOS).

1.2 This practice provides a guide to the selection of appropriate ASTM test methods for the determination of VOC content.

1.3 Certain organic compounds that may be released under the specified bake conditions are not classified as VOC as they do not participate in atmospheric photochemical reactions. Such nonphotochemically reactive compounds are referred to as exempt solvents in this practice.

1.4 VOC content is calculated as a function of (1) the volume of coating less water and exempt solvents, and (2) the volume of coating solids.

2. Referenced Documents

2.1 ASTM Standards:

- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products²
- D 2369 Test Method for Volatile Content of Coatings²
- D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings²
- D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings²
- D 3792 Test Method for Water Content of Water Reducible Paints by Direct Injection into a Gas Chromatograph²
- D 4017 Test Method for Water in Paints and Paint Materials by Karl Fisher Method²
- D 4457 Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph²
- D 5201 - 91 Practice for Calculating Formulation Physical Constants of Liquid Paints and Coatings²

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 3960 - 81. Last previous edition D 3960 - 90.

² Annual Book of ASTM Standards, Vol. 06.01.

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

2.2 Other Documents:

EPA Federal Reference Method 24—Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings⁴

EPA 450/3-84-019, U.S. Environmental Protection Agency Procedures for Certifying Quantity of Organic Compound Emitted by Paint, Ink, and Other Coatings⁴

EPA 450/3-83-013R, U.S. Environmental Protection Agency Glossary for Air Pollution Control of Industrial Coatings Operations⁴

Addition of Four Halocarbons to List of Negligibly Reactive Organic Compounds⁵

3. Terminology

3.1 Definitions:

3.1.1 *exempt solvent*—organic compounds that do not participate in atmospheric photochemical reactions.

3.1.2 *nonvolatile material*—the solid material remaining after volatiles have been removed from a coating under specified test conditions.

3.1.3 *volatile organic compound (VOC)*—any organic compound that participates in atmospheric photochemical reactions.

3.1.3.1 *Discussion*—The definition of VOC adopted by the U.S. EPA and a list of exempt compounds are included in Appendix X3.

3.1.4 *volatile organic compound content (VOC content)*—the mass of VOC released from a coating under specified test conditions.

3.1.4.1 *Discussion*—VOC content is expressed in this practice as a function of: (1) the coating volume less water and exempt solvents, and (2) the volume of coating solids.

4. Summary of Practice

4.1 The volatile content, density, water content, volume solids and exempt solvent content of the coating are determined in accordance with designated methods and instructions. For multicomponent coatings, the components are first mixed in the appropriate ratios and the applicable values determined on the mixture. These values are combined using

³ Annual Book of ASTM Standards, Vol. 15.05.

⁴ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁵ Published in the *Federal Register*, January 16, 1989, Vol. 54, No. 11, p. 1966. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

specified equations to calculate the VOC content of the coating.

NOTE 2—In Reference Method 24, the U.S. EPA defines a waterborne coating as any coating with more than 5 % water by weight in its volatile fraction, and requires/allows water determination for waterborne coatings only.

Significance and Use

5.1 This practice discusses applicable ASTM test methods used in the determination of the VOC content of paints and related coatings and provides equations for calculating the VOC content expressed as the mass of VOC in: (1) per unit volume of coating less water and exempt solvents, and (2) per unit volume of coating solids.

5.2 Volatile organic compound content is used to compare the amount of VOC released from different coatings used for the same application; that is, to coat the same area in the same dry film thickness (assuming the same application efficiency).

5.3 VOC content data are required by various regulatory agencies.

5.4 Only the expression of VOC content as a function of the volume of coating solids gives a linear measure of the difference in VOC released from different coatings used for the same application.

NOTE 3—Thus assuming the same transfer efficiency, a coating with VOC content of 3 lb of VOC/gal of solids would release 1/2 the VOC that a coating with 6 lb of VOC/gal of solids.

5.5 When VOC content is expressed as a function of the volume of coating less water and exempt solvents, the values obtained do not account for differences in the volume solids content of the coatings being compared; this expression, therefore, does not provide a linear measure of the difference in VOC emitted from different coatings used for the same application.

NOTE 4—Thus, a coating with VOC content of 3 lb of VOC/gal less water and exempt solvents would release about 85 % less VOC than a coating with 6 lb of VOC/gal of solids.

6. Nonvolatile and Volatile Content

6.1 Guide D 2832 includes suggested time/temperature drying schedules for the determination of the nonvolatile and volatile content of various types of coatings.

NOTE 5—For regulatory compliance testing, follow the method and conditions specified in the applicable regulation. Federal Reference Method 24 specifies the use of Test Method D 2369.

6.2 Test Method D 2369 includes a specific drying schedule and sample weight, and heating 1 h at $110 \pm 5^\circ\text{C}$ for the determination of the weight percent volatile content of solventborne and waterborne coatings.

6.2.1 For multicomponent coatings, Test Method D 2369 specifies the components should be mixed first, then the volatile content should be determined on the mixture. Test specimens are held in the aluminum dish for at least 1 h before baking.

NOTE 6—U.S. EPA will allow the test specimens to stand at room temperature for up to 24 h prior to baking at $110 \pm 5^\circ\text{C}$ for 1 h.

7. Water Content

7.1 To determine the water content of waterborne coatings two test methods are available:

7.1.1 In Test Method D 3792, a paint specimen is diluted with dimethyl formamide, an internal standard (2-propanol) is added, and an aliquot of the mixture is injected directly into a gas chromatograph.

7.1.2 In Test Method D 4017, a paint specimen is diluted with pyridine and a catalyst (1-ethylpiperidine) is added to obtain a rapid and definitive titration end point.

7.1.3 With waterborne multicomponent coatings, the components are first mixed in the appropriate ratios, then water content is determined using Test Methods D 3792 or D 4017.

8. Density

8.1 The density of the paint or coating at 25°C is determined in accordance with Test Method D 1475. Although both the pycnometer and weight-per-gallon cup are covered by the test method, and the former is more accurate and precise, the weight-per-gallon cup is recommended because of its speed and ease of use.

8.2 With multicomponent coatings, first mix the components in appropriate ratios in sufficient quantity to determine the weight-per-gallon using Test Method D 1475.

9. Exempt Solvents

9.1 In Test Method D 4457 an internal standard (1-propanol) is added to the test specimen, and then the specimen is injected directly into a gas chromatograph.

9.1.2 With multicomponent coatings, the exempt solvent content is determined on the mixture of the components.

10. Calculation of VOC Content

10.1 In this practice VOC content is expressed in two ways: (1) as the mass of VOC per unit volume of the coating less water and exempt solvents, and (2) as the mass of VOC per unit volume of coating solids. The following equations should be used to calculate VOC content and may be used for coatings both "as supplied" and "as applied" (see Note 7).

NOTE 7—For compliance with VOC regulations, the VOC content should be calculated after any thinning or dilution ("as applied"). Instructions for VOC calculations of such diluted coatings are available in EPA 450/3-84-019.

10.2 *VOC Content Expressed as the Mass of VOC per Unit Volume of Coating Less Water and Exempt Solvent:*

10.2.1 *General Expression:*

$$\text{VOC} = \frac{(\text{weight percent of total volatiles less water less exempt solvents})(\text{density of coating})}{100 \% - (\text{volume percent of water}) - (\text{volume percent of exempt solvents})}$$

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$$\text{VOC} = \frac{(W_o)(D_o)}{100 - V_w - V_{ex}} = \frac{(W_o - W_w - W_{ex})(D_o)}{100 - (W_w)(D_o/D_w) - (W_{ex})(D_o/D_{ex})} \quad (1)$$

where:

VOC = VOC content in g/L of coating less water and exempt solvent (see Note 8),

W_o = weight percent of organic volatiles, ($W_o - W_w - W_{ex}$),

W_v = weight percent of total volatiles, (100 % - weight % nonvolatiles, see Test Method D 2369),

W_w = weight percent of water, (see Test Methods D 3792 or D 4017),

W_{ex} = weight percent of exempt solvent, (see 3.1.3.1, Note 9, and Test Method D 4457),

V_w = volume percent of water, ($(W_w)(D_o/D_w)$),

V_{ex} = volume percent of exempt solvent, (see 3.1.3.1, Note 9), ($(W_{ex})(D_o/D_{ex})$),

D_o = density of coating, g/L, at 25°C, (see Test Method D 1475),

D_w = density of water, g/L, at 25°C, (0.997×10^3), and

D_{ex} = density of exempt solvent g/L, at 25°C, (see Test Method D 1475).

NOTE 8—To convert from g/L to lb/gal, multiply the result (VOC content) by 8.345×10^{-3} (lb/gal/g/L). To convert g/L to kg/L, divide the result by 10³.

NOTE 9—See Appendix X2.1 and X2.2 for comments on coatings containing one or more exempt solvents.

10.2.2 *Solventborne Coatings*—Calculate the VOC content in grams of VOC per litre of coating less water and exempt solvents using the appropriate equation:

10.2.2.1 For solventborne coatings that do not contain water or exempt solvents:

$$\text{VOC} = \frac{(W_o)(D_o)}{100} \quad (2)$$

10.2.2.2 For solventborne coatings that contain an exempt solvent but do not contain water (see 3.1.2.1 and Note 2):

$$\text{VOC} = \frac{(W_v - W_{ex})(D_o)}{100 - (W_{ex})(D_o/D_{ex})} \quad (3)$$

10.2.2.3 For solventborne coatings that contain water but do not contain exempt solvents (see 3.1.3.1 and Note 2):

$$\text{VOC} = \frac{(W_v - W_w)(D_o)}{100 - (W_w)(D_o/D_w)} \quad (4)$$

10.2.2.4 For solventborne coatings that contain both an exempt solvent and water, use Eq 1 in 10.2.1 (see 3.1.3.1 and Note 2).

10.2.3 *Waterborne Coatings*—Calculate the VOC content in grams of VOC per litre of coating less water and exempt solvent using the appropriate equation.

10.2.3.1 For waterborne coatings that contain no exempt solvents, use Eq 4 in 10.2.2.3 (see 3.1.3.1 and Note 2).

10.2.3.2 For waterborne coatings that contain exempt solvents, use Eq 1 in 10.2.1 (see 3.1.3.1 and Note 2).

10.3 *VOC Content Expressed in Terms of the Mass of VOC per Unit Volume of Coating Solids (Nonvolatiles)*:

10.3.1 Calculate the VOC content in grams of VOC per litre of coating solids according to the following equation:

$$\text{VOC}_m = \frac{(W_o)(D_o)}{V_n} \quad (5)$$

where:

VOC_m = VOC content in g/L of coating solids,

W_o = $W_v - W_w - W_{ex}$ (terms defined as in 10.2.1), and

V_n = volume percent of nonvolatile content of the liquid coating, (see Test Method D 2697, and Notes 10 and 11).

NOTE 10—The EPA Reference Method 24 does not include an analytical method for determining V_n , but states that the value be calculated from the coating manufacturer's formulation.

NOTE 11—An expression for calculating formula V_n from the coating formulation is included in Appendix X2.3, Eq X2.1.

11. Keywords

11.1 VOC; VOC calculations; VOC content; VOC content of paint; VOC content, test precision

APPENDIX

(Nonmandatory Information)

X1. AUTOMOTIVE COATINGS SUPPLIERS ROUND ROBIN

X1.1 A round robin was conducted at the laboratories of automotive coatings suppliers for determination of VOC using Practice D 3960. The analysts involved were persons experienced in running all the test methods involved in VOC determination. The data was analyzed statistically in accordance with Practice E 180. As was suspected from previous round robins conducted to evaluate Practice D 3960 (which involved some laboratories not familiar with these test methods), when well experienced analysts conduct the tests, the precision data is much improved.

X1.2 The interlaboratory study involved four laboratories and six samples; four solvent-reducible automotive topcoats and two water-reducible automotive topcoats. One operator in each of the four laboratories analyzed the sample in duplicate on 2 different days. The following duplicates, repeatability, and reproducibility coefficients of variation were obtained.

Automotive Topcoats	Duplicates, %	Repeatability, % (Within Laboratory)	Reproducibility, % (Between Laboratory)
Solvent-reducible	0.86	1.62	2.86
Water-reducible	3.94	5.29	9.75

X2. CALCULATION OF VOC CONTENT (SECTION 10)

X2.1 Measurement of Exempt Solvent Content—The value of the weight percent or volume percent of exempt solvent in the VOC expression (Eqs 1, 3, and 5) can be obtained using Test Methods D 4457 and D 1475 if the solvent is methylene chloride or 1,1,1-trichloroethane.

X2.2 Two or More Exempt Solvents—For solvent or water-reducible coatings containing more than one exempt solvent, the values for Ex and $(Ex)(D_o/D_w)$ to be used in Eq 3 (10.2.1, 10.2.2.2, 10.2.2.4 or 10.2.3.2) are the summations of the values of Ex and $(Ex)(D_o/D_w)$ for each individual solvent. Also, the value for Ex to be used in 10.3.1 to determine V_o in Eq 5 is the summation of the individual values.

X2.3 Volume Nonvolatile Content—The volume percent nonvolatile content, V_n , in Eq 5 (10.3.1) can be calculated from the summation of the individual contributions of each component in the coating formulation ("p" components) using the following equation (Notes X2.1 and X2.2):

$$V_n, \% = \sum_{j=1}^p [(V_n)](V_j/100) \quad (X2.1)$$

where:

V_n = volume percent of nonvolatile component "j," [(100 %) × (volume of nonvolatiles of "j" per unit volume of "j")], and

V_j = volume percent of component "j" in the coating [(100 %) × (volume of "j" used)/(total volume of coating)].

NOTE X2.1—Instructions for calculating the value for the formula weight of volatile solids (or formula volume percent nonvolatile) content of the coating are provided in Practice D 5201.

NOTE X2.2—Eq X2.1 is meant to clarify the equation (11-4) currently in the EPA certification manual EPA-450/3-84-019 for the expression of calculated volume percent nonvolatile content, V_n .

X2.4 Amount of VOC in a Coating Expressed in Terms of Mass of VOC per Unit Volume of Coating Including Water and Exempt Solvents—The amount of volatile organic compounds in both solvent- and waterborne coatings can be expressed in terms of the mass of volatile organic compounds per unit volume of coating including water and exempt solvents according to the following equation (Note X2.3)

$$VOA = \frac{(W_o)(D_o)}{100} \quad (X2.2)$$

where:

VOA = Amount of volatile organic compounds in g/L of coating including water and exempt solvents.

NOTE X2.3—Calculation of the amount of volatile organic compound based on the total volume of coating (including water and exempt solvents), as illustrated in Eq X2.2, does not provide a measure of the amount of VOC that would be released from two coatings used for the same application (that is, to coat the same area to the same dry film thickness assuming the same application efficiency for each coating) when one or both of the coatings contain water or exempt solvents. These units do not identify which of the coatings will release the greater amount of VOC as they treat water and exempt solvents as coating solids. These units, therefore, have not been used, recommended or accepted by U.S. EPA for demonstration of compliance with VOC content regulations as such calculations yield misleading results for coatings that contain water or exempt solvents.

NOTE X2.4—The expression in X2.2 is useful for the calculation of the mass of VOC released per unit of time (for example, the mass of VOC per unit of volume including water and exempt solvents times the volume of total coating used per unit of time). This expression may also be useful for certain labeling purposes where the amount of VOC per unit container is desired.

APPENDIX 3

X3. REGULATORY DEFINITION OF VOC

X3.1 The regulatory definition of volatile organic compounds (VOC) has evolved into the following statement by the EPA.^{4,5,6}

X3.1.1 Volatile Organic Compounds (VOC)—Any organic compound that participates in atmospheric photochemical reactions; that is, any organic compound other than those which the administrator designates as having negligible photochemical reactivity. VOC may be measured by a reference method, an equivalent method, an alternative method, or by procedures specified under any subpart. A reference method, an equivalent method, or an alternative method, however, may also measure nonreactive organic

compounds. In such cases, an owner or operator may exclude the nonreactive organic compounds when determining compliance with a standard. The administrator has designated the following organic compounds as negligibly reactive: methane, ethane, 1,1,1-trichloroethane (methyl chloroform), methylene chloride (dichloromethane), dichlorotrifluoroethane (HCFC-123), tetrafluoroethane (HCFC-134a), dichlorodifluoroethane (HCFC-141b) and chlorodifluoroethane (HCFC-142b), trichlorofluoromethane (CFC-11), dichlorodifluoromethane (CFC-12), chlorodifluoromethane (CFC-22), trifluoromethane (CFC-23), trichlorotrifluoroethane (CFC-113), dichlorotetrafluoroethane (CFC-114), and chloropentafluoroethane (CFC-15).

NOTE X3.1—The regulatory definition, under the control of the U.S. EPA, can and will change. A proposal to add compounds to this list is under consideration. To ensure currency, contact the local air pollution control agency.

⁴ Manual on Determination of Volatile Organic Compounds in Paint, Inks, and Related Products, *ASTM Manual Series, MNL 4*, ASTM, May 1989.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Practice for Selection of Coating Specimens for Appearance Measurements¹

This standard is issued under the fixed designation D 3964; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

In making appearance measurements, it is important that the specimens selected be representative of the material of interest. Once selected, the specimens must be carefully examined to determine if they are suitable for measurement, and if not, they must be cleaned or otherwise prepared. Careful attention to these factors is necessary if the measurements are to be valid.

Scope

1.1 This practice provides a guide to selection of specimens for appearance measurement as well as a discussion of factors to be considered in their preparation for measurement. Standardized selection and presentation procedures will assist in achieving agreement between evaluations carried out in different laboratories as well as helping to achieve better correlations between visual evaluations and instrumental measurements.

NOTE 1—This standard is not a practice for preparation of test panels for coatings; see Test Methods D 823.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

- 2.1 *ASTM Standards:*
D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
E 284 Definitions of Terms Relating to Appearance of Materials³

Terminology

- 3.1 *Definitions:*
3.1.1 For definitions of terms used in this practice, refer to definitions E 284.

Significance and Use

- 4.1 Many of the specimens that are regularly submitted to measurement depart in some degree from the ideal require-

ments. For this reason instrumental readings are affected importantly by the manner in which specimens are selected and presented for measurement. Reproducible measurements are facilitated by standardization and control of test conditions.

5. General Requirements

5.1 *Specimen Size*—The minimum size is dictated by the size of the specimen port of the instrument to be used for measurement. When an instrument provides a choice of specimen port sizes, use the largest port that can be completely covered by the specimen. A large measured area helps to minimize the effect of any small area nonuniformity and is therefore more likely to provide results that agree with the involuntary averaging that takes place when specimens are observed visually. A large specimen also permits the operator to make measurements on several areas of the specimen when desired thereby providing further specimen averaging.

5.2 *Opacity*—An opaque specimen shall be selected whenever possible for gloss and color evaluation. When the specimen is translucent or transparent, the following points should be considered:

5.2.1 *For Gloss Evaluation*—The specimen shall be sufficiently thick that a secondary reflection from the back or second surface of the specimen cannot enter the receptor optics of the glossmeter. When thin transparent specimens must be measured, adopt one of the following procedures:

5.2.1.1 Back the specimen with a light absorbing material of the same refractive index as the specimen, and in optical contact with it.

5.2.1.2 Use an agreed upon specimen thickness, including coating and substrate and place a black backing behind the specimen.

5.2.2 *For Color Evaluation*—The choice of backing of even slightly translucent specimens will affect their measurement. Use the most applicable of the backing techniques that follow. In each case it is essential that the backing material and color be reproducible, stable, and durable for reliable results.

5.2.2.1 Back the specimen with the same material with which it will be backed in its intended use.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved Oct. 31, 1980. Published December 1980.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.02.

5.2.2.2 Back the specimen with identical or similar material.

5.2.2.3 Back the specimen with a neutral material whose luminous reflectance is essentially the same as that of the specimen being evaluated.

5.2.2.4 Back the specimen with a black surface such as a painted panel or black glass.

5.2.2.5 Back the specimen with a white backing of known reflectance.

6. Physical Properties

6.1 The specimen shall be rigid and have a plane surface. Specimens of thin, flexible material that can bulge or sag when presented to an instrument require special holding equipment. When the specimen is moderately flexible, a plane surface may be achieved by pressing the specimen against the instrument with a flat, rigid object. The pressing technique must be reproducible. A vacuum plate or specially designed clamping device has proven satisfactory in some applications. Note that specimens coated on thin plastic film or paper may trap air between the specimen and the holder (vacuum plate or clamping device) causing the specimen to billow (blisters) resulting in erroneous readings.

6.2 *Surface Texture*—Some coatings have pronounced surface texture, making it desirable to rotate a specimen in its own plane. Those with marked directionality, usually due to the production process, will have different values when measured in different directions. Good practice dictates that measurements be made with the plane of measurement of the instrument both parallel to as well as perpendicular to the process direction.

7. Cleaning of Specimens

7.1 The specimen must be clean. There should be no dirt, dust, oil or foreign material on the surface that will affect the

instrumental evaluation of the specimen. If the specimen appears to require cleaning, the procedure should be agreed upon and it should be carried out with care. Any cleaning of a low-gloss or fragile surface may scratch or polish the surface and thus change the appearance of the specimen and render it useless for measurement. A high-gloss specimen can usually be washed with clear water and blotted with untreated lens tissue or paper towel. If the specimen is durable, a mild nonfluorescent, nonionic detergent that does not leave a film can be used with a soft cloth or bristle brush. If the specimen is durable and has an oily or stubborn contamination that should be removed, reagent grade isopropyl alcohol may be used. Follow all cleaning techniques with a warm water rinse and drying by blotting, using untreated lint-free paper towel.

NOTE 2—Ceramic or porcelain enameled instrument calibration standards require periodic cleaning and the above procedures can generally be used safely.

8. Handling Specimens

8.1 Handle specimens by their edges. Do not allow objects to contact the front surface of the specimen. When placing the specimen on the instrument, avoid sliding it across the specimen port.

9. Report

9.1 When the specimens submitted for appearance measurements depart from the ideal requirements, report the following:

9.1.1 Their condition (Section 6),

9.1.2 Method of cleaning (Section 7),

9.1.3 Method of backing (5.2.1, 5.2.2, and 6.1), and

9.1.4 Indicate direction of specimen submitted for measurement or averaging of data, or both, when required by directionality (6.2).

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Standard Practice for Interlaboratory Testing of Paint and Related Materials¹

This standard is issued under the fixed designation D 3980; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This practice covers the interlaboratory testing of paint and related materials. The information presented is intended to assist task groups in the effective planning of round-robin test programs.

1.2 The various parts appear in the following order:

	Sections
Part A—Definitions	3
Part B—Planning	4 to 11
Part C—Statistical Analysis	12 to 16
Part D—Analysis Presentation	17 to 20

1.3 This practice, in spite of its length, is not intended to cover all aspects of statistical design and analysis. For example, the analysis of variance used in the practice as the basis for estimating the precision is abridged in comparison to that normally used for establishing the significance of experimental factors. The publications listed in the appendix should be consulted for further information or explanations. For convenience, comparable practices developed by other ASTM technical committees are also shown.

Referenced Documents

2.1 ASTM Standards:

- D968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive²
- D3793 Test Method for Low-Temperature Coalescence of Latex Paint Films²
- E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

PART A—TERMINOLOGY

Definitions

3.1 The statistical terms used in this practice are defined, as nearly as possible, in every day language. For convenience, the terms are listed in order of use in this practice.

3.2 *experimental design*—the complete plan for conducting an investigation or an interlaboratory study of materials, processes, or test methods. It is conveniently set forth in the form of a single comprehensive table showing number of laboratories, materials to be tested, test methods, number of operators, testing conditions, time of testing, and

degree of replication and repetition. The experimental design should provide the basis for an efficient statistical analysis of the results (see Part B, Planning).

3.3 *replication*—the execution of multiple chemical or physical determinations on the same specimen or physical measurements on the same panel at the same time, as closely as possible. Two replicates are called duplicates, three are called triplicates, etc. The individual results are not suitable for estimating precision but are used only to ensure by comparison with similar measurements that there is not a gross error among them (see 15.1.3.3).

3.4 *repetition*—The execution of multiple physical measurements on the same panel or chemical determinations at different times, or physical measurements on different panels, areas of large structures, or specimens of a liquid sample within a short time interval, to establish the precision of a method. In most cases the measurements are the mean of replicates as defined in 3.3.

3.5 *population*—the totality of observations on or determinations of a certain property or component obtained by the same procedure; theoretically, an infinite collection of measurements on a given item of interest but, practically, a large number of measurements of the item. Thus, when statistical procedures are used to determine whether or not two (or more) materials or test methods differ significantly with respect to some measurable property, in effect, the determination is whether these materials belong to the same or to different populations.

3.6 *sample*—a randomly selected subset of a population intended to be representative of it so as to enable obtaining an estimate of the property or composition of the entire population. The reliability of such an estimate can be expressed in terms of confidence limits (see 3.19). Through tests of significance (see 3.21), it is possible to state, with a specified degree of confidence, whether two or more samples are drawn from the same or from different populations.

3.7 *average*—a typical numerical value that attempts to summarize or reflect the location of a group of observations by a single number. While it is a measure of central tendency, it does not provide information on the variability of the individual observations. The following are different types of averages: arithmetic mean, weighted mean, algebraic mean, geometric mean, harmonic mean, median, and mode.

3.7.1 *mean (arithmetic)*—the value obtained by dividing the sum of a set of observations or results by their number. This value is an estimate of the mean of the parent population. Although the arithmetic mean is affected by extreme values and therefore may not be typical, it is amenable to statistical treatment and is the most commonly used average.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.20 on Sampling, Statistics, etc.

Current edition approved May 27, 1988. Published December 1988. Originally published as D 3980 - 81. Last previous edition D 3980 - 87.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 15.05.

3.7.2 *median*—the central observation in an ordered set that contains an odd number of observations or the mean of the two central observations in an ordered set with an even number of observations. The median is not distorted by extreme values so that where the observations are not symmetrically distributed it is a more representative average than the mean.

3.8 *expected value*—the mean of an infinite series of independent determinations on the same item obtained by the same procedure; also thought of as the true value that would be obtained if all extraneous variations were eliminated. In practice, it can be closely approached by a finite series of tests, the number of tests needed being dependent on the extent of variation (scatter) of the observed values.

3.9 *accepted reference value*—a value that serves as an agreed-upon reference for measured values. It is derived as a theoretical value based on scientific principles or an assigned value based on experimental work by (a) competent national or international organization(s).

NOTE—When the accepted reference value is the theoretical value, it is sometimes referred to as the "true" value.

3.10 *accuracy*—the degree of agreement of individual or mean measurements with the expected or accepted reference value.

3.11 *error*—the deviation of a measured value or group of measured values from the expected value or accepted reference value.

3.11.1 *random error*—the chance variation that occurs in all experimental measurements despite the closest possible control of all factors. It is characterized by the fortuitous occurrence of both positive and negative deviations from the mean or the expected value. The algebraic mean of the deviations is zero in a large series of measurements.

3.11.2 *bias*—a systematic as opposed to a random error that contributes to the difference between the estimated mean of the population and the accepted reference or the true value.

3.12 *dispersion*—the variability (scatter) of the observed values, usually measured about some central value such as the mean.

3.13 *range*—the difference between the lowest and highest values in a set of observations or results. The range is a simple but useful indicator of the variability of test results.

3.14 *outlier*—an extreme value far enough from other results in a series to be suspected of not belonging to the particular population under consideration. Statistical criteria are available for judging whether a given outlier should be included in the analysis of results or discarded (see Section 13). Also called "wild," "rogue," "maverick."

3.15 *variance*—a measure of the dispersion of a series of results around their mean. The variance of the parent population is estimated by summing the squares of the individual deviations from the mean and dividing by one less than the number of results.

DISCUSSION—Since the variance of a set of results and the estimated variance of the parent population are not equal because of different divisors (n for the set, $n - 1$ for the population), the same symbol should not be used for both.

3.16 *standard deviation*—a measure of the dispersion of a series of results around their mean, defined as the positive square root of the variance.

DISCUSSION—The advantages of the standard deviation are that it is in the same units as the original results and is of the same order of magnitude as the deviations from the mean. The standard deviation is the basis for most statements of precision and may be obtained from an analysis of variance of results of an interlaboratory test program.

3.16.1 *pooled value*—the weighted mean obtained by combining in accordance with statistical rules two or more separate values shown to be members of the same (homogeneous) population. Variances, if shown to be homogeneous by appropriate statistical criteria, may be combined by weighting each variance in accordance with its degrees of freedom, summing them and dividing by the sum of the degrees of freedom. Standard deviations must not be arithmetically averaged; they must be squared to convert them to variances, combined, and the square root taken to obtain the pooled value.

3.16.2 *coefficient of variation*—a measure of relative precision calculated as the ratio of the standard deviation to the mean of a series of values, expressed in percent.

3.17 *degrees of freedom*—in a set or subset of observations, the number of values minus the number of constraints. In general, there is only one constraint—the number of values. For example, a set of n observations can be arranged into g groups. The group degrees of freedom is then $g - 1$ because n is already determined. Similarly the total df is $n - 1$ (see Table 8).

3.18 *probability*—the chance of occurrence of an event expressed in terms of a relative frequency, a fraction, or a percent. For example, the probability that a tossed coin will land head up is one in two, or 0.5, or 50 %.

3.19 *confidence limits*—the limits on either side of the mean value of a group of observations that will, in a stated fraction or percent of the cases, include the expected value. Thus the 95 % confidence limits are the values between which the population mean will be situated in 95 out of 100 cases.

3.20 *confidence level*—the probability level (usually with reference to a statistical table) with which the significance of differences between measurements is assessed. Thus, a difference that is significant at the 95 % level (0.05 probability level, sometimes called significance level) would represent a real difference 95 times in 100; however, 5 times in 100 a difference this large might arise by chance even with identical material because of experimental error.

3.21 *significant difference*—a difference between two values, means, or variances that is shown by tests of significance to be a real difference at the stated level of confidence.

3.21.1 *Student's t test*—a statistical test for assessing the significance of a deviation from the mean or of the difference between two means. The t -value is based on the ratio of the observed deviation, or difference, to the standard deviation and is compared with tabulated t -values that indicate the frequency with which a difference of this magnitude should occur by chance in samples having the appropriate degrees of freedom.

3.21.2 *variance ratio (F) test*—a statistical test for assessing the significance of the difference between two or more variances. The F test (named in honor of R. A. Fisher) is based on the ratio of the larger variance to the smaller

Experimental Design for Evaluation of a Single Method Using One Type of Apparatus

Laboratory	Operator	Repeat	Replicate	Materials				
				1	2	3	etc. to	M
I	A	1	a				"	
			b					
		2	a					
			b					
	B	1	a					
			b					
II	A	1	a					
			b					
		2	a					
			b					
	B	1	a					
			b					
etc. to	A	1	a					
			b					
		2	a					
			b					
	B	1	a					
			b					
L	A	1	a					
			b					
		2	a					
			b					
	B	1	a					
			b					

FIG. 1 Example of Design for Interlaboratory Study

material before dividing them into groups to be distributed among the laboratories. Where necessary, the same specimens may be sent in turn to each participating laboratory.

NOTE 2—Test panels are frequently prepared in one laboratory to establish the variability only of measurements obtained in different laboratories. Consequently, if panel preparation has a significant effect on test results, the resulting precision will be better than where panels are independently prepared by each laboratory. If, because of difficulty in obtaining a sufficient quantity, the same test panels are circulated to participants, the test method may, depending upon the variability of panel preparation and the sensitivity of the test method, also appear more precise than if different panels were sent to each participant.

11.4.1 Effect of Aging—If the specimens are such that their properties may change noticeably in a few days or weeks, coordinate the tests among the laboratories so that each laboratory performs the test on specimens of the same age.

11.5 Report Form—Supply each laboratory with report forms like Fig. 2 to ensure that all results and pertinent information are reported in a uniform manner. In addition

to space for measurement results, the form should provide space for such information as: relative humidity, temperature, instrument type, deviations from the specified procedure, unusual observations, and constructive comments, as required.

PART C—STATISTICAL ANALYSIS OF INTERLABORATORY TEST RESULTS

12. Scope

12.1 Appropriate statistical methods are described for computing the correlation, precision, and sensitivity of a test procedure from interlaboratory test results.

12.1.1 Outliers—For each material, ranges are computed between replicates run at each time by all laboratories, between means for each time for each laboratory, and between laboratory means of all repeats (usually two or three). Results from the different materials exhibiting similar variability or having similar mean values can be grouped and the ranges for each material calculated. For each type of range, the results are examined for suspected outliers and the

Report Form for Interlaboratory Tests
ASTM Subcommittee _____ Task Group _____
Interlaboratory Test on _____

Test Method 1 ^A											
Material	Operator ^B	Repeat 1					Repeat 2				
		Replicate				Mean	Replicate				Mean
		a	b	etc. to	r		a	b	etc. to	r	
1	A										
	B										
2	A										
	B										
etc. to											
M	A										
	B										

Date received _____ Date tested _____

Method of application (if required) _____

Film thickness (if required) _____

Film thickness method (if required) _____

Test temperature _____ Relative humidity _____

Equipment description:

Name _____ Model No. _____

Comments:

Name _____ Laboratory _____

^A If more than one test method being studied.
^B If more than one operator per laboratory or locale.

FIG. 2 Report Form

test described in 13.3 applied, as illustrated in 15.1.3.4 and 15.1.3.5, to determine whether any values can be rejected.

12.1.2 *Correlation*—If a test procedure is being evaluated for its ability to provide results that correlate with the known property values of materials, a correlation coefficient is computed and tested for significance.

12.1.3 *Precision*—The precision of a test procedure is determined by computing intralaboratory and interlaboratory variances from the test results obtained for each material. If the variances for the materials are homogeneous, they are pooled and an overall intralaboratory standard deviation and an overall interlaboratory standard deviation are calculated and used in the precision calculations. If the variances are inhomogeneous, means of providing homogeneity (such as use of the coefficient of variation or other transformations of the results) are given.

12.1.4 *Sensitivity*—A sensitivity criterion (figure of merit) is computed and used as a measure of the ability of a test procedure to distinguish between materials differing in the property being measured.

13. Test for Outliers

13.1 An outlying observation, or outlier, is one that appears to deviate markedly from other members of the set

in which it occurs. This outlier may be a single value, a mean, or a range.

13.2 When the experimenter is clearly aware that a gross deviation from prescribed experimental procedure has taken place, the resultant observation should be discarded, whether it agrees or not with the rest of the results. If a reliable correction procedure is available the observation may be corrected and retained.

13.3 In many cases, evidence for deviation from the prescribed procedure consists of the discordant value itself. In such cases a statistical test is applied to determine if the doubtful value should be discarded. A simple technique for this purpose is the rejection quotient procedure. It consists of (1) ranking the values (single observations, means, or ranges) in order of their magnitude; (2) taking the difference between the extreme value and the nearest value to it, and (3) dividing that difference by the appropriate range of the values. The resulting quotient, Q , is compared with the established rejection quotient (RQ) for the number of items in the series of values. If Q exceeds the RQ , the extreme value is suspect and may be discarded. Table 1 gives the equations for computing Q and the RQ values for three significance levels as a function of the number of items in the test results. It is recommended that outliers be discarded on the basis of the 0.01 significance level for intralaboratory results and the

TABLE 1 Table for Testing Extreme Values

If Q exceeds the rejection quotient, the suspect value may be rejected.

Number n of Values	Ratio	Significance Level ^a		
		0.10	0.05	0.01
3	$Q = \frac{X_2 - X_1}{X_n - X_1}$ or $\frac{X_n - X_{n-1}}{X_n - X_1}$	0.941	0.970	0.994
4		0.765	0.829	0.926
5		0.642	0.710	0.821
6		0.560	0.628	0.740
7		0.507	0.569	0.680
8	$Q = \frac{X_2 - X_1}{X_{n-1} - X_1}$ or $\frac{X_n - X_{n-1}}{X_n - X_2}$	0.544	0.608	0.717
9		0.503	0.564	0.672
10		0.470	0.530	0.635
11		0.445	0.502	0.605
12		0.423	0.479	0.579
13	$Q = \frac{X_3 - X_1}{X_{n-2} - X_1}$ or $\frac{X_n - X_{n-2}}{X_n - X_3}$	0.563	0.611	0.697
14		0.539	0.586	0.670
15		0.518	0.565	0.647
16		0.500	0.546	0.627
17		0.483	0.529	0.610
18		0.469	0.514	0.594
19		0.457	0.501	0.580
20		0.446	0.489	0.567
21		0.435	0.478	0.555
22		0.426	0.468	0.544
23		0.418	0.459	0.535
24		0.410	0.451	0.528
25		0.402	0.443	0.517
26		0.396	0.436	0.510
27		0.389	0.429	0.502
28		0.383	0.423	0.495
29		0.378	0.417	0.489
30		0.373	0.412	0.483

^a Risk or probability of rejecting a valid extreme result.

0.05 significance level for interlaboratory results. When results from several materials with similar property levels are being analyzed, all the results must be included in the calculation of Q . Also, the test should be applied only once to a set of laboratory results. Although two or more values can be rejected at the same time, the remaining results should not again be tested for outliers.

13.4 Following is an example of the use of the rejection quotient procedure for evaluating extreme values:

13.4.1 In the evaluation of the precision of a test method, a sample was tested within a single laboratory twice on each of two days by each of two operators for a total of eight determinations. Differences between replicates for both operators on both days are ranked as follows:

Rank	1	2	3	4	5	6	7	8
Difference	0.7	3.4	3.9	4.6	5.2	6.0	6.7	7.0

One of the differences between replicates (the smallest difference) appears to be suspect.

From Table 1, for eight items:

$$Q = (X_2 - X_1)/(X_{n-1} - X_1) = (X_2 - X_1)/(X_7 - X_1) = (3.4 - 0.7)/(6.7 - 0.7) = 2.7/6.0 = 0.450$$

To reject one of the eight differences, Q must exceed 0.544 at the 0.10 significance level (90 % confidence level), 0.608 at the 0.05 significance level (95 % confidence level), and 0.717 at the 0.01 significance level (99 % confidence level). Since a significance level of 0.01 should be used for intralaboratory test results, it is concluded that the difference of 0.7 is not rejectable and must be included in the calculation of test precision.

14. Correlation

14.1 To be useful, a test method must provide values for materials that either relate directly to the known property or component values for these materials or that relate to the values from another test method that is related to the known values. The degree of association between the values of a test method under study and the values of a standard test method can be determined by computing the correlation coefficient.

14.1.1 The correlation coefficient, r , may be defined by the equation:

$$r = \frac{\sum(X - \bar{X})(Y - \bar{Y})}{[\sum(X - \bar{X})^2 \sum(Y - \bar{Y})^2]^{1/2}} = \frac{\sum XY - (\sum X \sum Y / n)}{[\sum X^2 - (\sum X)^2 / n][\sum Y^2 - (\sum Y)^2 / n]^{1/2}}$$

where:

X = value obtained for a material by standard test method,
 Y = corresponding value obtained for a material by new test method,

$\bar{X}$ = mean value obtained for a material by standard test method,

$\bar{Y}$ = mean value obtained for a material by new test method, and

n = number of values obtained with each test method.

14.1.2 The reliability of the correlation coefficient depends on the number of materials tested as well as the degree of linear relation between the two test methods. It should be pointed out that a calculated r -value represents the relation of the two test methods only over the range of values obtained. Thus, if the materials selected for evaluating a test method differ only slightly in their property or component levels, the calculated r may not correspond to the correlation that would be obtained if materials differing widely in these values had been tested.

14.1.3 The correlation coefficient, r , is dimensionless and its values range from 1.0, perfect direct relation, to -1.0, which indicates perfect inverse relation. An r of 0 shows no relation between the two test methods. Whether a value of r between 0 and 1 is significantly different from 0 can be determined from Table 2 that gives the critical values at several probability levels as a function of the degrees of freedom, $n - 2$, where n is the number of materials tested.

14.1.4 The correlation coefficient squared, r^2 , (called the coefficient of determination) provides additional information about the degree of correlation between the two test methods. It is a useful concept in the sense that r^2 is equal to the fraction of the variation of the dependent variable Y that may be ascribed to the effect of the independent variable X . To claim good correlation exists between two test methods, a value of at least 0.9 for r is required, since $r^2 = 0.81$ indicates only 81 % of the variation in Y can be ascribed to X . If a value of 0.7 or less is obtained for r , the degree of association is considered to be inadequate for assuming that the two procedures are measuring the same property. It should be pointed out that a high value for r does not guarantee that values of Y can be predicted precisely from values of X .

14.1.5 An example of the computation of the correlation coefficient to determine the degree of association between values of two test procedures is shown in Table 3. This single laboratory test consisted of measuring the drying time of 10

TABLE 2 Critical Values for Correlation Coefficient r

df ^a	Probability Level		
	0.10	0.05	0.01
1	0.988	0.997	1.00
2	0.900	0.950	0.990
3	0.805	0.878	0.959
4	0.729	0.811	0.917
5	0.669	0.754	0.874
6	0.621	0.707	0.834
7	0.582	0.666	0.798
8	0.549	0.632	0.765
9	0.521	0.602	0.735
10	0.497	0.576	0.708
11	0.476	0.553	0.683
12	0.457	0.532	0.661
13	0.441	0.514	0.641
14	0.426	0.497	0.623
15	0.412	0.482	0.605
16	0.400	0.468	0.590
17	0.389	0.455	0.575
18	0.378	0.444	0.561
19	0.368	0.433	0.549
20	0.360	0.423	0.537

^a df = the number of degrees of freedom (number of pairs minus 2).

samples by two test procedures. The drying time values shown represent the mean of two replicate determinations. Since the computed $r = 0.853$ and $r^2 = 0.728$, it is concluded that the degree of association is adequate to assume that both procedures are measuring drying time because r exceeds

TABLE 3 Example of Calculating Correlation Coefficient

Sample	Drying Time, h	
	Thumb and Forefinger	Thumb and Balance
1	7	7
2	7.5	7.5
3	8	7
4	9	9
5	8	8
6	6	6.5
7	8	8
8	8	9
9	9	9
10	8.5	8.5

Sample	X	Y	XY	X ²	Y ²
1	7	7	49	49	49
2	7.5	7.5	56.25	56.25	56.25
3	8	7	56	64	49
4	9	9	81	81	81
5	8	8	64	64	64
6	6	6.5	39	36	42.25
7	8	8	64	64	64
8	8	9	72	64	81
9	9	9	81	81	81
10	8.5	8.5	72.25	72.25	72.25
Total	79.0	79.5	634.5	631.5	639.75

$$r = \frac{\Sigma XY - (\Sigma X \times \Sigma Y / n)}{\left(\frac{\Sigma X^2 - (\Sigma X)^2 / n}{n} \right)^{1/2} \left(\frac{\Sigma Y^2 - (\Sigma Y)^2 / n}{n} \right)^{1/2}}$$

$$= \frac{634.5 - (79 \times 79.5 / 10)}{\left(\frac{631.5 - 624.1}{10} \right)^{1/2} \left(\frac{639.75 - 632.025}{10} \right)^{1/2}}$$

$$= \frac{634.5 - 628.05}{\left(\frac{7.4}{10} \right)^{1/2} \left(\frac{7.725}{10} \right)^{1/2}} = \frac{6.45}{57.165^{1/2}}$$

$$= \frac{6.45}{7.561} = 0.853$$

$$r^2 = 0.728$$

0.765, the critical value at the 99 % confidence level for 10 samples (8 degrees of freedom) in Table 2.

14.2 There may be instances where no actual instrumental values are available for the materials tested, only their relative ranking being known. In these cases, a rank correlation coefficient, r_s , can be computed to express the degree of relationship between the ranking of the X values of one test method and the Y values of the other test method.

14.2.1 In computing r_s , the results from the two procedures are each arranged in order of performance and each material assigned a rank. In the cases where two or more materials have the same rank, each is assigned the value corresponding to the mean of the ranks that would be assigned to them if the rankings were sequential. By taking the differences, RD , between the two sets of rankings for the materials, r_s is calculated from Spearman's equation as follows:

$$r_s = 1 - [6\Sigma(RD)^2 / n(n^2 - 1)] = 1 - [(6\Sigma(RD)^2 / (n^3 - n))]$$

where:

n = number of materials and

RD = difference between the rankings of a material.

A test to determine if r_s is significantly different from 0 can be made conveniently with Table 4 in which $\Sigma(RD)^2$ values at two probability levels are given as a function of the number of pairs.

14.2.2 An example of the procedure for computing a rank correlation coefficient is shown in Table 5. In this example, the performance of 10 products subjected to two test procedures is represented by a relative ranking of their performance. It is seen from the significance table of $\Sigma(RD)^2$ as a function of number of pairs that the value of 11 obtained in this example is highly significant (99 %) since $\Sigma(RD)^2$ is less than 39 for the probability level of 0.01. It is concluded that the degree of association between the two test procedures is good.

15. Precision

15.1 The precision of a test method is expressed in two terms, repeatability and reproducibility. Paragraphs 15.1.1 and 15.1.2 provide the complete mathematical formulas for calculating precision, but a shortened procedure for analysis of results from a balanced design is given in 15.1.3. Where values have been discarded as a result of application of the test for outliers in 13.1, the mean of all the values for the respective determination can be used to replace them to retain a balanced design. It should be recognized that the analysis used in this practice is abridged because interactions between factors are disregarded. Consequently, some information that could be obtained from a complete analysis is sacrificed, as in Practice E 180.

15.1.1 *Repeatability (Intralaboratory)*—Test repeatability is determined from the estimated intralaboratory variances computed from the repeat determinations made within each laboratory on each material.

15.1.1.1 For this practice, the equation defining the estimated variance, s_w^2 , (Note 3) within a single laboratory is as follows:

$$s_w^2 = \Sigma(X_i - \bar{X})^2 / (n_R - 1)$$

TABLE 4 Critical Values of $\Sigma(RD)^2$ for the Rank Correlation Method

n	Probability Level		n	Probability Level	
	0.05	0.01		0.05	0.01
5	0-40	...	23	1 187-2 861	980-3 088
6	4-66	0-70	24	1 370-3 230	1 115-3 485
7	12-100	4-108	25	1 570-3 360	1 287-3 913
8	22-146	10-158	26	1 789-4 061	1 475-4 375
9	40-200	24-216	27	2 028-4 524	1 681-4 871
10	61-269	39-291	28	2 287-5 021	1 906-5 402
11	86-352	58-382	29	2 569-5 551	2 149-5 971
12	121-451	84-498	30	2 873-6 117	2 414-6 576
13	163-565	115-613	31	3 199-6 721	2 700-7 220
14	213-697	154-756	32	3 550-7 362	3 008-7 904
15	272-848	201-919	33	3 926-8 042	3 338-8 630
16	342-1 018	257-1 103	34	4 328-8 762	3 693-9 397
17	423-1 209	322-1 310	35	4 757-9 523	4 073-10 207
18	515-1 423	398-1 540	36	5 213-10 327	4 478-11 064
19	621-1 659	484-1 796	37	5 698-11 174	4 908-11 964
20	740-1 920	583-2 077	38	6 213-12 065	5 366-12 912
21	873-2 207	695-2 385	39	6 758-13 002	5 853-13 907
22	1 022-2 520	820-2 722	40	7 334-13 986	6 367-14 953

Use of Table—If the observed total, $\Sigma(RD)^2$, for the number of ranked pairs, n , is equal to or less than the lower tabular value, or equal to or greater than the higher tabular value, the correlation is significant for the indicated probability. High values correspond to negative correlations, low values to positive correlations.

TABLE 5 Example of Rank Correlation Calculation

Rank	Ranking of Panels Subjected to									
	Natural Exposure					Cyclic Condensation				
1					B					A and B
2					C					
3					A				D	
4					D				C	
5					E and F				E	
6									F and H	
7					H					
8					G				G	
9					I				J	
10					J				I	

Panel Designation	A	B	C	D	E	F	G	H	I	J	Total
Natural exposure rank	3	1	2	4	5.5	5.5	8	7	9	10	55
Cyclic exposure rank	1.5	1.5	4	3	5	6.5	8	6.5	10	9	55
Rank difference (RD)	1.5	-0.5	-2	1	0.5	-1	0	0.5	-1	1	0
Rank difference squared (RD) ²	2.25	0.25	4	1	0.25	1	0	0.25	1	1	11

$$\text{Rank total} = (n(n+1)/2) = (10(11)/2) = 55$$

$$r_s = 1 - [6\Sigma(RD)^2 / (n^3 - n)] = 1 - [(6 \times 11) / (1000 - 10)] = 1 - (66/990) = 1 - 0.067 = 0.933$$

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technique, described

in 15.1.3.

If the results

are not balanced,

the computations

are made on the

repeat determinations

performed within each

laboratory on each

material.

15.1.1.3

If, by inspection,

the intralaboratory

variances of

all laboratories for

a material appear to

be homogeneous

(approximately the

same) they can be

pooled to give a

single intralaboratory

variance for that

material.

15.1.1.4

If the intralaboratory

variances for a

material do

not appear to be

homogeneous, a

statistical test, such

as the Cochran⁴

or Bartlett⁵ tests,

should be used to

determine whether

the variances can

be pooled.

15.1.1.5

Pool the variances

for all the materials

(unless they appear

to be nonhomogeneous)

to give a single intra-

material by the analysis of variance technique, described in 15.1.3. If the results are not balanced, the computations are made on the repeat determinations performed within each laboratory on each material.

15.1.1.3 If, by inspection, the intralaboratory variances of all laboratories for a material appear to be homogeneous (approximately the same) they can be pooled to give a single intralaboratory variance for that material.

15.1.1.4 If the intralaboratory variances for a material do not appear to be homogeneous, a statistical test, such as the Cochran⁴ or Bartlett⁵ tests, should be used to determine whether the variances can be pooled.

15.1.1.5 Pool the variances for all the materials (unless they appear to be nonhomogeneous) to give a single intra-

⁴ Cochran, W. G., and Cox, G. M., *Experimental Designs*, John Wiley and Sons, New York, NY, 1957.

⁵ Youden, W. J., *Statistical Methods for Chemists*, John Wiley and Sons, New York, NY, 1951.

laboratory variance for all materials. Obtain the intralaboratory standard deviation, s_w , by taking the square root of the variance and report.

15.1.1.6 Compute the maximum allowable difference (MAD) by multiplying s_w by the q factor (Table 6) for the appropriate number of replicates in each laboratory and the degrees of freedom $(n_m \text{ materials}) \times (n_L \text{ laboratories}) \times (\text{time} - 1)$. In case test results are missing, compute the $n_L(t-1)$ degrees of freedom for each material and total them.

15.1.1.7 Examine the results from each laboratory for each material to establish whether all results are within the MAD for intralaboratory determinations. If this range is exceeded and outliers were not previously rejected in accordance with 13.3, discard the discordant results and recalculate the variance, standard deviation, and MAD. Compare with the original values to establish whether the rejection is justified; for example, a marked reduction in MAD with the elimination of only one set of results is evidence that they differ significantly from the other results and their retention would adversely affect the precision of the test method.

15.1.1.8 If the intralaboratory variances for the materials do not appear homogeneous but appear to be dependent on the level of the mean value of the materials, homogeneity may be achieved generally by converting the standard deviations to coefficients of variation:

$$v_w = 100 s_w / \bar{X}$$

15.1.1.9 If the coefficients of variation are homogeneous, pool them in accordance with the equation, as follows:

$$v_p = \left[\frac{(n_1 - 1)v_1^2 + (n_2 - 1)v_2^2 + \dots + (n_m - 1)v_m^2}{(n_1 - 1) + (n_2 - 1) + \dots + (n_m - 1)} \right]^{1/2}$$

where:

v_1 = coefficient of variation for Material 1, and
 n_1 = number of values used to establish v_1 , etc.

When $n_1 = n_2 = n_m$, this condenses to $(\sum v_i^2 / n_m)^{1/2}$

where:

$\sum v_i^2$ = sum of squared coefficients, and
 n_m = total number of materials.

Calculate the maximum allowable difference in percent relative by multiplying the coefficient of variation by q for the number of repeats and degrees of freedom.

15.1.1.10 If the coefficients of variation do not appear to be homogeneous, a transformation of the test results (such as to logarithms, arc sine, or square root) may provide homogeneous variances.

15.1.1.11 If homogeneous variances or coefficients of variation cannot be obtained from the intralaboratory results, calculate the precision for appropriate levels of the material value.

15.1.2 *Reproducibility (Interlaboratory)*—Test reproducibility is determined from the estimated interlaboratory variance which is the variance of the mean values obtained by the laboratories (s_L^2) plus the intralaboratory variance. Thus, interlaboratory variance is as follows:

$$s_b^2 = (s_w^2 / n_R) + s_L^2$$

where n_R = number of repeats in each laboratory.

NOTE 4—The subscript, b, derives from the previously used term "between-laboratories".

15.1.2.1 The equation defining s_L^2 and the procedures for its computation are discussed in 15.1.1.1 and 15.1.1.2.

15.1.2.2 The equation defining the variance of laboratory means, s_L^2 , is as follows:

$$s_L^2 = \Sigma(\bar{X} - \bar{\bar{X}})^2 / (n_L - 1)$$

where:

$\bar{X}$ = mean obtained for a material by a laboratory,
 $\bar{\bar{X}}$ = grand mean of the values obtained for a material by all laboratories,
 n_L = number of laboratories,
 $(\bar{X} - \bar{\bar{X}})^2$ = sum of squares, ss, and
 $n_L - 1$ = degrees of freedom, df.

To facilitate calculations, the equation is converted to

$$s_L^2 = \frac{\Sigma \bar{X}^2 - [(\Sigma \bar{X})^2 / n_L]}{n_L - 1}$$

15.1.2.3 When the results obtained in the interlaboratory test are from a balanced design and there are no missing values, compute the sum of squares and variance for each material by the analysis of variance technique, described in 15.1.3. Calculate the interlaboratory variance using the equation given in 15.1.2.

15.1.2.4 Pool the interlaboratory variances for all materials (unless they appear nonhomogeneous) to give a single interlaboratory variance for the test. Calculate the interlaboratory standard deviation, s_b , and report.

15.1.2.5 Compute the maximum allowable difference by multiplying s_b by the q factor (Table 6) for the appropriate number of laboratories and the degrees of freedom $(n_L - 1)$ times the number of materials.

15.1.2.6 Examine the results from all laboratories to establish that the range of laboratory means does not exceed the MAD. If the range is exceeded and laboratory outliers were not previously rejected, discard the discordant results and recalculate the MAD as in 15.1.1.7.

15.1.2.7 If the interlaboratory variances for the materials do not appear to be homogeneous, but appear to vary with the level of the means of the materials, convert the standard deviations to coefficients of variation, and if homogeneous, pool them as shown in 15.1.1.8 and 15.1.1.9. Calculate the MAD in percent by multiplying the coefficient of variation by q for the number of laboratories and degrees of freedom.

15.1.2.8 If the coefficients of variation do not appear homogeneous, a transformation of the test results (such as to logarithms, arc sine, or square root) may provide homogeneous variances. If neither variances nor coefficients of variation are homogeneous for the interlaboratory results, calculate the reproducibility for each level of test value of the materials as in 15.1.1.11.

15.1.3 *Analysis of Variance of Balanced Results:*

15.1.3.1 Where a design recommended in Part B has been used and balanced results are available (that is, no missing values from a balanced design or missing values replaced by the appropriate mean value), the analysis of variance technique should be used to compute the intralaboratory (within)

TABLE 6 Values of the Studentized Range (*q* values) for the 95 % Confidence Level

DFA	n ^B	2	3	4	5	6	7	8	9	10
1	1	18.0	26.7	32.8	37.2	40.5	43.1	45.4	47.3	49.1
2	2	6.09	8.28	9.80	10.89	11.73	12.43	13.03	13.54	13.99
3	3	4.50	5.88	6.83	7.51	8.04	8.47	8.85	9.18	9.46
4	4	3.93	5.00	5.76	6.31	6.73	7.06	7.35	7.60	7.83
5	5	3.61	4.54	5.18	5.64	5.99	6.28	6.52	6.74	6.93
6	6	3.46	4.34	4.90	5.31	5.63	5.89	6.12	6.32	6.49
7	7	3.34	4.16	4.68	5.06	5.35	5.59	5.80	5.99	6.15
8	8	3.26	4.04	4.53	4.89	5.17	5.40	5.60	5.77	5.92
9	9	3.20	3.95	4.42	4.76	5.02	5.24	5.43	5.60	5.74
10	10	3.15	3.88	4.33	4.66	4.91	5.12	5.30	5.46	5.60
11	11	3.11	3.82	4.26	4.58	4.82	5.03	5.20	5.35	5.49
12	12	3.08	3.77	4.20	4.51	4.75	4.95	5.12	5.27	5.40
13	13	3.06	3.73	4.15	4.46	4.69	4.88	5.05	5.19	5.32
14	14	3.03	3.70	4.11	4.41	4.64	4.83	4.99	5.13	5.25
15	15	3.01	3.67	4.08	4.37	4.59	4.78	4.94	5.08	5.20
16	16	3.00	3.65	4.05	4.34	4.56	4.74	4.90	5.03	5.15
17	17	2.98	3.62	4.02	4.31	4.52	4.70	4.86	4.99	5.11
18	18	2.97	3.61	4.00	4.28	4.49	4.67	4.83	4.96	5.07
19	19	2.96	3.59	3.98	4.26	4.47	4.64	4.79	4.92	5.04
20	20	2.95	3.58	3.96	4.24	4.45	4.62	4.77	4.90	5.01
24	24	2.92	3.53	3.90	4.17	4.37	4.54	4.68	4.81	4.92
30	30	2.89	3.48	3.84	4.11	4.30	4.46	4.60	4.72	4.83
40	40	2.86	3.44	3.79	4.04	4.23	4.39	4.52	4.63	4.74
60	60	2.83	3.40	3.74	3.98	4.16	4.31	4.44	4.55	4.65
120	120	2.80	3.36	3.69	3.92	4.10	4.24	4.36	4.47	4.56
∞	∞	2.77	3.32	3.63	3.86	4.03	4.17	4.29	4.39	4.47

DFA = degrees of freedom associated with standard deviation or coefficient of variation.

n^B = number of repeats for repeatability or number of laboratories for reproducibility.

interlaboratory (between) variances.⁶ In the analysis, it must be ascertained that the intralaboratory variances and mean values are consistent among the laboratories. Interlaboratory values are not necessarily consistent, depending upon the precision of the test method(s). Lack of consistency may arise from a variety of causes, such as materials or equipment. As noted in 3.3, the individual replicates are used only for determining gross errors in test results.

15.1.3.2 Following is an example of the application of the analysis of variance to interlaboratory test results. In this example, one robin, one laboratory coated, baked, and distributed the panels to five co-operators. Each co-operator received three panels and was asked to determine the Knoop hardness at three different times within a given period to eliminate fatigue effects. The results submitted are given in Table 7.

15.1.3.3 The first step (whether using the analysis of variance or not) is to examine each replicate value for possible errors. This is most easily done by calculating the range in results for each time and comparing the ranges at similar levels in results. In the example given here, in which the range appears to increase with increasing hardness, the results for each enamel must be compared separately, but in cases where the values for all materials are at about the same level all results must be considered together. The calculated ranges in the example are presented at the bottom of Table 7.

⁶ A Basic software program to analyze test results in accordance with this practice, even if values are missing or have been rejected as outliers, is available from ETM Information Services Inc., 1829 Chaine Court, Gloucester, Ontario, N1C 2W6, Canada. Equivalent programs may be available from other sources.

15.1.3.4 Next, apply the test for outliers, 13.3, or the control chart technique (D3) of Practice E 180, to see if a suspect value can be rejected. When more than two replications are made at a time and only one result is divergent, that result can be discarded and the mean calculated from the remaining values, thus retaining the balanced design of the interlaboratory study. When only duplicate determinations are made it is usually necessary to reject both results. In the example, Replicate b of Laboratory V on Enamel A at Time 3 has caused the range to be larger than acceptable, possibly the result of inverting figures when taking the readings or preparing the report. The mean for Time 3 is therefore calculated from the two remaining values which changes the subtotal for Laboratory V to 3.9, the total and mean of Enamel A to 18.7 and 1.247, and the Laboratory V total to 140.2.

15.1.3.5 The ranges between the mean results at different times and the laboratory means (or totals) are also examined for discordant results to see if any should be discarded before conducting the analysis of variance. In the example none are rejectable; although the mean (and total) for Enamel E obtained by Laboratory IV appears suspect, the calculated quotient does not exceed the R.Q. of 0.710 for a significance level of 0.05 and five values, as shown:

$$\frac{48.9 - 34.4}{48.9 - 26.7} = 0.653$$

15.1.3.6 An analysis of variance is performed on the results obtained for each material by the test procedure, that is, a total of six analyses. It is necessary to compute first the variances for each material because the variances may differ significantly for different types of materials or for different

laboratory variance for all materials. Obtain the intralaboratory standard deviation, s_w , by taking the square root of the variance and report.

15.1.1.6 Compute the maximum allowable difference (MAD) by multiplying s_w by the q factor (Table 6) for the appropriate number of replicates in each laboratory and the degrees of freedom (n_m materials) $\times$ (n_L laboratories) $\times$ (time - 1). In case test results are missing, compute the n_L ($t - 1$) degrees of freedom for each material and total them.

15.1.1.7 Examine the results from each laboratory for each material to establish whether all results are within the MAD for intralaboratory determinations. If this range is exceeded and outliers were not previously rejected in accordance with 13.3, discard the discordant results and recalculate the variance, standard deviation, and MAD. Compare with the original values to establish whether the rejection is justified; for example, a marked reduction in MAD with the elimination of only one set of results is evidence that they differ significantly from the other results and their retention would adversely affect the precision of the test method.

15.1.1.8 If the intralaboratory variances for the materials do not appear homogeneous but appear to be dependent on the level of the mean value of the materials, homogeneity may be achieved generally by converting the standard deviations to coefficients of variation:

$$v_w = 100 s_w / \bar{X}$$

15.1.1.9 If the coefficients of variation are homogeneous, pool them in accordance with the equation, as follows:

$$v_p = \left[\frac{(n_1 - 1)v_1^2 + (n_2 - 1)v_2^2 + \dots + (n_m - 1)v_m^2}{(n_1 - 1) + (n_2 - 1) + \dots + (n_m - 1)} \right]^{1/2}$$

where:

v_1 = coefficient of variation for Material 1, and

n_1 = number of values used to establish v_1 , etc.

When $n_1 = n_2 = n_m$, this condenses to $(\sum v_i^2 / n_m)^{1/2}$

where:

$\sum v_i^2$ = sum of squared coefficients, and

n_m = total number of materials.

Calculate the maximum allowable difference in percent relative by multiplying the coefficient of variation by q for the number of repeats and degrees of freedom.

15.1.1.10 If the coefficients of variation do not appear to be homogeneous, a transformation of the test results (such as to logarithms, arc sine, or square root) may provide homogeneous variances.

15.1.1.11 If homogeneous variances or coefficients of variation cannot be obtained from the intralaboratory results, calculate the precision for appropriate levels of the material value.

15.1.2 *Reproducibility (Interlaboratory)*—Test reproducibility is determined from the estimated interlaboratory variance which is the variance of the mean values obtained by the laboratories (s_L^2) plus the intralaboratory variance. Thus, interlaboratory variance is as follows:

$$s_b^2 = (s_w^2 / n_R) + s_L^2$$

where n_R = number of repeats in each laboratory.

NOTE 4—The subscript, b, derives from the previously used term "between-laboratories".

15.1.2.1 The equation defining s_w^2 and the procedures for its computation are discussed in 15.1.1.1 and 15.1.1.2.

15.1.2.2 The equation defining the variance of laboratory means, s_L^2 , is as follows:

$$s_L^2 = \sum (\bar{X} - \bar{\bar{X}})^2 / (n_L - 1)$$

where:

$\bar{X}$ = mean obtained for a material by a laboratory,

$\bar{\bar{X}}$ = grand mean of the values obtained for a material by all laboratories,

n_L = number of laboratories,

$(\bar{X} - \bar{\bar{X}})^2$ = sum of squares, ss, and

$n_L - 1$ = degrees of freedom, df.

To facilitate calculations, the equation is converted to

$$s_L^2 = \frac{\sum \bar{X}^2 - ((\sum \bar{X})^2 / n_L)}{n_L - 1}$$

15.1.2.3 When the results obtained in the interlaboratory test are from a balanced design and there are no missing values, compute the sum of squares and variance for each material by the analysis of variance technique, described in 15.1.3. Calculate the interlaboratory variance using the equation given in 15.1.2.

15.1.2.4 Pool the interlaboratory variances for all materials (unless they appear nonhomogeneous) to give a single interlaboratory variance for the test. Calculate the interlaboratory standard deviation, s_b , and report.

15.1.2.5 Compute the maximum allowable difference by multiplying s_b by the q factor (Table 6) for the appropriate number of laboratories and the degrees of freedom of ($n_L - 1$) times the number of materials.

15.1.2.6 Examine the results from all laboratories to establish that the range of laboratory means does not exceed the MAD. If the range is exceeded and laboratory outliers were not previously rejected, discard the discordant results and recalculate the MAD as in 15.1.1.7.

15.1.2.7 If the interlaboratory variances for the materials do not appear to be homogeneous, but appear to vary with the level of the means of the materials, convert the standard deviations to coefficients of variation, and if homogeneous, pool them as shown in 15.1.1.8 and 15.1.1.9. Calculate the MAD in percent by multiplying the coefficient of variation by q for the number of laboratories and degrees of freedom.

15.1.2.8 If the coefficients of variation do not appear homogeneous, a transformation of the test results (such as to logarithms, arc sine, or square root) may provide homogeneous variances. If neither variances nor coefficients of variation are homogeneous for the interlaboratory results, calculate the reproducibility for each level of test value of the materials as in 15.1.1.11.

15.1.3 *Analysis of Variance of Balanced Results:*

15.1.3.1 Where a design recommended in Part B has been used and balanced results are available (that is, no missing values from a balanced design or missing values replaced by the appropriate mean value), the analysis of variance technique should be used to compute the intralaboratory (within)

TABLE 7 Knoop Hardness of Baked Enamels
(a) Summary of Test Results

Laboratory		I				II				III				IV				V				Material	
Enamel	Replicate	Time 1	Time 2	Time 3	Sub-total	Time 1	Time 2	Time 3	Sub-total	Time 1	Time 2	Time 3	Sub-total	Time 1	Time 2	Time 3	Sub-total	Time 1	Time 2	Time 3	Sub-total	Total	Mean
A	a	1.0	1.4	1.2		0.9	1.1	1.0		1.4	1.2	1.1		1.3	1.3	1.6		1.3	1.2	1.3		19.0	1.267
	b	1.2	1.2	1.1		1.1	1.1	1.3		1.2	1.4	1.2		1.6	1.5	1.4		1.3	1.5	2.1			
	c	1.1	1.0	1.3		1.0	1.1	1.0		1.0	1.3	1.3		1.3	1.7	1.6		1.6	1.2	1.1			
	Mean	1.1	1.2	1.2	3.5	1.0	1.1	1.1	3.2	1.2	1.3	1.2	3.7	1.4	1.5	1.5	4.4	1.4	1.3	1.5	4.2		
B	a	2.5	2.6	2.6		2.8	3.2	3.2		2.9	2.6	2.6		3.4	3.0	3.1		3.1	3.2	3.4		43.7	2.913
	b	2.7	2.3	2.4		3.0	2.9	3.0		2.6	2.5	2.6		3.3	3.2	2.8		2.9	3.6	3.1			
	c	2.6	2.3	2.8		2.9	2.9	3.1		2.9	2.4	2.6		3.2	3.4	2.8		3.3	3.4	3.4			
	Mean	2.6	2.4	2.6	7.6	2.9	3.0	3.1	9.0	2.8	2.5	2.6	7.9	3.3	3.2	2.9	9.4	3.1	3.4	3.3	9.8		
C	a	4.7	4.8	4.9		4.7	5.7	5.2		4.5	4.6	4.0		5.7	6.2	5.6		4.7	4.6	5.7		75.8	5.058
	b	4.3	5.0	4.6		4.9	5.3	5.7		4.0	4.4	4.3		6.0	6.0	5.7		4.9	5.1	5.1			
	c	4.5	4.9	4.6		5.1	5.5	5.3		4.1	4.5	4.3		5.7	6.4	6.1		5.1	5.0	5.4			
	Mean	4.5	4.9	4.7	14.1	4.9	5.5	5.4	15.8	4.2	4.5	4.2	12.9	5.8	6.2	5.8	17.8	4.9	4.9	5.4	15.2		
D	a	6.2	6.5	6.5		8.0	6.9	7.9		6.7	6.3	6.2		8.5	8.7	8.7		8.0	7.7	7.7		110.2	7.347
	b	5.6	6.8	6.1		7.9	7.3	7.2		7.3	6.5	6.7		8.3	8.2	9.3		8.4	7.1	7.8			
	c	5.9	6.5	6.0		7.5	7.1	7.4		7.0	6.7	6.3		8.1	8.3	9.0		8.2	7.4	8.2			
	Mean	5.9	6.6	6.2	18.7	7.8	7.1	7.5	22.4	7.0	6.5	6.4	19.9	8.3	8.4	9.0	25.7	8.2	7.4	7.9	23.5		
E	a	8.6	8.6	9.5		11.0	9.4	10.3		10.1	9.3	8.9		16.6	16.5	15.9		11.9	11.7	11.3		168.9	11.260
	b	8.2	9.2	9.2		10.4	9.2	9.9		9.7	9.5	9.2		16.0	17.0	15.4		11.8	11.5	11.0			
	c	8.1	8.9	9.8		10.7	9.6	10.4		10.5	10.0	8.8		16.3	16.9	16.1		12.3	11.0	10.7			
	Mean	8.3	8.9	9.5	26.7	10.7	9.4	10.2	30.3	10.1	9.6	8.9	28.6	16.3	16.8	15.8	48.9	12.0	11.4	11.0	34.4		
F	a	14.6	16.1	14.8		18.6	18.7	17.7		15.4	13.8	13.9		17.4	16.1	17.3		18.6	20.2	19.1		251.4	16.760
	b	14.3	16.2	15.5		18.0	19.3	17.2		15.2	13.2	14.6		18.2	16.2	16.7		18.0	19.6	18.7			
	c	14.9	15.7	15.3		17.7	19.0	17.0		14.7	13.5	14.1		17.8	16.6	17.0		18.3	19.9	19.5			
	Mean	14.6	16.0	15.2	45.8	18.1	19.0	17.3	54.4	15.1	13.5	14.2	42.8	17.8	16.3	17.0	51.1	18.3	19.9	19.1	57.3		
Laboratory Total		116.4				135.1				115.8				157.3				144.4					
Grand Total																						669.0	

(b) Range of Triplicates at Same Time
Number at Stated Level

Enamel	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
A	1	0	6	4	2	1	...	...	...	...	1
B	1	0	5	5	4	...	...	...	...	...	...
C	...	...	2	3	5	4	1	...	...	...	...
D	...	...	...	1	4	5	4	1	...	...	...
E	...	...	...	...	1	4	6	3	1	...	...
F	...	...	...	...	...	2	6	4	2	1	...

For 15 values $Q = \frac{X_n - X_{n-2}}{X_n - X_9}$. The only divergent range is with Enamel A where $\frac{1 - 0.4}{1 - 0.2} = \frac{0.6}{0.8} = 0.75$. R.Q. at 0.01 significance = 0.647.

levels of the property measured. The analysis of variance of test results is facilitated by the construction of an analysis of variance table as shown in Table 8.

15.1.3.7 After calculating the means for the replicates, rejecting outliers where necessary, prepare a table of the means, which are considered to be the individual results, for each material as shown in Table 9 for Enamel A.

15.1.3.8 Perform the computations outlined in Table 8 to obtain the total sum of squares and the laboratories and the intralaboratory sums of squares. For each material from the sums of squares and the degrees of freedom, compute mean squares and variances for intralaboratory and for laboratory means as outlined in Table 8 and shown by the example in Table 9.

NOTE 5—In this example, triplicate measurements were made so that n_R equals 3 but in cases where duplicates are sufficient (see 11.2.1) the laboratory total sums of squares and the laboratory mean variances are obtained using two as the divisor. The number of replicates does not affect n_R .

15.1.3.9 Calculate the intralaboratory and interlaboratory standard deviations for each material as in Table 10. Determine the maximum allowable differences for each and compare with the ranges in the results as shown in Table 9. If required, discard discordant results and recalculate.

15.1.3.10 If the standard deviations for the different materials are homogeneous, pool the variances and calculate the overall repeatability and reproducibility, as described in 15.1.1.6, 15.1.1.7, 15.1.2.5, and 15.1.2.6.

15.1.3.11 If the results are not homogeneous, convert the standard deviations to coefficients of variation, pool them and calculate the repeatability, as described in 15.1.1.8, 15.1.1.9, and 15.1.2.7 and shown in Annex A1.

15.1.3.12 Prepare a table showing the standard deviations, coefficients of variation (if required) and the precision as given in Table 11.

16. Sensitivity

16.1 If a reasonably linear relationship ($r > 0.7$) has been

TABLE 8 Analysis of Variance Table

Source of Variance	Sum of Squares (ss)	Degrees of Freedom (DF)	Mean Square	Expected Mean Square ^a
	Eq 1 = $\Sigma X^2 - (\Sigma X)^2/n$	$n - 1$	...	...
Laboratories	Eq 2 = $\Sigma L^2/n_L - (\Sigma X)^2/n$	$n_L - 1$	ss/DF	$s_w^2 + n_L s_L^2$
Intralaboratory	Eq 1 - Eq 2	$(n - 1) - (n_L - 1) = n - n_L$	ss/DF	s_w^2

- ^a = total number of determinations,
 = number of laboratories,
 = number of determinations (repeats) by each laboratory,
 = sum of individual results (usually mean of duplicates),
 = sum of squared individual results,
 = sum of individual results, squared,
 = sum of laboratory totals,
 = intralaboratory variance,
 = variance of laboratory means, and
 = $(s_w^2/n_L) + s_L^2$ = interlaboratory variance.

Occasionally, in an exchange where the intralaboratory variability is very high without a corresponding large difference between laboratory means, subtracting s_w^2 from $s_L^2 + n_L s_L^2$ yields a negative result. Since a variance cannot be negative, the usual practice in such cases is to consider s_L^2 to be zero.⁷

TABLE 9 Example of ANOVA for Enamel A—Means of Triplicates^a

Laboratory	I	II	III	IV	V	Time Total
Time 1	1.1	1.0	1.3	1.4	1.4	6.2
Time 2	1.2	1.1	1.2	1.5	1.3	6.3
Time 3	1.2	1.1	1.2	1.5	1.2	6.2
Laboratory Total	3.5	3.2	3.7	4.4	3.9	18.7
$n = 15$	$\Sigma X = 18.7$		$\bar{X} = 1.2467$		$\Sigma L^2 = 70.75$	
$n_L = 3$	$\Sigma X^2 = 23.63$		$(\Sigma X)^2/n = 23.3127$		$\Sigma L^2/n_L = 23.5833$	
Source of Variation	Total ss	Net ss	df	Mean Square	Variance	
Grand total	23.63	0.31733	14			
Laboratories	23.58333	0.27067	4	0.06767		
Intralaboratory		0.04666	10	0.00467	0.00467 = s_w^2	
Laboratory means				0.06300 + 3 =	0.021 = s_L^2	
Correction factor $(\Sigma X)^2/n$	23.31267					

Conclusion:

Intralaboratory:

$$s_w^2 = 0.00467 \quad s_w = 0.0683$$

From Table 8, q for 10 df is 3.88 for three determinations. Therefore, the maximum acceptable difference (MAD) = $0.0683 \times 3.88 = 0.265 \approx 0.27$ for three determinations (each the mean of triplicates). All laboratories comply since the maximum range is 0.2 for Laboratory V.

Interlaboratory:

$$s_L^2 = s_w^2/n_L + s_L^2 = \frac{0.00467}{3} + 0.021 = 0.00155 + 0.021 = 0.02255$$

$$s_L = (0.02255)^{1/2} = 0.150$$

From Table 8, q for 4 df = 6.31 for five laboratories. Therefore MAD = $0.15 \times 6.31 = 0.95$ for five laboratories. All laboratories comply since the maximum range between laboratory means = $1.467 - 1.067 = 0.4$ for Laboratories II and IV.

Except for Laboratory V, Time 3.

and between instrumental values and known property or component values for the materials tested by a test method, the certainty with which the test method can detect differences in the parameter should be ascertained.

16.2 A useful measure of this certainty is the sensitivity criterion.⁸ This criterion, SC , is defined as

$$\frac{\Delta A/\Delta P}{s_A/s_P}$$

where:

$\Delta A/\Delta P$ = change in instrumental value A for a unit difference in known parameter P ,

s_A = estimated experimental error for a single instrumental measurement, and

s_P = estimated experimental error for a known parameter value.

In many instances s_P is not available so that s_P and ΔP are assumed to be 1.0, which removes them from the equation. Thus

$$SC = \Delta A/s_A$$

16.3 The value used for s_A depends on the manner in which the test is conducted. For example, if materials are tested by the same operator with a single instrument, s_w is used. Where the materials are tested in different laboratories, s_b is used.

16.4 In the case where it is desired to know which of two alternative test methods, A or B, has the greatest sensitivity for measuring P , a sensitivity ratio, SR , is calculated as follows.⁸

⁷ Neter, J. and Wasserman, W., *Applied Linear Statistical Models*, Irwin, Homewood, IL, 1974, p. 534.

⁸ Mandel, J., and Stiehler, R. D., "Sensitivity—A Criterion for the Comparison of Methods of Test," *Journal of Research*, National Bureau of Standards, RP2527, Vol. 53, No. 3, 1954, p. 155.

TABLE 10 Summary for All Enamels^A

Enamel	ΣX	ΣX^2	$(\Sigma X)^2/n$	Net Total ss	$\Sigma L^2/n_R$	Net Laboratory ss		
A	18.7	23.63	23.3127	0.3173	23.5833	0.2707		
B	43.7	128.75	127.3127	1.4373	128.5233	1.2107		
C	75.8	388.20	383.0427	5.1573	387.58	4.5373		
D	110.2	821.38	809.6027	11.7773	820.0667	10.464		
E	188.9	2011.15	1901.814	109.335	2007.8367	106.0227		
F	251.4	4267.24	4213.464	53.776	4261.1133	47.6493		
Total—668.7								
Intralaboratory								
Enamel	ss	df	Mean Square = s_w^2	s_w	Mean	$v_w\%$		
A	0.0467	10	0.00467	0.068	1.247	5.48		
B	0.2267	10	0.0227	0.1605	2.913	5.17		
C	0.620	10	0.062	0.249	5.053	4.93		
D	1.3133	10	0.1313	0.3625	7.347	4.93		
E	3.3133	10	0.3315	0.5755	11.26	5.11		
F	6.1267	10	0.6127	0.783	16.76	4.67		
Total—60								
Interlaboratory								
Enamel	df	Mean Square	$n_R s_L^2$	s_L^2	s_w^2/n_R	s_b^2	s_b	$v_b\%$
A	4	0.0677	0.063	0.021	0.0016	0.0226	0.15	12.05
B	4	0.3027	0.280	0.0933	0.0076	0.1009	0.32	10.30
C	4	1.1343	1.072	0.3574	0.0207	0.3781	0.615	12.17
D	4	2.616	2.485	0.8282	0.0438	0.872	0.93	12.71
E	4	26.5057	26.174	8.7248	0.1104	8.6352	2.97	26.4
F	4	11.9123	11.30	3.7667	0.2042	3.971	1.99	11.89
Total—24								

^A See Annex A1 for discussion of statistical analysis.

TABLE 11 Summary of Interlaboratory Test by Method _____ Indicating Intra- and Interlaboratory Agreement

Material	Mean	Standard Deviation				Coefficient of Variation	
		Intralaboratory		Interlaboratory		Intralaboratory	Interlaboratory
		s_w	df	s_b	df	v_w	v_b
1							
2							
etc. to							
M							

$$SR = \frac{SC_{(A)}}{SC_{(B)}} = \frac{\frac{\Delta A/\Delta P}{s_A/s_P}}{\frac{\Delta B/\Delta P}{s_B/s_P}} = \frac{\Delta A/s_A}{\Delta B/s_B}$$

where, if SR is greater than 1.0, Test Method A is superior to B, and if SR is less than 1.0, Test Method B is superior.

16.5 Obviously, a sensitivity ratio should not be used to compare test methods unless it has been established that one of these test methods provides instrumental values that correlate well with known property or component values of materials. For example, if Test Method A has been found to give instrumental values (observed or transformed) that correlate reasonably well ($r > 0.7$) with known property values, then a plot can be made of Test Method B values versus Test Method A values for the materials tested.

16.5.1 If the plot of points shows a curvilinear relationship, then the values for Test Method B should be transformed to provide a linear relationship. When this has been done, the slope of the straight line relating the points is $\Delta A/\Delta B$ and

$$SR = \text{slope}/(s_A/s_B)$$

16.5.2 In some instances, the SR for two test methods will not be constant over the range of materials tested because of

changes in the test errors with a change in parameter level. In such cases, an SR should be calculated for each of the parameter value levels. A plot of SR values versus parameter values is useful in comparing the sensitivities of the two test methods.

PART D—ANALYSIS PRESENTATION

17. Scope

17.1 This part describes the essential requirements for the preparation of a comprehensive report on an ASTM cooperative test program, for the guidance of working groups and committees or for publication, and of the precision statement to be used in test methods.

18. General Report Requirements

18.1 The essential requirements for a report on a cooperative test program are as follows:

18.1.1 The presentation in one document of details pertaining to the program, including a complete description of the experiment with a clear statement of purpose, procedure, instructions, and list of participants.

18.1.2 The presentation of all the original results reported by the participants, together with their comments.

TABLE 12 Summary of Results from Interlaboratory Test on _____ by Method _____

Laboratory	Material 1			Material 2			etc. to Material M		
	Number of Determinations	Mean	Range	Number of Determinations	Mean	Range	Number of Determinations	Mean	Range
I									
II									
etc. to									
L									
Column totals									
Column means	...			...			...		

18.1.3 An integrated compilation of the results into tables and in the form of graphs or charts that may be studied to obtain a clear view of the experiment as a whole.

18.1.4 The presentation, in the most concise form, of the conclusions drawn from an analysis of the results.

18.1.5 The presentation of a summary, conclusions, or a list of recommendations.

18.2 The report should provide maximum ease of reference to the details of the experiment, of observing results in tabulation, and the essential conclusions. The report should be available to all members of the task group well in advance of meetings so that there is sufficient time for study and development of new ideas for discussion at meetings.

19. Procedures for Preparing Summaries

19.1 For each material or each test method, prepare a table that compiles in an orderly block arrangement the results from all of the laboratories (including operators and time). A format based on the one given in Fig. 1, but modified to include means and ranges, can be used. Using the design shown in Table 12, prepare a table for each test method giving the laboratory mean values and ranges for the different materials. Prepare a table for each test method listing for each material the means, the intra- and interlaboratory standard deviations, the degrees of freedom, and, if applicable, the coefficients of variation. The format can be based on parts of Table 10 or on Table 11. It should be evident from this tabulation whether the standard deviation varies with the magnitude of the property being measured and, consequently, whether the coefficient of variation is required.

19.2 An appropriate block of results may be compiled into a frequency distribution table that can be prepared in several different ways as follows:

19.2.1 Tabulate the values in increasing order of magnitude and express the frequency with which each value occurs as a fraction or percent calculated by dividing the number of times the value occurs by the total number of values in a group. When a large number of values is being treated, they may be divided into classes of equal range (for example, ten classes) and the frequency of each class calculated.

19.2.2 Calculate the deviation of each value from the mean value of its group noting the sign and tabulate these in order of magnitude. Calculate the frequency of each deviation as a fraction or percent of all the values treated. When a large number of values is being treated, the deviations may be divided into classes of equal range.

19.2.3 Frequency distributions may be expressed in a cumulative way by arranging the values in order of magni-

tude and summing at each stage of this arrangement the total occurrence of all values up to and including the selected stage.

19.2.4 Graphical presentations are very useful and concise allowing rapid inspection and judgment of a collection of related results. They may be prepared from all the results from an experiment or an appropriate segment to obtain a view of the nature of the distribution. If it is revealed that an adequate normal distribution exists, relatively simple mathematical treatments may be applied. Frequency distributions may be presented in graphical form, as histograms whereby values or classes covering a range of values are plotted against their frequency, or as cumulative frequency graphs prepared by plotting at each point the cumulative frequency of all the values up to that point against the value at that point. One of the best ways of displaying results is the dot diagram.⁹ Figure 3 shows the relation of dry-to-touch time to the day on which a test was made and to the type of drier used, and also shows the overall distribution of the results. It can readily be seen that there is definitely a difference between days but none between drier types, as compared to the overall distribution.

19.3 The final summary should be a description of results from the cooperative program, expressed in terms of repeatability and reproducibility stated with supporting information, that is, degrees of freedom.

19.4 Prepare a draft report, with the test results and their analysis, for deposit in ASTM Headquarters files.

20. Applicability and Precision Statements for Test Methods

20.1 The major functions of the interlaboratory test of a measurement procedure are the determination of:

20.1.1 The applicability of the procedure for measuring a property of a material or groups of materials, and

20.1.2 The precision with which repeated measurements according to the test method can be expected to be made.

20.2 Applicability implies both the correlation of a measurable property with an unmeasurable property of a material and the sensitivity with which differing levels of that property are discriminated by the test method. Correlation may be evaluated by means of a rank order correlation coefficient. Sensitivity may be evaluated by means of the sensitivity criterion (ratio of the range in property levels of a material to the standard deviation). As applicability, or

⁹ Box, G., Hunter, W., and Hunter, S., *Statistics for Experimenters*, John Wiley and Sons, New York, NY, 1978, p. 221.

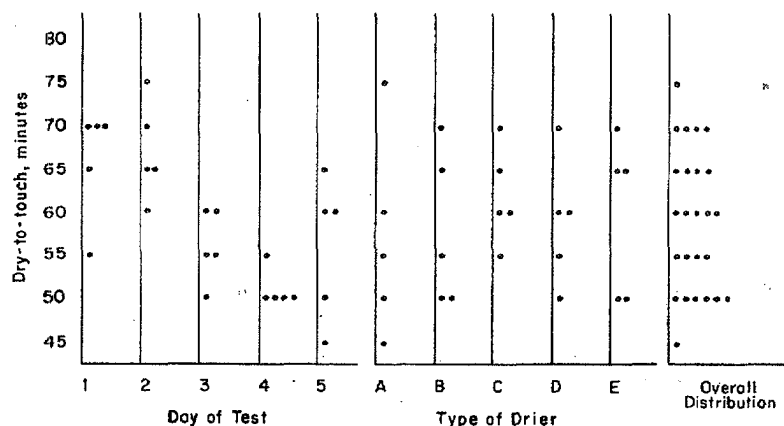


FIG. 3 Illustration of Dot Diagram

validity, of a test method depends on the magnitude of the values of correlation and sensitivity, as well as on the number of materials used in the interlaboratory test, inclusion of a statement of applicability is recommended as follows:

Applicability of Test Method—Based on measurements of _____ materials having known qualitative ratings that range from _____ to _____ (good to poor, for example) the rank correlation coefficient for the test method was found to be _____ and the sensitivity criterion was found to be _____.

20.3 Precision is useful only if the applicability function of the test method is found to be favorable. Precision is defined as the degree of agreement among repeated independent measurements of the same property. Statements of precision may be given in terms of the standard deviation, but this form is not very useful. In addition, when values obtained vary directly with the property levels of the materials tested, the coefficient of variation must be used to express the general precision. Statements of precision can also be expressed in terms of the range in values within which the correct value can be expected to lie a specified percent of the time. The most common precision index is the range that should not be exceeded for a stated number of results at a given confidence level, assuming that the frequency distribution is normal and that a random sample was used in the interlaboratory test. Because only a sample of the total population of measurements is obtained in a test, statements of precision should indicate the number of degrees of freedom and the number of observations that were used in the interlaboratory test to obtain the indicated values.

20.3.1 As can be seen from Tables 11 and 12, the measurements made in an interlaboratory test permit a number of parameter variations. Precision indexes can be devised with respect to differences within and among laboratories, materials, operators, and repeat measurements and

with respect to the interactions among combinations of these parameters. It is evident from this discussion that to provide all of these precision indexes would make this section unnecessarily long and impair its usefulness. The decision on how many of the possible indexes are to be included in a test method is left to the discretion of those who formulate and use the test method. In this practice, however, the precision statement is based on the two indexes—repeatability and reproducibility as defined in 3.26:

Precision of Test Method—In an interlaboratory study of this test method in which _____ operators in _____ laboratories tested _____ coatings with a broad range of _____ (property) levels (analyzed _____ materials containing _____), the intralaboratory standard deviation (coefficient of variation) was found to be _____ units (percent) with _____ degrees of freedom (df) and the interlaboratory standard deviation (coefficient of variation) _____ units (percent) with _____ df after rejecting _____ results(s) from _____ laboratory (ies) for one time because the range between replicates (repeats) differed significantly from all other ranges for material _____, or all results from one laboratory for material _____ because the mean differed significantly from all other means. Based on these standard deviations (coefficients) the following criteria should be used for judging, at the 95% confidence level, the acceptability of results:

Repeatability—_____ results, each the mean of _____ replicates (if applicable), obtained by the same operator should be considered suspect if they differ by more than _____ units (% relative).

Reproducibility—Two results, each the mean of _____ replicates obtained by operators in different laboratories should be considered suspect if they differ by more than _____ units (% relative).

NOTE 6—Where the results obtained are in percent instead of some unit add "absolute" after X% in the repeatability and reproducibility statements to distinguish from cases where use of the coefficients of variation results in percent relative precision limits.

NOTE 7—Users of ASTM test methods should be aware that the precision obtained from an interlaboratory study is for measurements made on the same batch of each material. Consequently, normal batch-to-batch variability, which is governed by manufacturing quality control, is not included in the precision values established for a test method.

ANNEX

(Mandatory Information)

A1. Discussion of Table 10

A1.1 Two points are evident: (1) neither the intralaboratory nor interlaboratory variances can be pooled because the standard deviations increase with higher hardness values; (2) the intralaboratory coefficients of variation are homogeneous, but there is one discordant value (Enamel E) in the interlaboratory coefficients.

A1.2 The tests referred to in 15.1.1.4 can be applied to confirm that the interlaboratory coefficients cannot be pooled or, as shown below, the coefficients are pooled to determine if any maximum allowable difference is then needed. Pooling the coefficients of variation in accordance with the formula in 15.1.1.8 yields the following:

$$\text{Pooled } v_w = \left(\frac{153.2873}{6} \right)^{1/2} = (25.5479)^{1/2} = 5.055 \%$$

For three determinations $v_w \cdot q = 5.055 \times 3.40 = 17.19 \%$.

$$\text{Pooled } v_b = \left(\frac{1411.8364}{6} \right)^{1/2} = (235.3061)^{1/2} = 15.34 \%$$

For five laboratories $v_b \cdot q = 15.34 \times 4.17 = 63.97 \%$.

Therefore the acceptable range for Enamel E is $0.172 \times 1.26 = 1.94$ for intralaboratory results and $0.64 \times 11.26 = 7.21$ for interlaboratory results. The actual maxima obtained for Enamel E in the round robin are for the intralaboratory range 1.3 for Laboratory II, and for the interlaboratory range between Laboratories I and IV. Thus, while the results from Laboratory IV for Enamel E could not be rejected in 15.3.5 on the basis of Enamel E alone, they can be on the basis of all six enamels.

A1.3 Instead of rejecting the results from all laboratories on one material, it is preferable to discard only those of the divergent laboratory. When this procedure is followed for Enamel E the revised values are: $\Sigma X = 120.0$, $n = 12$, $\bar{X} = 10.00$, $\Sigma X^2 = 1213.58$, $(\Sigma X)^2/n = 1200$, net total ss = 13.58, $s^2/n_R = 1210.7667$, net laboratory ss = 10.7667, intralaboratory ss = 2.8133, $df = 8$, $s_w^2 = 0.3517$, $s_w = 0.593$, $s_w^2/n_R = 5.93 \%$, interlaboratory $df = 3$, mean square = 3.5889, $s_b^2 = 3.2372$, $s_b = 1.0791$, $s_b^2/n_R = 0.1172$, $s_b = 1.1963$, $s_b = 1.09$, $v_b = 10.94 \%$.

A1.4 The interlaboratory coefficients of variation are now all homogeneous so they can be pooled with total degrees of freedom of 58 for intralaboratory and 23 for interlaboratory coefficients.

$$\begin{aligned} \text{Pooled } v_w &= \left[\frac{5(127.1548) + 4(35.1667)}{(5 \times 5) + (1 \times 4)} \right]^{1/2} = \left(\frac{776.44035}{29} \right)^{1/2} \\ &= (26.7738)^{1/2} = 5.174 \%; v_w \cdot q = 17.65 \% \\ &\text{for three determinations.} \end{aligned}$$

$$\begin{aligned} \text{Pooled } v_b &= \left[\frac{5(714.9841) + 4(119.6289)}{25 + 4} \right]^{1/2} = \\ &\left(\frac{4053.4359}{29} \right)^{1/2} = (139.7737)^{1/2} = 11.82 \% \end{aligned}$$

Rejecting one laboratory's results for one material reduced the pooled interlaboratory coefficient of variation from 15.34 % to 11.82 %, which is certainly justification for discarding those results.

A1.5 From the pooled coefficient of variation the maximum acceptable difference, $v_b \cdot q$ is 34.4 % for two laboratories, 46.4 % for four laboratories, and 49.5 % for five laboratories (Note A1.1). Based on these values, the acceptable range in Knoop hardness results for each enamel is given in Table A1.1 and compared with the maxima found in the study.

NOTE A1.1—The MAD used for determining the acceptability of results in the round robin must be based on the number of participating laboratories while that used in the final precision statement is for two laboratories.

TABLE A1.1 Knoop-Hardness Ranges For Enamels

Enamel	Intralaboratory		Interlaboratory	
	Calculated	Found	Calculated	Found
A	0.22 ^a	0.2	0.62 ^a	0.4
B	0.51	0.4	1.44	0.73
C	0.89	0.6	2.5	1.63
D	1.3	0.8	3.64	2.33
E	1.8	1.3	4.64 ^a	2.57 ^a
F	3.0	1.7	8.3	4.83

^a These values are smaller than in Table 9 because the precision estimate is better from all six enamels than from just one.

^b For four laboratories.

APPENDIX

(Nonmandatory Information)

- (1) *ASTM Manual for Conducting an Interlaboratory Study of a Test Method*, ASTM STP 335, ASTM, 1963.
- (2) *ASTM Manual on Quality Control of Materials*, ASTM STP 15, ASTM, 1951.
- (3) ASTM Practice D 1749, for Interlaboratory Evaluation of Test Methods Used with Paper and Paper Products, *Annual Book of ASTM Standards*, Vol 15.09.
- (4) ASTM Practice D 2904, for Interlaboratory Testing of Textile Materials, *Annual Book of ASTM Standards*, Vol 07.01.
- (5) ASTM Practice E 173, for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals, *Annual Book of ASTM Standards*, Vol 03.05.
- (6) ASTM Practice E 691, for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods, *Annual Book of ASTM Standards*, Vol 14.02.
- (7) Bennet, C. A., and Franklin, N. L., *Statistical Analysis in Chemistry and the Chemical Industry*, John Wiley and Sons, New York, NY, 1954.
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- (16) Mann, H. B., *Analysis and Design of Experiments*, Dover, 1949.
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- (18) Quenouille, M. H., *The Design and Analysis of Experiments*, Hafner, 1953.
- (19) Youden, W. J., "Graphical Diagnosis of Interlaboratory Test Results," *Industrial Quality Control*, Vol XV, No. 11, 1959, p. 28.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Method¹

This standard is issued under the fixed designation D 4017; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method is applicable to all paints and paint materials, including resins, monomers, and solvents, with the exception of aldehydes and certain active metals, metal oxides, and metal hydroxides. While the evaluation was limited to pigmented products containing amounts of water in the 30 to 70 % range, there is reason to believe that higher and lower concentrations can be determined by this test method.

1.2 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7.*

Referenced Documents

- 2.1 *ASTM Standards:*
- D 1193 Specification for Reagent Water²
- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³
- E 203 Test Method for Water Using Karl Fischer Reagent³

Summary of Test Method

3.1 The material is dissolved in pyridine, or another appropriate solvent, and titrated directly with standardized Karl Fischer reagent, to an electrometric end point. The sluggish reaction with water in pyridine is accelerated with a chemical catalyst, 1-ethylpiperidine.

3.2 Pyridine is used as a solvent to minimize interference problems caused by ketones. It is also used because the more common solvent, methanol, will not dissolve many common resins and because methanol reacts with some resins to produce water.

Significance and Use

4.1 Control of water content is often important in controlling the performance of paint and paint ingredients, and it is critical in controlling volatile organic compound (VOC) content.

4.2 Paint materials are often insoluble in common Karl Fischer solvents such as methanol. Pyridine has been found

to be a nearly universal solvent for these materials; however, the Karl Fischer reaction is too slow in that solvent at room temperature. To speed it up, 1-ethylpiperidine is added at 5 % as a buffer, or "catalyst".

5. Apparatus

5.1 *Karl Fischer Apparatus*, manual or automatic, encompassed by the description in Test Method E 203. Apparatus should be equipped with a 25-mL buret, Class A, or equivalent.

5.2 *Syringe*, 100-μL capacity, with needle.

5.3 *Syringes*, 1-mL and 10-mL capacity, without needle, but equipped with caps.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water conforming to Type II of Specification D 1193.

6.3 *Karl Fischer Reagent*.⁵

6.4 *Pyridine*.

6.5 *1-Ethylpiperidine*.⁶

6.6 *Hydrochloric Acid (HCl)*, concentrated.

NOTE 1—All reagents must be fresh. Do not use reagents that are more than 9 months old. Karl Fischer reagent deteriorates with age. Check expiration dates on the reagent bottle.

7. Hazards

7.1 Karl Fischer reagent contains four toxic compounds, namely iodine, sulfur dioxide, pyridine, and methanol or glycol ether. The reagent should be prepared and dispensed in a hood. Care must be exercised to avoid inhalation or skin contact. Following accidental contact or spillage, wash with large quantities of water.

7.2 Pyridine and methanol solvents should be treated with

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved May 25, 1990. Published July 1990. Originally published as D 4017 - 81. Last previous edition D 4017 - 88.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

⁵ So-K-3 available from Fischer Scientific Co., or equivalent has been found suitable for this purpose.

⁶ Use 1-ethylpiperidine, manufactured by the Aldrich Chemical Co., Milwaukee, WI 53233, for the best results.

TABLE 1 Specimen Guidelines

Expected water, %	Approximate Specimen Weight, g	Approximate Titrant Volume at 5 mg/mL titre, mL
0.5-1.0	5	5-10
1-3	2-5	10-20
3-10	1-2	10-20
10-30	0.4-1.0	20-25
30-70	0.1-0.4	15-25
>70	0.1	20

the same care as Karl Fischer reagent.

7.3 1-ethylpiperidine is of unknown toxicity and, therefore, should be handled with the same care as the materials listed in 7.1 and 7.2.

8. Procedure

8.1 Standardization of Karl Fischer Reagent:

8.1.1 Add enough fresh pyridine to cover the electrode tip, plus 1 mL of 1-ethylpiperidine catalyst per 20 mL of pyridine. Catalyst performs best at a concentration of about 5 % of the volume present.

8.1.2 Fill the 100- μ L syringe to about half full with distilled water and weigh to the nearest 0.1 mg.

8.1.3 Pretitrate the pyridine to the end point indicated by the equipment manufacturer, by adding just enough Karl Fischer Reagent I (KFR) to cause the end point to hold for at least 30 s.

8.1.3.1 The use of the catalyst greatly increases the reaction rate between water and Karl Fischer reagent. To obtain reliable results, increase the electrode sensitivity and reduce titration rate to a minimum. Most instruments have controls for these functions. Consult the instructional manual for information on these controls.

8.1.4 Empty the contents of the syringe into the titrator vessel. Immediately replace the stopper of the sample port and titrate with KFR to the end point as described in 8.1.3.

8.1.5 Repeat standardization until replicate values of F agree within 1 %. Determine the mean of at least two such determinations. Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculations.

8.1.6 Calculation:

8.1.6.1 Calculate the KFR titre F as follows:

$$F = J/P \quad (1)$$

where:

J = water added, g, and

P = KFR used, mL.

The value for F should be recorded to the four significant digits and should be the mean of at least two determinations. Typical values are in the range of 0.004000 to 0.006000 g/mL.

8.2 Analysis of Samples With More Than 0.5 % Water:

8.2.1 The titration vessel should already contain pretitrated pyridine and catalyst, as described in 8.1.1 and 8.1.3 in the standardization procedure. Best results are obtained with fresh solvent, that is, containing no previously titrated specimen in the vessel.

8.2.2 With a 1-mL or 10-mL syringe, draw the amount of material indicated in Table 1.

NOTE 2—Paint samples tend to settle in the syringe and give high percent water content. Obtain a freshly stirred or mixed specimen for each test run.

8.2.2.1 Remove the syringe from the specimen, pull the plunger out a little further, wipe the excess material off the syringe, and place a cap on the syringe tip. Weigh the filled syringe to the nearest 0.1 mg.

8.2.3 Remove the cap, and empty the syringe contents into the pretitrated pyridine vessel. Pull the plunger out and replace the cap.

8.2.4 Stir rapidly for 1 to 2 min before starting titration. Some instruments can be set to do this automatically. If the specimen is still not dissolved or dispersed, continue stirring until it is dissolved, or use a different solvent in place of pyridine in 8.2.1.

8.2.5 Titrate the specimen slowly with KFR to the end point described in 8.1.3.

8.2.6 Reweigh the emptied syringe, and calculate the specimen weight by difference.

8.2.7 Calculation:

8.2.7.1 Calculate the percent water L as follows:

$$L = (P \times F \times 100)/S \quad (2)$$

8.3 Analysis of Materials With Less Than 0.5 % Water:

8.3.1 For 0.1 to 0.5 %, follow procedure in 8.2 (1-g specimen), except substitute a 1-mL microburet for the 25-mL buret in the Karl Fischer apparatus.

8.3.2 For less than 0.1 %, use a 1-mL microburet and increase specimen size as much as needed, up to 10 g. It should be possible to measure moisture levels down to 1 ppm (0.0001 %) by this approach.

NOTE 3—Specimens with less than 0.1 % water may require special handling techniques to prevent pickup of atmospheric moisture. The precision of this test method was determined with specimens containing higher water levels.

9. Recommendations for Good Results

9.1 Make sure electrodes are clean.

9.2 Follow manufacturer's instructions to ensure that venting into the titration vessel is only through a dessiccant.

9.3 Samples should be thoroughly mixed before taking a specimen.

9.4 Paints and paint materials are often slow to dissolve or disperse. To ensure that all of the water is extracted into the pyridine or solvent, stir rapidly for 1 or 2 min before starting the titration.

9.5 Run the titration slowly with rapid stirring.

9.6 Throw out the first result in fresh pyridine.

9.7 Use only Aldrich's 1-ethylpiperidine.⁶ It has been found that other brands produce variable results.

10. Maintenance

10.1 *Cleanup*—Clean the titration vessel by rinsing with fresh pyridine. Do not use methanol or other solvents.

10.2 *Dryness*—Check frequently to be sure that all drying tubes are in good condition and tightly connected. Replace dessiccant when indicator color changes through half of the tube.

10.3 *Electrode Performance*—If electrode response is sluggish or otherwise off standard, take the following steps, in turn, to correct the problem. Test the electrode with a

give high
specimen for

pull the
off the
the filled

contents
out and

titration;
ly. If the
stirring
place of

the end
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(2)
Water
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ation after each step, to determine if the next step is required.

10.3.1 Wipe the electrode tip with a clean paper towel.

10.3.2 Wash the electrode by dipping in concentrated hydrochloric acid for at least 1 min. Rinse first with distilled water, then with methanol.

10.3.3 Follow manufacturer's instructions on resetting and point meter.

10.3.4 Replace power source. See manual for replacement procedure.

10.3.5 Replace the electrode.

Precision and Bias

11.1 The precision estimates are based on an inter-laboratory study in which one operator in each of seven different laboratories analyzed in duplicate on two different days seven samples of water-based paints of various types containing between 25 to 75 % water. The results were

analyzed statistically in accordance with Practice E 180. The within-laboratory coefficient of variation was found to be 1.7 % relative at 98 df, and the between-laboratory coefficient of variation was 5.3 % relative, at 42 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

11.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 4.7 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 15.0 % relative.

11.2 *Bias*—Bias has not been determined for this test method.

12. Keywords

12.1 Karl Fischer reagent method; moisture content; water content.

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Standard Test Method for Reflection Haze of High-Gloss Surfaces¹

This standard is issued under the fixed designation D 4039; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for using two specular gloss measurements to obtain a haze index for high-gloss nonmetallic specimens (1-4).² It is particularly useful for evaluating the haze in clear finishes on nonglossy substrates, and the haze in reflected images produced by the surfaces of opaque glossy pigmented finishes.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 523 Test Method for Specular Gloss³

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

D 3964 Practice for Selection of Coating Specimens for Appearance Measurements³

3. Terminology

3.1 Definitions:

3.1.1 *specular gloss*—the relative luminous reflectance factor of a specimen at the specular direction.

3.1.2 *reflection haze*—perceived cloudiness in an image reflected by a glossy surface.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *60° specular gloss*—specular gloss measured with the 60° geometry specified in Test Method D 523.

3.2.2 *20° specular gloss*—specular gloss measured with the 20° geometry specified in Test Method D 523.

3.2.3 *haze index, H*—a measure of reflection haze, where $H = (G_{60} - G_{20})$; G_{60} is the 60° specular gloss and G_{20} is the 20° specular gloss.

4. Summary of Test Method

4.1 Measurements of 60° and 20° specular gloss are made on a specimen. The haze index is computed as the difference between the two measurements.

4.2 This test method is applicable to nonmetallic specimens having a 60° specular gloss value greater than 60 in accordance with Test Method D 523.

5. Significance and Use

5.1 Haze is produced by irregularities in the reflecting surface that affect the distribution of flux reflected around the specular angle. The procedures specified in Test Method D 523 are not designed to measure reflected-flux scattering characteristics of high-gloss finishes, particularly where specimens having different surface refractive indexes are being compared. A better index of scattering can be obtained by making two specular reflectance measurements, that is, the use of two receptor apertures of different size.

5.1.1 In this test method, values for reflection haze are obtained from two measurements of specular gloss, one made with a large receptor aperture and the other made with a small receptor aperture. The geometric conditions have been chosen to permit the use of glossmeters that provide 60° specular gloss measurements (large receptor aperture) and 20° specular gloss measurements (small receptor aperture) as specified in Test Method D 523.

5.2 Measured gloss values of specimens depend on the angle of illumination, refractive index of the material, and the geometric distribution of the reflected light. The gloss scale of both the 60° and 20° geometries is defined as 100 for polished black glass with a refractive index of 1.567. However, the polished black glass standards used in this test method usually have a lower refractive index (approximately 1.53). Therefore, they will have a gloss value less than 100. These gloss values will be different for 60° and 20° measurements. If the refractive index is 1.53, the black gloss standard will have a 60° gloss value of approximately 94 and a 20° gloss value of approximately 89. Therefore, this haze-free standard will have a haze index value of $94 - 89 = 5$. A haze-free coating (refractive index of approximately 1.53) will have a corresponding haze index of 5 units, the difference between its 60° and 20° gloss values. Because 20° gloss changes much more rapidly with index than 60° gloss, the reflection haze value of a specimen also depends on the index of refraction of the material. To establish a correction for the effect of refractive index would require measurement of the index for each specimen, which is impractical. Comparisons of reflection haze evaluated by this test method are therefore limited to specimens of essentially the same refractive index.

6. Apparatus

6.1 Glossmeter(s) capable of measuring 60° and 20° specular gloss in accordance with the specifications given in Test Method D 523.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved June 26, 1987. Published August 1987. Originally published as D 4039 - 81. Last previous edition D 4039 - 81.

² Boldface numbers in parentheses refer to the list of references at the end of this test method.

³ *Annual Book of ASTM Standards*, Vol 06.01.

Preparation and Selection of Test Specimens

7.1 This test method does not cover preparation techniques. When a test requires the preparation of specimens from a liquid coating, specify the techniques of specimen preparation.

7.2 Select test specimens in accordance with Practice D 3964.

7.2.1 To determine the maximum gloss obtainable from a material, such as a paint or a varnish, use methods in Test Methods D 823 to produce a film of uniform thickness on a smooth, planar substrate.

7.2.2 Use surfaces of good planarity, because surface damage, waviness, or curvature may affect test results significantly. The directions of brush marks or similar texture effects should be parallel to the plane of the axis of the two beams.

Calibration and Standardization

8.1 Calibrate the glossmeters with primary and secondary working standards in accordance with the procedures given in Test Method D 523.

Procedure

9.1 Select an area near the center of the test specimen and determine the 60° specular gloss.

9.2 Determine the 20° specular gloss on the same area of the specimen.

Calculation

10.1 Compute the haze index, H , as follows:

$$H = G_{60} - G_{20}$$

where:

G_{60} = value of 60° specular gloss and

G_{20} = value of 20° specular gloss.

NOTE 1—Because of the difference in the assigned values for the black glass standard at 20° and 60°, the haze index for specimens without haze will have value of approximately 5.

11. Report

11.1 Report the following information:

11.1.1 For each specimen, the measured G_{60} and G_{20} values and the computed haze index, H .

11.1.2 Where preparation of the test specimen has been necessary, describe or otherwise identify the method of preparation.

11.1.3 Identify the glossmeter used by make and model.

11.1.4 Identify the gloss standards used.

12. Precision

12.1 On the basis of interlaboratory studies of this procedure in which six laboratories conducted single determinations on eight high-gloss coatings differing in visually perceived reflection haze, the within-laboratory standard deviation for haze index values was found to be 0.9, and the between-laboratories standard deviation was found to be 2.5.

12.2 Based on these standard deviations, the following criteria should be used for judging the acceptability of single determinations at the 95 % confidence level.

12.2.1 *Repeatability*—Two results obtained by a single operator should be considered suspect if they differ by more than 2 units of haze index.

12.2.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 7 units of haze index.

REFERENCES

- (1) Nimeroff, I., "Two-Parameter Gloss Methods," *Journal of Research*, National Bureau of Standards, Vol 58, No. 3, March 1957, pp. 127-135.
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- (3) Horning, S. C., and Morse, M. P., "The Measurement of the Gloss of Paint Panels," *Official Digest*, Federation of Paint and Varnish Production Clubs, March 1947, p. 153.
- (4) Hunter, R. S., "Gloss Evaluation of Materials," *ASTM Bulletin*, No. 186, December 1952, p. 48.
- (5) Hunter, R. S., *The Measurement of Appearance*, Wiley-Interscience, New York, NY, 1975.

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Standard Test Method for Viscosity of Printing Inks and Vehicles by the Falling-Rod Viscometer¹

This standard is issued under the fixed designation D 4040; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Tables A1.1 and A1.2 editorially corrected and the year date changed to Nov. 15, 1991.

1. Scope

1.1 This test method covers the procedure for determining the falling-rod viscosity of printing inks, vehicles, and similar liquids that are essentially nonvolatile and unreactive under ordinary room conditions.

1.2 For printing inks, which are typically non-Newtonian, this test method is applicable in the apparent viscosity range of about 10 to 300 P at a shear rate of 2500 s⁻¹. For Newtonian liquids, the applicable viscosity range is about 10 to 1000 P (1 P = 0.1 Pa·s).

1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 8.

2. Referenced Document

2.1 ASTM Standard:

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²

3. Terminology

3.1 Definitions:

3.1.1 *viscosity, V* —the ratio of shear stress to shear rate. The viscosity of a liquid is a measure of the internal friction of the liquid in motion. The cgs unit of viscosity is 1 g/cm·s (1 dyne·s/cm²) and is called a poise. The SI unit is 1 N·s/cm² and is equal to 10 P.

3.1.2 *shear stress, S* —shearing force per unit area; the unit is 1 g/cm·s² (1 dyne/cm²). In the falling-rod viscometer, shear stress is proportional to total weight W per unit of shearing area A times the gravitational constant g , in accordance with the equation: $S = Wg/A$.

3.1.3 *shear rate, D* —velocity gradient through the stressed liquid; the unit is 1/s or 1 s⁻¹. In the falling-rod viscometer, shear rate is inversely proportional to fall time F per unit

distance L over which a unit thickness x of the liquid is stressed: $D = L/xF$.

3.1.4 *Newtonian*—refers to a liquid whose viscosity is constant at all shear rates.

3.1.5 *non-Newtonian*—refers to a liquid whose viscosity varies with shear rate. Such liquids may be either shear-thinning (pseudoplastic) or shear-thickening (dilatant). Most printing inks are shear-thinning.

3.1.6 *apparent viscosity, V_D* —the viscosity of a non-Newtonian fluid at a particular shear rate D . A shear rate of 2500 s⁻¹ has been found useful for printing inks and is specified in this test method.

3.1.7 *yield stress, S_o* —the minimum shear stress required to initiate motion in a non-Newtonian liquid.

3.2 Description of Terms Specific to This Standard:

3.2.1 *power law*—a mathematical model that presumes that the viscosity of a liquid varies with shear rate in accordance with a power function as follows:

$$k = \frac{S}{D^N}$$

where:

k = a constant related to the viscosity of the liquid and
 N = a constant describing the rate at which shear stress varies with shear rate.

The value of N is precisely 1.0 for a Newtonian fluid, less than 1.0 for a shear-thinning liquid, and greater than 1.0 for a shear-thickening liquid.

3.2.2 *power law plot*—a logarithmic plot of shear stress versus shear rate based on the expanded form of the power law equation:

$$\ln S = \ln k + N \ln D$$

For liquids conforming to the power law, the logarithmic plot of S versus D is linear over the shear rate range of interest. The slope of the line is the power law constant N .

3.2.3 *shortness*—the property of a non-Newtonian fluid that prevents it from being drawn into a filament.

3.3 Symbols (for Power-Law Calculations):

B = intercept of a straight line.

F = measured fall time, s.

F_c = corrected fall time, s.

F_{2500} = fall time equivalent to a shear rate of 2500 s⁻¹, s.

K_{2500} = apparent viscosity constant at 2500 s⁻¹, cm⁻¹s⁻¹.

N = slope of the power law plot, a measure of non-Newtonianism, cm²/dyne·s.

SF = shortness factor, s⁻¹.

S'_o = pseudo yield value, dyne/cm².

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved Nov. 15, 1991. Published January 1992. Originally published as D 4040 - 81. Last previous edition D 4040 - 89.

² Annual Book of ASTM Standards, Vol 05.01.

- = measured specimen temperature, °C.
- = reference temperature, °C.
- = apparent viscosity at 2.5 s^{-1} , P.
- = apparent viscosity at 2500 s^{-1} , P.
- = total weight, g.
- = added weight, g.
- = weight of rod, g.
- = weight required to obtain a shear rate of 2500 s^{-1} , g.

Summary of Test Method

4.1 This test method is based on measurements of the time required for a weighted rod to fall through an aperture containing the test specimen.

4.2 Fall times are corrected to a reference temperature of 25°C (or other mutually agreed-upon temperature). The test method specifies precise measurement of actual specimen temperature in order to detect fluctuations due to cooling by metal, heat of friction during shearing, and body heat of the operator.

4.3 Each specific instrument must be calibrated in order to establish the fall time that is equivalent to a shear rate of 2500 s^{-1} .

4.4 Fall times as a function of weight are extrapolated to 2500 s^{-1} by means of the power law (logarithmic) relationship between shear stress and shear rate. Apparent viscosity at 2500 s^{-1} and the degree of non-Newtonianism are determined by calculation or graphically. The calculation of several low shear parameters is also covered.

Significance and Use

5.1 Apparent viscosity at the relatively high shear rate of 2500 s^{-1} does not completely define the rheological properties of printing inks but is useful in the practical control of ink viscosity during production and the specification acceptance between supplier and purchaser.

5.2 The slope of the power law plot is the preferred measure of non-Newtonianism. The yield value, which is obtained by extrapolation of high-shear measurements to a shear rate approaching zero, does not conform to the definition of the true yield stress (see 3.1.7). The yield value and other low shear parameters are also subject to a high degree of variability (see the precision table in Section 16).

6. Apparatus

6.1 Fall-Time Runs:

6.1.1 *Falling-Rod Viscometer*,³ equipped with a swinging platform and automatic timing device⁴ accurate to at least 0.1 s, preferably 0.01 s. A special lightweight rod is useful for liquids in the 10-P range.

6.1.2 *Set of Tapped or Slotted Weights*—Weights of 50 or 100 to 500 g are usually provided with the instrument. Extra 500-g weights, approximately 4, totaling about 2000 g are required to handle fluids at the upper end of the practical

range. A 25-g weight is useful for liquids in the 10-P range.

6.1.3 *A Thermostatically Controlled Cabinet*, optional (if room conditioning is not available). Alternatively, a special collar⁵ through which water is circulated from a constant-temperature bath.

6.1.4 *Thermistor*,⁶ equipped with a probe having a response time of 3 to 6 s.

6.1.5 *Ring Stand and Clamp*, or other device for holding the thermistor probe in a suitable position.

6.1.6 *Small Plastic Spatula*—Metal spatulas are not suitable.

6.1.7 *Plastic Scraper*, consisting of a piece of flexible plastic, approximately 30 by 70 mm, having a semicircle cut out at one end; semicircle should fit the rod.

6.2 Instrument Calibration:

6.2.1 *Balance*, weighing to 0.1 g.

6.2.2 *Metric Rule or Scale*, at least 100 mm in length.

6.2.3 *Vernier Caliper*, accurate to 0.01 mm, having a capacity of at least 30 mm.

6.3 Graphical Solutions:

6.3.1 *Chart Paper*, logarithmic 2×2 to 2×3 cycles.⁷

6.3.2 *Triangle*, 45°, with a hypotenuse length of at least 100 mm (approximately 8 in.).

6.3.3 *Protractor*.

7. Materials

7.1 *ASTM Standard Viscosity Oils*,⁸ a minimum of two, preferably three, spanning the practical range of the falling-rod viscometer (used for calibration purposes only).

7.2 *Lithographic Varnish* or similar vehicle having a viscosity of about 200 P.

7.3 *Lint- and Metal-Free Rags or Tissues*.

7.4 *Naphtha* or other low-boiling solvent in a wash bottle or closed metal container.

8. Hazards

8.1 *Safety Precautions*—Since solvents may be hazardous to the skin and eyes, in addition to other precautions, wear rubber gloves and safety glasses during cleanup to avoid solvent contact with skin and eyes. In case of contact, wash skin with water; flush eyes for 15 min with water and call a physician. See supplier's Material Safety Data Sheets for further information on each solvent used.

8.2 Instrument Cautions:

8.2.1 Avoid any operation that will scratch the rod. Do not use a metal spatula. Never drop the rod through an empty aperture.

8.2.2 Weight loads in excess of 3000 g may cause bending of the rod.

³ Collars are available as accessories from the respective manufacturers.

⁶ Suitable thermistors include Tele-Thermometer Model 43TA, with Model 423 probe from Yellow Springs Instrument Co., Inc., Box 279-T, Yellow Springs, OH 45387; and Model TSB with Model TP-7P probe from TRI-R Instruments Inc., 48 Merrick Rd., Rockville Centre, NY 11570.

⁷ Suitable chart paper includes Keuffel & Esser Nos. 46-7200 (22), 46-7280 (22.7), 46-7320 (23), all 8½x11 green drawing; also available in green or orange tracing or 11x16½.

⁸ Certified standard viscosity oils are available from Cannon Instrument Company, P.O. Box 16, State College, PA 16801. Table 3 in Test Method D 445 shows satisfactory oils including S-600 (16 P at 25°C), S-2000 (57 P), S-8000 (220 P), and S-30 000 (750 P). Viscosity at various temperatures is indicated on the label of each container.

³ Commercial units include the Laray Viscometer available from Testing Machines, Inc., 400 Bayview Ave., Amityville, NY 11701; Thwing-Albert Model LR from Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, PA 19154; Churchill from Churchill Instrument Co., Ltd., Greenford, England.

⁴ Platform and timing device are standard on newer viscometer models. For equipping older models, see Bassenir, R., "Evaluation of the Laray Viscometer," *American Ink Maker*, Vol 39, No. 4, April 1961, pp. 24-26 and 60.

8.2.3 To minimize heat buildup from body temperature during a run, avoid contacting the viscometer block with bare hands. When instructions call for holding the block steady, wear a glove or place a small cloth in the palm of the hand.

8.2.4 When making fall-time measurements, work quickly and without interruption so that the entire run is completed within 5 to 10 min.

NOTE 1—Many modern printing inks and vehicles contain some solvent, and volatile loss during a run can seriously bias test results unless rigorous control of exposure time is exercised. Volatile loss can be detected if successive drops of the rod with the same weight result in increasingly longer fall times.

9. Preparation of Apparatus

9.1 Set the viscometer on a sturdy bench located in an area free of direct drafts, direct sunlight, and other sources of heat. Level the viscometer by turning the adjustable feet up and down until the spirit level bubble is centered.

9.2 Pass a hand over the upper and lower photocells to assure that the timer is activated and deactivated.

9.3 Attach the clamp to the ring stand and place next to or behind the viscometer. Drape the thermistor probe over the clamp; reset the clamp so that the probe end falls close to the viscometer block.

9.4 Clean the block and rod thoroughly with tissues wetted with naphtha. Remove residual solvent with clean dry tissue. Roll the clean dry rod over a flat surface to check for straightness. If rod is bent, discard and obtain a new rod/orifice set.

9.5 Examine the markings at the ends of the rod. Select one marking as an indication of the "proper" end to be always inserted into the aperture first.

10. Calibration

10.1 Determine instrument constants in accordance with the procedure given in Annex A1.

10.2 *Optional*—If a graphical method is to be used for direct conversion of test results to viscosity, prepare Master Sliding Scale Calibration Graph as in Annex A2.

10.3 Periodically check calibration as in Section A1.2.

11. Sample Preparation

11.1.1 Transport the sample to the test area and preserve in a closed container. Skin paper should be used for oxidative drying inks.

11.1.2 Ink samples should be uniform dispersions. If pigment settling is suspected, insert a spatula in the container and gently stir. Be careful not to introduce air bubbles.

11.1.3 Prior to the run, a portion of the sample may be transferred to a slab and gently spread out in order to remove bubbles, skin, or other debris.

NOTE 2: *Caution*—Do not work the sample vigorously; this practice causes a significant increase in sample temperature. Be sure to close the container immediately after removing the desired portion.

12. Conditioning

12.1 The temperature of the room (cabinet or collar) should be set at $23 \pm 1^\circ\text{C}$ (or 2°C below the reference temperature).

NOTE 3—In accordance with Note 8, the allowable range for specimen temperature is $\pm 2^\circ\text{C}$ from the reference temperature. However, during the course of testing, heat of shearing and body heat of the operator both contribute to continuous temperature rises in test specimens, notwithstanding room, cabinet, or collar conditions. To allow for inevitable temperature rises, temperature controls are set at the lower end of the allowable range.

12.2 Equilibration of test samples is not necessary. Specimen sizes are small (less than 2 mL); when spread out on a slab and applied to the viscometer, both hot and cold samples quickly reach the temperature of the metal.

12.3 If the viscometer has been idle for more than an hour, it may be necessary to bring it into equilibrium with the conditioning temperature (23°C or other specified in 12.1). Make preparations for an exploratory run (13.2 to 13.5) using a varnish if the test specimen contains volatiles. Read specimen temperature; if too low (a possibility considering that metal serves as a heat sink), add a 1000-g weight to the rod and make a few drops (13.7 to 13.9 but without recording time) until the specimen temperature reaches that of conditioning. Continue the run or, if a varnish was used, clean up.

13. Procedure for Fall-Time Runs

13.1 If required, prepare, level, and condition the instrument as described in 10.12.1 and 12.3.

13.2 With the proper end of the clean rod down, hold the rod vertically over the clean aperture and gently lower until it rests on the swinging platform.

13.3 Transfer a uniform specimen to the tip of a clean plastic spatula. The specimen size should be sufficient to fill the well of the viscometer.

13.4 Hold the rod with the fingertips and carefully raise about 20 mm. Transfer the specimen from the spatula to the rod as close as possible to the bottom of the well. Rotate the rod slowly to distribute the specimen around the well, ensuring that the well is full. Allow the rod to fall to the platform.

13.5 Place the thermistor probe in the well close to but not touching the rod. The probe can remain in the well throughout the run. Turn the thermistor on.

13.6 Using experience or the information in Table 1 as a guide, select a weight load that will produce a fall time as close to 1 or 2 s as is practical.

NOTE 4—For comparison of non-Newtonian liquids, runs must not be made at pre-specified rod weights. Rather, weights should be adjusted to obtain pre-specified fall times, the first of which corresponds as closely to a shear rate of 2500 s^{-1} as is practical.

13.7 Hold the block level and steady with one hand (see 8.2.3). With the other hand, carefully place the selected weights on top of the rod. If weights are slotted, evenly distribute the slots around the circumference of the rod. Make certain that the rod is vertical. (If weights tend to make the viscometer unsteady, retain the hand on the block so that the rod falls smoothly in 13.8.)

13.8 Set the timer. Release the platform and allow the rod to fall naturally. If the fall time is within the desired range (for example, 1 to 2 s for the first weight, etc.), record the added weight W_A , fall time F , and specimen temperature T on worksheet.

13.9 Remove the weights from the rod. Pull the rod up

TABLE 1 Weight/Fall-Time Relationships for Newtonian Liquids^A
Rod weight = 130 g

Viscosity of Fluid, P	Fall Time, s					
	1	2	4	6	10	20
10	200	50	...	...	...	...
50	1 300	700	300	100	50	...
100	3 000	1 400	700	400	150	25
250	7 000 ^B	3 800 ^B	1 800	1 100	700	300
500	15 000 ^B	7 000 ^B	3 200 ^B	2 200	1 400	600
1000	25 000 ^B	16 000 ^B	8 000 ^B	5 500 ^B	3 000	900

^AWeights required may be more or less depending on instrument type and degree of wear. Printing inks will require additional weight depending on degree of Newtonianism.

^BWeights are impractical or not recommended.

13.9 Repeat the drop (13.7 to 13.9) with the same or adjusted weights until two fall times with a specific set of weights agree within 2 % (0.04 s at a 2-s fall time, 0.2 at a 20-s fall time, etc.).

13.10 Repeat the drop (13.7 to 13.9) with the same or adjusted weights until two fall times with a specific set of weights agree within 2 % (0.04 s at a 2-s fall time, 0.2 at a 20-s fall time, etc.).

NOTE 5—Newtonian liquids may be run with only one or two sets of weights. Non-Newtonian liquids require at least four or five.

13.12 If the specimen is depleted during the run, clean the rod and start over from 13.1, preferably using ink fresh from the container. Make certain that the quantity of specimen is sufficient.

13.13 Immediately after completing the run, turn the thermistor off, remove the probe from the well, and clean the probe, the viscometer orifice, and the rod thoroughly.

NOTE 6—Since each test involves replicate fall-time measurements at up to five weights, a single viscosity determination is usually considered adequate.

14. Calculation

NOTE 7—This section covers calculations by computer or programmable calculator. The list of symbols is given in 3.3. The procedure for graphical solution of test results is described in the Annex A2.

14.1 Enter into the computer the values for the instrument constants, W_R , F_{2500} and K_{2500} and the reference temperature T_R .

14.2 Enter data from fall-time runs in sets of W_A , replicate values of F that agree within 2 %, and the corresponding values of T .

14.3 Compute the non-Newtonianism parameter N by simultaneous solution of the following general equation:

$$\log W = B - N \log F_c \quad (1)$$

where:

$$W = W_A + W_R \quad (2)$$

and

$$F_c = F + 0.1 F (T - T_R) \quad (3)$$

NOTE 8—Equation 3 corrects each measured fall time by 10 % per degree differential between the measured temperature and the reference

temperature and is applicable only within 2°C of T_R . As noted in the third column of Table A1.2, specimen temperature increases progressively during a run. For accurate results, it is important that the temperature correction be applied to the fall time corresponding to each added weight. Otherwise, significant error will be introduced into the slope of the power law plot.

14.4 Examine (by computer) the value of N . Any value over 1.0 is improbable for a printing ink or a vehicle and suggests error in the test measurements; check data or repeat runs. Alternatively, treat any value between 1.0 and 1.05 as 1.0.

14.5 Compute the viscosity at 2500 s⁻¹ as follows:

$$V_{2500} = K_{2500} W_{2500} \quad (4)$$

where:

$$W_{2500} = \text{antilog} (B - N \log F_{2500}) \quad (5)$$

14.6 *Optional*—Compute the viscosity at 2.5 s⁻¹ from either Eqs 6 or 7 as follows:

$$V_{2.5} = V_{2500} (1000)^{1-N} \quad (6)$$

or

$$V_{2.5} = 1000 K_{2500} \text{antilog} (B - N \log 1000 F_{2500}) \quad (7)$$

14.7 *Optional*—Compute the pseudo yield value as follows:

$$S'_0 = 2.5 (V_{2.5} - V_{2500}) \quad (8)$$

NOTE 9—Since the logarithmic nature of the power law precludes a zero shear rate, Eq 8 was derived to approximate the Bingham yield value, which is normally determined by extrapolating the linear portion of a shear stress/shear rate plot to zero rate of shear.

14.8 *Optional*—Compute the shortness factor as follows:

$$SF = S'_0 / V_{2500} \quad (9)$$

15. Report

15.1 Report apparent viscosity at 2500 s⁻¹, degree of non-Newtonianism, reference temperature, and identifying code referring to the specific viscometer.

15.2 *Optional*—Report the viscosity at 2.5 s⁻¹, the pseudo yield value, and the shortness factor.

16. Precision

16.1 An interlaboratory study of this test method was conducted in which a single operator in each of nine laboratories made one run consisting of at least four replicated data points on four inks on two different days. The inks ranged in viscosity from 10 to 300 P. The results were calculated on a single programmable calculator. One labora-

TABLE 2 Precision of Falling-Rod Viscosity Determinations

Test Results	Standard Deviation, % relative	Degrees of Freedom	Maximum Allowable Difference, % relative
Repeatability			
<i>N</i>	3.3	4	7.6
<i>V</i> ₂₅₀₀	5.4	4	15.3
<i>V</i> _{2.5}	11.5	4	31.4
<i>S</i> _o	14.6	3	41.2
<i>SF</i>	17.7	3	50.1
Reproducibility			
<i>N</i>	4.6	28	12.9
<i>V</i> ₂₅₀₀	7.9	28	22.3
<i>V</i> _{2.5}	17.5	21	49.5
<i>S</i> _o	22.3	21	63.1
<i>SF</i>	23.6	21	66.9

tory was a consistent outlier and was deleted from the entire analysis, and the 10 P ink was deleted from analysis of the low shear parameters. The estimated standard deviations and

the degrees of freedom are given in Table 2. (Since the standard deviation was proportional to the test value, precision statements are made in terms of percent of the observed value.) Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

16.1.1 *Repeatability*—Two results obtained by the same operator on different days should be considered suspect if they differ by more than the maximum allowable difference indicated in Table 2.

16.1.2 *Reproducibility*—Two results, each the mean of results obtained on different days by operators in different laboratories, should be considered suspect if they differ by more than the maximum allowable difference indicated in Table 2.

17. Keywords

17.1 printing inks; inks; vehicles; viscosity; apparent viscosity; non-Newtonianism; yield value; power law viscosity; shortness; viscometers; falling-rod viscometers

ANNEXES

(Mandatory Information)

A1. CALIBRATION OF FALLING-ROD VISCOMETERS

A1.1 Determination of Instrument Constants:

A1.1.1 Measure the distance between photocells of the timing device. Adjust to 100 ± 0.5 mm. Divide by 10 and record as *L* in centimetres in chart patterned after Table A1.1.

A1.1.2 Weigh the clean rod to 1 g. Record as *W_R*.

A1.1.3 Using a caliper, measure the diameter of the rod to 0.01 mm. Record as *d* in centimetres.

A1.1.4 With the caliper, measure the length of the block over which the liquid is sheared (total height of block minus ink well) to 0.01 mm. Record as *h* in centimetres.

A1.1.5 Calculate apparent shearing area from the equation $A = \pi dh$. Record as *A* in square centimetres.

NOTE A1.1—The shearing length of the block contains both a tapered and a parallel section; therefore, it is understood that *A* is not the true shearing area but an apparent shearing area.

A1.1.6 The thickness *x* of the test liquid is set by the clearance between the aperture and the rod. Since the aperture consists of two radii, both of which are difficult to measure, a value for a mean gap clearance $\bar{x}$ can be

TABLE A1.1 Instrument Constants for Falling-Rod Viscometers

Section	Symbol	Definition	Unit	Typical values	
				Laray	Thwing-Albert
A1.1.1	<i>L</i>	timed fall distance (distance between photocells)	cm	10.0	10.0
A1.1.2	<i>W_R</i>	weight of rod	g	130	130
A1.1.3	<i>d</i>	diameter of rod	cm	1.20	0.80
A1.1.4	<i>h</i>	height of shearing portion of aperture	cm	2.80	4.20
A1.1.5	<i>A</i>	apparent shearing area = πdh	cm ²	10.5	10.5
A1.1.6	$\bar{x}$	mean gap clearance $\bar{x} = [AL/g][V/WF_c]^\dagger$	cm or μ m	0.0045	0.0028
	<i>V</i>	viscosity of calibrating oil at reference temperature	P	45	28
	<i>g</i>	gravitational constant (approx. 980 cm/s ²)	cm/s ²		
	<i>W</i>	total weight = rod weight + added weight	g		
	<i>F_c</i>	corrected fall time	s		
A1.1.7	<i>F</i> ₂₅₀₀	fall time corresponding to shear rate of 2500 s ⁻¹ = $L/2500 \bar{x}$	s	0.8	1.4
A1.1.8	<i>K</i> ₂₅₀₀	apparent viscosity constant at 2500 s ⁻¹	cm ⁻¹ s ⁻¹	0.0372	0.0372
		$K_{2500} = \frac{\eta}{2500A}$			

† Editorially corrected.

TABLE A1.2 Typical Worksheet for Falling-Rod Viscometer

Run #		GG32		Test sample Calibrating Oil S-2000, V = 45.4		Date of run 1-10-80		
				Instrument Laray LV #1		Rod weight 130 g		
				Reference temperature 25°C		Room temp. 22.8°C		
Test Measurements			Mean Results			Corrected Results		
Added Load W_A , g	Recorded Fall Time F , s	Specimen Temperature T , °C	Mean Fall Time ^A $\bar{F}$, s†	Mean Specimen Temperature ^A $\bar{T}$, °C	Temperature Difference $(\bar{T}-25)$ ^a , °C	Corrected Fall Time ^C F_c , s	Total Weight ^D W , g	Newtonian Viscosity Multiplier ^E WF_c , g·s
600	1.45	24.49	1.57	24.61	-0.39	1.51	730	1102
	1.55	24.55						
	1.59	24.66						
400	2.02	24.70	2.02	24.76	-0.24	1.97	530	1044
	2.02	24.82						
200	3.35	24.97	3.35	24.99	-0.01	3.35	330	1105
	3.27	24.98						
	3.35	25.00						
100	4.72	25.03	4.73	25.03	+0.03	4.74	230	1090
	4.85	25.03						
	4.75	25.04						
25	7.27	25.11	7.26	25.08	+0.08	7.28	155	1126
	7.25	25.07						
								Mean WF_c 1097

Using results only for fall times that agree within 2 %.
 The number 25 refers to the reference temperature. Change figure if appropriate.
 Formula: $F_c = F + 0.1 F (\bar{T} - 25)$.†
 † Weight of rod plus added load.
 Complete this column only if sample is a calibrating oil or a Newtonian fluid.
 Editorially corrected.

computed from fall-time runs on Newtonian oils in the following manner:

A1.1.6.1 Using the procedure described in Section 13, make fall-time runs on two or three standard oils in random sequence on two different days.

A1.1.6.2 For each added weight, compute the mean of fall times that agree within 2 %. Also compute the mean of the corresponding temperature. Record as $\bar{F}$ and $\bar{T}$ on worksheet (see Table A1.2). Correct each mean fall time according to Eq. 3, (see 14.3). Record as F_c .

A1.1.6.3 Take the sum of each added weight and the rod weight. Record as the total weight, W .

A1.1.6.4 Multiply each total weight by the corresponding corrected fall time and record as WF_c . Examine trends in WF_c within each run. Values that change progressively with weight are indicative of inadequate temperature sensing or non-Newtonianism in the oil. Check the source of error or repeat with new oil if required.

A1.1.6.5 Compute a mean WF_c for each run and divide into the viscosity of the oil at 25°C (or other reference temperature). The resulting values of V/WF_c should be essentially the same for all oils. Compute the mean V/WF_c .

NOTE A1.2—If the label on the oil container does not list viscosity specifically at the reference temperature, the desired viscosity may be obtained from plots of log viscosity versus reciprocal of temperature on semi-logarithmic chart paper.

A1.1.6.6 Compute the mean gap clearance $\bar{x}$ by multiplying V/WF_c by AL/g . Record to three significant figures in centimetres (10^{-4} cm = 1 μ m).

A1.1.7 Calculate the fall time corresponding to a shear rate of 2500 s^{-1} from the equation: $L/2500 \bar{x}$. Record as F_{2500} in seconds to three significant figures.

A1.1.8 Compute $g/2500 A$ and record as the "apparent viscosity constant at 2500 s^{-1} ," K_{2500} , in $cm \cdot s^{-1}$.

A1.2 Recalibration:

A1.2.1 Periodically determine several corrected fall times with one oil used for the original calibration.

A1.2.2 Calculate values for WF_c and compare with those originally obtained for the oil.

A1.2.3 If the new values do not reasonably agree with the original ones, use the new fall time results to calculate a new value for the mean gap clearance $\bar{x}$. If $\bar{x}$ increases by less than 5 % of the original value, the existing calibration may be retained. If the increase is between 5 and 20 %, repeat the entire calibration process. If gap clearance increases by 20 % or more, replace the rod and aperture.

A2. GRAPHICAL SOLUTION OF VISCOSITY

A2.1 Construction of Sliding Scale Calibration Graph:

A2.1.1 On a sheet of 2 by 2 to 2 by 3 cycle logarithmic

chart paper, label the 2-cycle scale "Corrected fall time, s." (See Fig. A2.1.) Add one zero to the second cycle. Mark

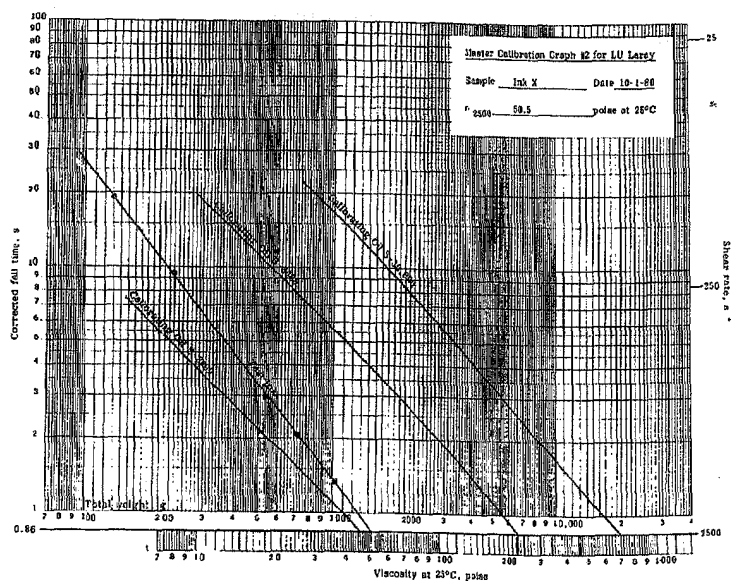


FIG. A2.1 Typical Sliding Scale Calibration Graph

position of F_{2500} (from A1.1.7) on time scale. If below 1.0 s, the position can be located using another sheet of log paper as a guide. Draw a horizontal line across the entire sheet of paper and label "2500 s^{-1} " at the far end.

A2.1.2 Along the 3-cycle axis, mark the start of the cycles as 100, 1000, and 10 000. Label the scale "Total weight, g" so as not to interfere with the line previously drawn for "2500 s^{-1} ."

A2.1.3 Using the the results from fall-time runs made with standard oils (A1.1.6.1) and found satisfactory in accordance with A1.1.6.4, plot corrected fall time versus total weight for each oil-day combination. Using the triangle, draw the best 45° line through the points so that each plot line intercepts the line labeled "2500 s^{-1} ."

A2.1.4 From a second sheet of the same type of chart paper, cut a piece along the 3-cycle axis so that the resulting strip contains the scale plus one or two graph divisions. Add zeros to the scale so that the second cycle starts at 10 and the third at 100; if required for high-viscosity fluids, a fourth cycle starting at 1000 may be obtained by pasting two strips together. Label the strip "Viscosity at 25°C, P." Locate the 25°C viscosity of each calibrating oil on the scale and draw vertical lines to the top. If three oils were used, there should be three lines on the strip.

A2.1.5 Position the top of the strip directly beneath the 2500 s^{-1} line drawn on the first sheet of chart paper so that the viscosity lines on the strip match as closely as possible the intercepts at 2500 s^{-1} . Paste securely in place. Cut off portions of the strip that extend beyond the edges of the chart paper. Relabel "Total weight" scale if strip hides pertinent coordinates.

A2.1.6 The chart paper containing the viscosity strip represents the "Master Sliding-Scale Calibration Graph" for

a specific instrument. Prepare an overlay containing the desired identification information and paste in a suitable place (see example in Fig. A2.1). Make sufficient copies of the master for normal use of the instrument. Be sure to preserve the original.

NOTE A2.1—Use a duplication procedure that reproduces copy exactly. Office copiers and "quick" printing methods are prone to provide distorted copy.

NOTE A2.2—The Master Sliding-Scale Calibration Graph is essentially similar to the Inmont-Lehman chart, which is also based on the power law but presumes that a shear rate of 2500 s^{-1} corresponds to a fall time of 1.0 s in all instruments. The charts issued by viscometer manufacturers are Cartesian ($1/F$ versus W) and are based on Bingham

A2.2 Graphical Solution of Viscosity:

A2.2.1 Make the fall-time run on the test sample according to Section 13. Compute the corrected mean fall times and the total weights according to A1.1.6.2 and A1.1.6.3.

A2.2.2 On a copy of the Master Sliding Scale Calibration Graph (Section A2.1), plot corrected fall time versus total weight for the test material.

A2.2.3 Using the 45° triangle as a guide, check whether the best straight line through the plotted points is 45° or greater to the weight axis. A line less than 45° is improbable for a printing ink or a vehicle and suggests error in the test measurements; check calculations or repeat the run.

A2.2.4 Draw the best straight line through the points, making certain that the angle is at least 45° to the weight axis. Extend the line so that it intersects the horizontal axis at 2500 s^{-1} . Read viscosity as the intercept on the sliding scale. Record as V_{2500} , apparent viscosity at 2500 s^{-1} .

A2.2.5 Center the protractor at the intercept and read the angle of the plot line. Subtract the measured angle from 90° and obtain the tangent from mathematical tables or a

table calculator. Record as the non-Newtonianism parameter N .

NOTE A2.3—By turning Fig. A2.1 90° to the left, the graph can be viewed as a logarithmic plot of shear stress versus shear rate, and the

shear thinning nature of the test ink made more evident. The tangent of the angle with respect to the time (shear rate) axis is the power law constant N .

A2.2.6 *Optional*—Calculate $V_{2.5}$, $S'_{0.0}$, and SF according to Eq 6 (see 14.6), Eq 8 (see 14.7) and Eq 9 (see 14.8).

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser¹

This standard is issued under the fixed designation D 4060; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the resistance of organic coatings to abrasion produced by the Taber Abraser on coatings applied to a plane, rigid surface, such as a metal panel.

1.2 Because of the poor reproducibility of this test method, it should be restricted to testing in only one laboratory when numerical abrasion resistance values are to be used. Interlaboratory agreement is improved significantly when rankings of coatings are used in place of numerical values.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- D 2240 Test Method for Rubber Property—Durometer Hardness³

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 Abrasion resistance can be expressed as one or more of the following terms:

3.1.1.1 *wear index*—1000 times the loss in weight in milligrams per cycle.

3.1.1.2 *weight loss*—the loss in weight in milligrams,

determined at a specified number of cycles.

3.1.3 *wear cycles per mil*—the number of cycles of abrasion required to wear a film through to the substrate per mil of film thickness.

4. Summary of Test Method

4.1 The organic coating is applied at uniform thickness to a plane, rigid panel and, after curing, the surface is abraded by rotating the panel under weighted abrasive wheels.

4.2 Abrasion resistance is calculated as loss in weight at a specified number of abrasion cycles, as loss in weight per cycle, or as number of cycles required to remove a unit amount of coating thickness.

5. Significance and Use

5.1 Coating on substrates can be damaged by abrasion during manufacturing and service. This test method has been useful in evaluating the abrasion resistance of attached coatings. Ratings produced by this test method have correlated well with ratings produced by the falling abrasive values in Test Method D 968.

6. Apparatus

6.1 Taber Abraser.⁴

6.2 *Abrasive Wheels*—Resilient calibrase wheels No. CS-10 or CS-17, as required, shall be used. Because of the slow hardening of the rubber bonding material in this type of wheel, the wheels should not be used after the date marked on them, or one year after their purchase if the wheels are not dated.

NOTE 1—The hardness of the wheels can be checked by Test Method D 2240. An acceptable hardness for both types of wheels is 81 ± 5 units on Shore Durometer A-2 Scale.

NOTE 2—The CS-17 wheels produce a harsher abrasion than the CS-10 wheels.

6.3 *Resurfacing Medium*, an S-11 abrasive disk, used for resurfacing the abrasion wheels.

6.4 *Vacuum Pick-Up Assembly*, consisting of a vacuum unit, a variable transformer suction regulator, a nozzle with bracket attachment, and a connecting hose with adaptor.

7. Test Specimens

7.1 Apply a uniform coating of the material to be tested to a plane, rigid panel. Specimens shall be a disk 4 in. (100 mm) in diameter or a plate 4-in. (100-mm) square with rounded corners and with a 1/4-in. (6.3-mm) hole centrally located on

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved May 25, 1990. Published July 1990. Originally published as D 4060 - 81. Last previous edition D 4060 - 84.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 09.01.

⁴ Available from Teledyne Taber, North Tonawanda, NY 14120.

each panel. Prepare a minimum of two coated panels for the material.

NOTE 3—The coatings should be applied in accordance with Test Methods D 823, or as agreed upon between the purchaser and the seller.

NOTE 4—The thickness of the dry coatings should be measured in accordance with Test Methods D 1005, D 1186, or D 1400.

Standardization

8.1 Mount the selected abrasive wheels on their respective change holders, taking care not to handle them by their abrasive surfaces. Adjust the load on the wheels to 1000 g.

8.2 Mount the resurfacing medium (S-11 abrasive disk) on the turntable. Lower the abrading heads carefully until the wheels rest squarely on the abrasive disk. Place the vacuum pick-up nozzle in position and adjust it to a distance of 1/32 in. (1 mm) above the abrasive disk.

8.3 Set the counter to "zero" and set the suction regulator to approximately 50 points on the dial. The setting may be increased to 90 if more effective removal of the abrasings appears necessary.

8.4 Start the vacuum pick-up and then the turntable of the abrader. Resurface the wheels by running them 50 cycles against the resurfacing medium.

NOTE 5—The wheels should be resurfaced in this manner before testing each specimen and after every 500 cycles.

Conditioning

9.1 Cure the coated panel under conditions of humidity and temperature as agreed upon between purchaser and seller.

9.2 Unless otherwise agreed upon between purchaser and seller, condition the coated panel for at least 24 h at $23 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ relative humidity. Conduct the test in the same environment or immediately on removal therefrom.

Procedure

10.1 Weigh the test specimen to the nearest 0.1 mg and record this weight, if either the wear index or the weight loss is to be reported.

10.2 Measure the coating thickness of the test specimen in several locations along the path to be abraded.

10.3 Mount the test specimen on the turntable. Place the abrading heads on the test film and the vacuum pick-up nozzle in position as outlined in 8.2. Set the counter and suction regulator as outlined in 8.3.

10.4 Start the vacuum pick-up and then the turntable of the abrader. Subject the test specimen to abrasion for the specified number of cycles or until wear through of the coating is observed. In determining the point of wear through, stop the instrument at intervals for examination of the test specimen.

10.5 Remove any loose abrasings remaining on the test specimen by light brushing. Reweigh the test specimen.

10.6 Repeat 10.1 to 10.5 on at least one additional test specimen of the material under test.

11. Calculation

11.1 *Wear Index*—Compute the wear index, I , of a test specimen as follows:

$$I = \frac{(A - B) 1000}{C}$$

where:

A = weight of test specimen before abrasion, mg,
 B = weight of test specimen after abrasion, mg, and
 C = number of cycles of abrasion recorded.

NOTE 6—In calculating wear index it may be advisable to discard the last 200 cycles because the results may be affected by abrasion of the exposed substrate.

11.2 *Weight Loss*—Compute weight loss, L , of the test specimen as follows:

$$L = A - B$$

where:

A = weight of test specimen before abrasion, mg, and
 B = weight of test specimen after abrasion, mg.

11.3 *Wear Cycles Per Mil*—Compute the wear cycles per mil, W , of the test specimen as follows:

$$W = D/T$$

where:

D = number of cycles of abrasion required to wear coating through to substrate and
 T = thickness of coating, mils (0.001 in.) (to one decimal place).

NOTE 7—In calculating the wear cycles, it is advisable to discard the first and last readings because the first may be affected by an uneven surface and the last by abrasion of parts of the substrate.

12. Report

12.1 Report the following information for each test material:

12.1.1 Temperature and humidity during conditioning and at the time of testing,

12.1.2 Thickness of coating when wear cycles are specified,

12.1.3 Kind of calibrase abrasive wheels used,

12.1.4 Load applied to the abrasive wheels,

12.1.5 Number of wear cycles recorded for each test specimen,

TABLE 1 Precision of Taber Abrasion Values

	Within Laboratory		Between Laboratories	
	Coefficient of Variation, %	Maximum Allowable Difference, %	Coefficient of Variation, %	Maximum Allowable Difference, %
Weight loss at 500 cycles	12	48	36	105
Weight loss at 1000 cycles	10	46	30	90
Wear index at 500 cycles	13	52	36	106
Wear index at 1000 cycles	10	46	30	92
Cycles per mil	13	44	31	92

12.1.6 Wear index, weight loss, or wear cycles per mil for each test specimen, and

12.1.7 Mean and range of the abrasion resistance values of the replicate coated panels.

13. Precision⁵

13.1 On the basis of an interlaboratory test of this test method in which operators in five laboratories tested four coatings having a broad range of abrasion resistance, the within-laboratory coefficients of variation and between-laboratories coefficients of variation were found to be those in Table 1. Based upon these coefficients, the following

criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.1 *Repeatability*—Two results by the same operator should be considered suspect if they differ by more than the maximum allowable difference values shown in Table 1.

13.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than the maximum allowable difference values shown in Table 1.

NOTE 8—When this test method is used to rank a series of coatings by magnitude of abrasion resistance, the precision is significantly better than shown in Table 1. In the interlaboratory study for evaluating precision, all laboratories ranked the coatings in the same order of abrasion resistance.

⁵ Supporting data are available from ASTM Headquarters. Request RR: D01-1037.

14. Keywords

14.1 wear index

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Standard Test Method for Leveling of Paints by Draw-Down Method¹

This standard is issued under the fixed designation D 4062; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1. This test method² covers the laboratory determination of the relative leveling of water and solvent-reducible architectural paints in white and light tints by comparing the ridges produced in a draw-down film to a series of plastic leveling standards.

2. Unpigmented, texture, and deep-tint coatings cannot be readily evaluated with the shadowing produced by oblique lighting employed in this test method. Such coatings may be evaluated by comparing them with the plastic standards at various angles of reflection. For this purpose ordinary room lighting is satisfactory.

3. Since other factors may influence the tendency of liquid paints to sag, this test method is not intended to measure sagging.

4. *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Description of Term Specific to This Standard

1.1 *leveling*—of a paint, a measure of its ability to flow after application so as to obliterate any surface irregularities such as brush marks, orange peel, peaks, or craters that have been produced by the mechanical process of application.

Summary of Test Method

1.2 The material to be tested is presheared and then applied to a sealed chart by means of a special leveling test blade designed to lay down a film with parallel ridges simulating brush marks. After allowing the completed draw-down to dry in a horizontal position, leveling of the test paint is rated by viewing the draw-down under a strong, oblique light source and comparing the contrast of lightness and shadow caused by the paint ridges to that of a series of plastic leveling standards under the same lighting conditions.

Significance and Use

1.3 Instrumental evaluations of leveling by this test

method have been shown to correlate with those made by brush application. Leveling can affect the hiding and appearance of applied architectural coatings, the presence of brushmarks and surface irregularities being more conspicuous with gloss and semi-gloss finishes than with flat finishes.

5. Apparatus

5.1 *Leveling Test Blade*^{3,4}—A grooved draw-down blade designed to lay down a wet film with parallel ridges (see Fig. 1).

5.2 *Draw-Down Plate*,⁵ with two parallel, smooth-faced straightedges to guide the blade during film application and ensure that the ridges are straight.

5.3 *Syringe and Needle*—A 10-mL LuerLok syringe and 1½-in. (38-mm) No. 15 gage needle for placing a fixed amount of the paint in front of the blade while simultaneously preshearing it (as during brushing) prior to drawing-down. Either glass or disposable plastic syringes may be used with water-reducible paints. Glass syringes only may be used with solvent-reducible paints due to swelling of disposable syringes by the solvent.

5.4 *Test Chart*,⁶ plain white (for white or light tints) or predominantly black chart (for deep tints) coated with a suitable varnish or lacquer to render the test surface impervious to the volatile portion of the paint.

5.5 *Light Source*,⁷ oblique, to illuminate the test draw-down and leveling standards (see Fig. 2).

5.6 *Levelness Standards*⁸—Three-dimensional full-scale replicas of draw-downs made with the leveling test blade and nine paints exhibiting very poor to very good leveling, for comparison with the test draw-down.

5.7 *Catch Papers*, disposable, any type for catching excess paint is adequate.

³ The Leneta Leveling Test Blade, available from the Leneta Company, P. O. Box 86, Ho-Ho-Kus, NJ 07423, has been found satisfactory for this purpose.

⁴ The Leneta Leveling Test Blade used in this test method is a modification of the threaded draw-down bar described in an article by Dodge, J. S. "Quantitative Measures of Leveling," *Journal of Paint Technology*, Vol 44, No. 564, January 1972.

⁵ The Leneta Leveling Test Draw-Down Plate has been found satisfactory for this purpose. Available from the Leneta Company. A suitable draw-down plate may be constructed from the description given in 5.2 of this test method.

⁶ Leneta Form WB, a plain white sealed chart, and Leneta Form 7B, a black and white sealed chart, available from the Leneta Company, have been found suitable for light- and deep-tint paints, respectively. Equivalents may be used.

⁷ A standard oblique fluorescent light source (The Level-Luminator), has been found satisfactory for this purpose. Available from the Leneta Company. A suitable light source may be constructed from the description given in 6.5 and Fig. 2 of this test method.

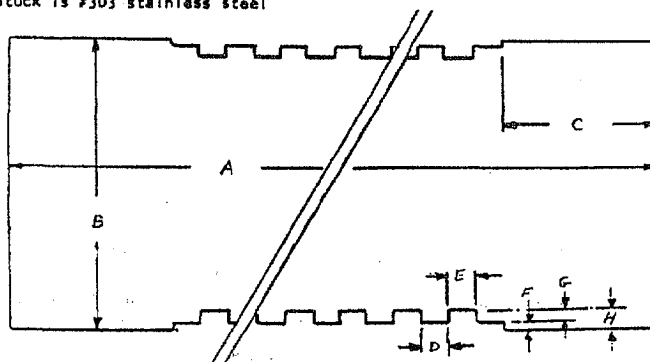
⁸ Leneta Draw-Down Levelness Standards, a series of nine 3 by 5 in. (75 by 125 mm) plastic full-scale replicas of draw-downs of paints having very poor to very good leveling, have been found satisfactory for this purpose. Available from the Leneta Company.

This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved May 27, 1988. Published October 1988. Originally published as D 4062 - 81. Last previous edition D 4062 - 81 (1987).

This test method was essentially developed by the Leneta Company as outlined in Leneta Catalog No. 3, pp. 26-7 (1976).

Stock is #303 stainless steel



Dimensions:

	mm	in. (approx.)
A—Length	165	6.5
B—Diameter	22	0.87
C—Supporting edge	25	1.0
D—Minor doctoring edge	1.25	0.05

Dimensions:

	mm	in. (approx.)
E—Major doctoring edge	1.25	0.05
F—Minor clearance	0.10	0.004
G—Clearance step	0.20	0.008
H—Major clearance	0.30	0.012

NOTE—This is actually a cylindrical rod, the term "blade" being employed as a conventional reference to film applicators. Auxiliary plastic side arms not shown. See Figs. 1 (b) and 1 (c).

FIG. 1 (a) Leneta Leveling Test Draw-Down Blade

6. Preparation of Sample

6.1 Adjust the temperature of the paint to $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) or to a temperature agreed upon between buyer and seller.

6.2 Thoroughly mix the paint by hand with a spatula to a smooth, uniform composition and consistency to ensure that the specimen to be taken for testing is representative.

7. Procedure

7.1 Affix the test chart to the draw-down plate. Position the leveling blade at the far end of the chart, between the parallel straightedges, with the longer arm of the blade adjacent to the left edge and pointing toward the operator.

7.2 Place a catch paper just below but in contact with the chart so that it is slightly to the right of the longer arm of the blade.

7.3 With the needle not attached, take up 8 to 10 mL of the material under test into the syringe (Note 1). Wipe off the

syringe orifice and attach the No. 15 gage needle. Eject within 3 to 5 s the entire amount in front of the blade, forming a puddle. Immediately lay the syringe down, grasp the blade arms with both hands, and draw the leveling blade rapidly but smoothly over the test paint at a rate of about 2 ft (0.6 m) per s, keeping the long arm on the left parallel to the surface during the draw-down.

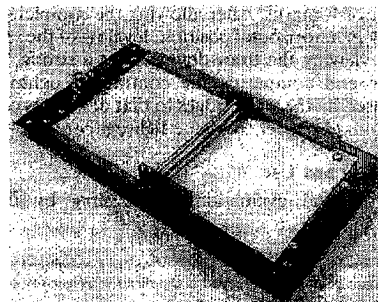
NOTE 1—Filling the syringe and cleaning the orifice are greatly facilitated by temporarily attaching to the orifice a 2-in. (50-mm) length of clear vinyl tubing with inside diameter of $\frac{1}{16}$ in. (3.2 mm) and outside diameter of $\frac{3}{16}$ in. (5 mm), which is removed prior to attaching the needle.

7.4 Allow the completed draw-down to dry overnight in a horizontal position, preferably at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, or under other conditions



NOTE—Plastic sidearms are for guidance to assure rectilinearity of blade movements.

FIG. 1 (b) Photograph of the Leneta Leveling Test Draw-Down Blade



NOTE—Illustration of use of draw-down plate and catch papers. Note that sidearms are attached to the test blade and parallel straightedge guides on draw-down plate.

FIG. 1 (c) Application With the Leneta Leveling Test Draw-Down Blade

agreed upon between the buyer and the seller.

7.5 Place the dry draw-down, with its ridges perpendicular to the direction of the light, in front of the oblique light as shown in Fig. 2. Place two leveling standards similarly oriented on either side of the test draw-down and view the center portion of the draw-down and the standards from above (that is at a 90° angle to the surface), or as close to 90° as possible if in a lighted room (see Fig. 2). Then successively interchange standards until one is found having the same distinctness of lightness and shadow as the test draw-down. Leveling poorer than Standard No. 1 is designated as 0 or very poor leveling. Leveling better than Standard No. 9 is designated as 10 which represents perfect leveling or no perceptible ridges. Estimate and record the number of the leveling standard that corresponds to the leveling of the test draw-down.

8. Report

8.1 Report the leveling of the test paint on the scale of 0 to 10 as determined by comparison with the numbered leveling standards.

9. Precision

9.1 On the basis of a study in which seven operators, each using a different blade, rated twelve different paints, the between-laboratories standard deviation was found to be 1.5 units on the 0 to 10 scale. Based on this standard deviation, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Reproducibility*—Two results, each one by operators in different laboratories, should be considered suspect if they differ by more than 1.5 scale units.

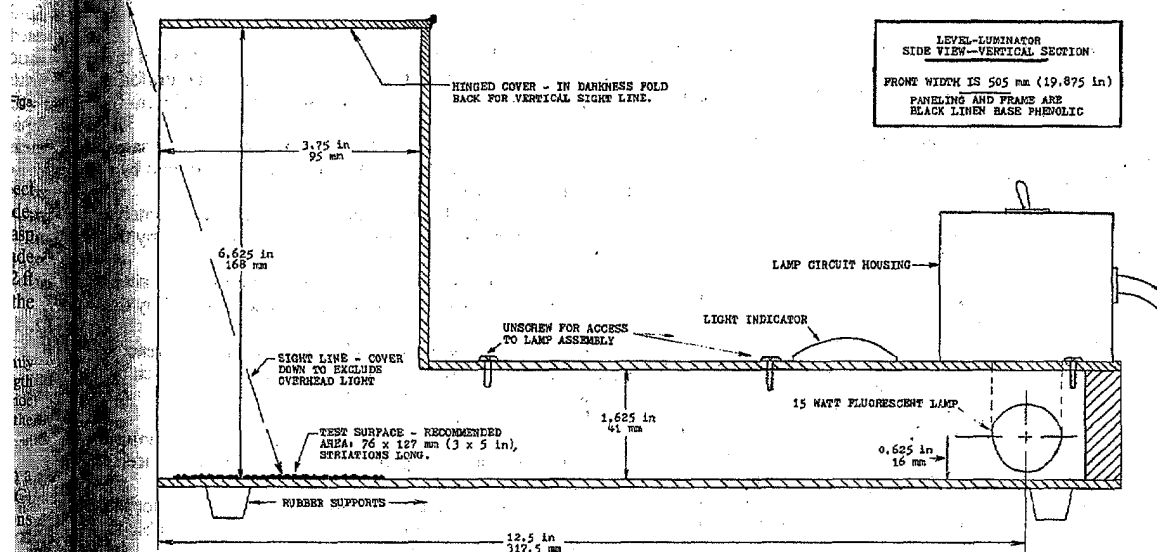


FIG. 2 Oblique Light Source (The Level Luminator) (Side View)

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Standard Test Method for Effects of Gamma Radiation on Coatings for Use in Light-Water Nuclear Power Plants¹

This standard is issued under the fixed designation D 4082; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers a standard procedure for evaluating the lifetime radiation tolerance of coatings to be used in nuclear power plants. This test method is applicable to Coating Service Levels I and II.

2. Referenced Documents

2.1 ASTM Standards:

D 659 Method of Evaluating Degree of Chalking of Exterior Paints²

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²

D 714 Test Method for Evaluating Degree of Blistering of Paints²

D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints²

2.2 ANSI Standard:³

N512 Protecting Coatings (Paints) for Nuclear Industry

3. Significance and Use

3.1 Variations can occur in surface preparation, application, and curing of coating materials. They may affect the performance of a coating system exposed to radiation when considered in conjunction with applicable engineered safety requirements. This test method is designed to provide a uniform test to assess the suitability of coatings, used in nuclear power facilities, under continuous radiation exposure for the projected 40-year lifetime of the facilities, including radiation during a DBA. Specific plant radiation exposure may exceed or be less than the amount specified in 6.2 of this standard. The gamma dose used may exceed the actual anticipated plant gamma dose in order to account for expected beta exposure as well. Coatings in Level II areas (outside primary containment) are expected to be exposed to lower accumulated radiation doses.

4. Preparation of Test Samples

4.1 *Steel Panels*—Panels shall be prepared in accordance with ANSI N512.

¹ This test method is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters. Current edition approved Feb. 24, 1989. Published April 1989. Originally published as D 4082 - 83. Last previous edition D 4082 - 83.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

4.2 *Concrete Blocks*—Blocks shall be prepared in accordance with ANSI N512.

5. Sampling

5.1 Prepare and test specimens at least in duplicate, or as otherwise specified by the owner.

6. Procedures

6.1 Irradiation Dose Rate:

6.1.1 Make the gamma energy field at the position of the test specimen 1×10^6 rads/h, or greater. It shall be uniform to within 10 % from one position of the specimen to another.

6.1.2 Make provisions so that all areas receive the same average exposure and dose, if the specimen is irradiated by a nonuniform source.

6.1.3 Determine the dose rate by a procedure acceptable to the coating manufacturer or as otherwise specified by the owner.

6.2 *Irradiation Accumulated Dose*—Make the total accumulated dose 1×10^9 rads, unless otherwise specified by the owner.

6.3 *Radiation Source*—Simulate conditions at a reactor site, closest to the preferred type of gamma source, such as, but not limited to, a fuel assembly.

6.4 Test Environment:

6.4.1 Specimens may be in air or in water during exposure to the gamma source, depending on the intended service as prescribed by the owner.

6.4.2 Do not exceed a temperature of 140°F (60°C) for the specimen during irradiation, or as otherwise acceptable to the coating manufacturer or as specified by the owner.

7. Examination and Reporting

7.1 Examine and evaluate specimens immediately after irradiation for the following coating defects:

7.1.1 *Chalking* (Method D 659)—Report extent.

7.1.2 *Checking* (Test Method D 660)—Report extent.

7.1.3 *Cracking* (Test Method D 661)—Report extent.

7.1.4 *Blistering* (Test Method D 714)—Report number and extent.

7.1.5 *Flaking* (Test Method D 772)—Report extent.

7.1.6 *Delamination*—Report extent.

7.1.7 *Peeling*—Report extent.

7.1.8 Report any observation of unusual appearance or deterioration.

8. Acceptance Criteria

8.1 *Checking, Cracking, Flaking, Delamination, Peeling, and Blistering*—None permitted.

Documentation

9.1 Document the following:

- 9.1.1 The procedures and conditions relating to the test specimen preparation.
- 9.1.2 The type of radiation source and the test procedure and environment.
- 9.1.3 Both the initial dose rate and the total accumulated dose.

10. Testing Laboratory

- 10.1 Testing shall be conducted by an independent laboratory that shall not be affiliated in any manner with the coatings manufacturer unless otherwise specified by the owner.

owner.

10.2 The testing laboratory shall be responsible for the documentation and certification of all test results.

10.3 The testing laboratory shall be responsible for meeting the quality assurance requirements of the owner.

11. Precision and Accuracy

11.1 The precision and accuracy of this test method is reflected in the precision and certified accuracy of the test instruments used.

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Standard Practice for Photographic Documentation of Coating and Lining Failures and Defects¹

This standard is issued under the fixed designation D 4121; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the requirements for photographs and photographic equipment to be used to document coating and lining failures or defects. Photographs may also be used to establish acceptance criteria.

1.2 The practice represents only one method to provide consistent, comparable photographs. The important factors to consider when using any type of photographic equipment are also described.

2. Significance and Use

2.1 The proper documentation, through photographic methods, in standard size and color is required for accurate comparison of a wide variety of coating and lining failures or defects. An integral part of the study of defects and failures is the ability to compare by a standardized reproducible method present and past appearances of defect characteristics and failure modes. The equipment should not be complicated or require any special skill to produce good definition and color.

3. Requirements for Photographs

3.1 Requirements are as follows:

3.1.1 Color slides in 2 by 2-in. (50 by 50-mm) cardboard mounts, suitable for projection in all standard 2 by 2-in. slide projectors,

3.1.2 Prints in a minimum 3 by 3-in. (76 by 76-mm) size. Unless a larger scale is needed, the standard 3½ by 3½-in. (89 by 89-mm) size should be used. Prints may be either color or black and white, although color usually provides the greatest information to those viewing the photographs, and

3.1.3 A scale of the area photographed.

4. Requirements for Standardized Photographic Equipment

4.1 The equipment should consist of the following:²

4.1.1 A 3 by 3-in. (76 by 76-mm) copy stand complete with compensating lense. The focal depth should be approximately ¾ in. (10 mm) above the subject plane to approximately ¼ in. (6 mm) below the subject plane.

4.1.2 An 8 by 8-in. (203 by 203-mm) copy stand complete with compensating lense. The focal depth should be approximately 1½ in. (38 mm) above the subject plane to approximately 1 in. (25 mm) below the subject plane.

4.1.3 A camera that will accept 12 or 20 exposures 126 size cartridge and have a lens system compatible with the copy stands in accordance with 4.1.1 and 4.1.2.

4.1.4 A flash cube or an electronic flash unit designed specifically for use with the camera-stand assembly. The electronic flash should have approximately a 40-s recovery time. The electronic flash should be battery-operated and self contained.

4.2 Equipment other than that described above may be used for photographic documentation; but, there may be varying scales and lighting effects.

5. Precautions and Limits

5.1 The following factors are the principal variables affecting photograph clarity over which the user has control:

5.1.1 *Degree of Enlargement*—Enlarging a given negative beyond a certain point yields no additional clarity to the photographs; more clarity must come from either a larger or clearer negative. Enlargement should be great enough so sufficient detail can be seen without the use of a magnifying glass.

5.1.2 *Negative Size*—The larger the negative, the more detail and clarity it can record. It is suggested that a minimum negative size of 26 by 26 mm be used. Negative sizes much larger than this might be needed rarely, if ever.

5.1.3 *Film Speed*—"Faster" films have progressively less resolution. In general, 35-mm ASA 200 film gives poor resolution if enlarged much beyond 4 by 5 in. when printing.

5.1.4 *Lighting*—Insufficient lighting gives poor resolution. Lighting of the wrong color such as incandescent bulbs using daylight film, distorts colors. Bright lights or flash perpendicular to a glossy or shiny surface causes glare. Also, perpendicular lighting may fail to show surface irregularities. Lighting at too oblique an angle may exaggerate the irregularities.

5.1.5 *Camera Stability*—A rigid support such as a copy stand or tripod will minimize camera movement.

5.1.6 *Camera Optics and Quality*—Professional quality cameras and equipment may be impractical due to cost, difficulty to operate, delicacy of equipment, or the need for an immediate print. Acceptable photographs can be obtained with some of the simpler, good-quality cameras.

6. Documentation

6.1 Each photograph shall be identified with the following information:

6.1.1 Date and time of photograph,

6.1.2 Where the photograph was taken and what it is intended to show, and

6.1.3 Name of the photographer.

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.03 on Quality Systems. Current edition approved March 26, 1982. Published May 1982.

² The Kodak Ektagraphic EF Visualmaker satisfies the requirements of this practice.

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Standard Test Method for Measurement of Dry Film Thickness of Protective Coating Systems by Destructive Means¹

This standard is issued under the fixed designation D 4138; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of dry film thickness of coating films by microscopical observation of precision-cut angular grooves in the coating film. Field use of this method may require repair of the coating film.

1.2 The substrate may be any rigid material, for example, metal, plastic, concrete, glass, etc. The surface may be plane or moderately curved (pipes as small as 1 in. (25 mm) in diameter may be measured in the axial direction).

1.3 This test method is not recommended for excessively elastomeric or excessively brittle films, although cooling or heating the surface can often modify the coating in the desired direction.

1.4 The range of thickness measurement is 0 to 50 mils (0 to 1.3 mm).

1.5 Measurements may be made on field structures, on manufactured products, or on test panels.

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²

3. Significance and Use

3.1 Measurement of dry film thickness of organic coatings by physically cutting through the film and optically observing and measuring the thickness offers the advantage of direct measurement as compared with nondestructive means.

3.2 The use of this test method is not necessarily limited by the type of substrate material as are nondestructive magnetic-type means.

3.3 Constituent coats of an overall thickness of a coating system can be measured individually by this method.

4. Apparatus

4.1 The measurement shall be performed by first incising a scribe mark of *known configuration* through the coating(s) film to substrate and then viewing the scribe mark with an illuminated microscope with measuring reticle. Scribe cutter and illuminated microscope may be combined as a single instrument (see Fig. 1).³ Instrument calibration shall be performed by taking measurements on applied films of known thickness. (See Test Methods D 1005.)

4.2 The configuration of the tungsten carbide cutting tips shall be designed to provide a very smooth incision in the paint film at a precise angle to the surface (see Fig. 2). Separate tip designs (angles) shall provide cuts of known slopes such as 1 to 1, 1 to 2, and 1 to 10. These tips shall be nominally designated 1 $\times$, 2 $\times$, and 10 $\times$ to indicate the ratio of the lateral measurement (microscope) to film thickness (cut depth in film). Metal guide studs on the gage body shall, together with the cutting tip, form a firm base to assure that the tip aligns vertically with the painted surface for a precisely aligned incision.

4.3 The illuminated, 50-power microscope shall contain a reticle scaled from 0 to 100 divisions (see Fig. 3). The total viewing field of the microscope shall be approximately 125 mils (3.18 mm).

5. Test Specimens

5.1 If multiple coats of paint are to be measured, successive contiguous coats should be of contrasting colors to aid sharp discrimination of interfaces.

5.2 Generally, test specimens shall be prepared (as test panels) or chosen (as sites on a structure) to be representative of localized coating thickness and variability.

5.3 For test panels, if *measurement* repeatability is desired for a particular paint system, painstaking care shall be taken in panel preparation. Steel panels shall be ground plane or rolled to 8.5- μ in. (215-nm) inches flatness. Coating shall be uniformly applied in accordance with Test Methods D 823. Panels shall be placed in a horizontal position during drying. Uniform application thickness shall be verified by another measurement method such as Test Methods D 1005.

6. Procedure

6.1 Select a test panel or choose a site for thickness measurement.

¹ This test method is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities.

Current edition approved Feb. 26, 1988. Published April 1988. Originally published as D 4138 - 82. Last previous edition D 4138 - 82.

² Annual Book of ASTM Standards, Vol 06.01.

³ A Tooke Gage, manufactured by Micrometrics, P.O. Box 13804, Atlanta, GA 30324, has been found suitable for this purpose.



FIG. 1 Tooke Inspector Gage—Model Mark II

6.2 Using an appropriate surface marker of contrasting color, mark a line on the surface about 2 in. long.

6.3 Select a cutting tip based on estimated film thickness as follows:

Tip	Thickness Range, mils (mm)	Conversion Factor
1×	20 to 50 (0.51 to 1.3)	1.0
2×	2 to 20 (0.05 to 0.51)	0.5
10×	0 to 3 (0 to 0.76)	0.1

If thickness is unknown, make a trial determination with 2×

6.4 To incise a groove, grasp the gage with the studs and cutting tip firmly forming a tripod on the painted surface. Place the gage at right angles to and about 2 in. (51 mm) perpendicular from a marked line.

6.5 Draw the gage across the paint film toward the body, with guide studs leading the cutting tip, and increase pressure on the cutting tip until it barely cuts into the substrate before it crosses the marked line.

6.6 Take readings at the intersection of the marked line and incision. Read by measuring on the reticle the distance from the substrate/coating demarcation up the longer machined slope of the incision to the upper cut edge of each respective coating or the coating system. Make sure that the machined slope of the incision is measured, not the other, steeper slope. (The machined upper edge usually leaves a less jagged cut than the other side.) If multiple coats are observed, individual thicknesses of each coat may be read. The actual coating thickness is derived by multiplying the reticle reading by the conversion factor for the respective cutting tip.

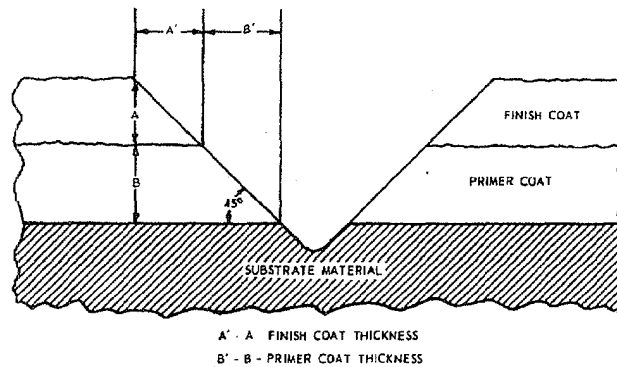


FIG. 2 Typical Incision Provided by Tungsten Carbide Cutting Tip of Tooke Inspector Gage

7. Report

7.1 Report the following information:

7.1.1 *Results of a Thickness Determination*—If more than one measurement is made and specific results for each location are not needed, report the minimum, the maximum, and the average thickness.

8. Precision

8.1 The replicability of individual observations of a uniform coating on a smooth substrate has been determined to be within $\pm 10\%$. (The percentage error increases as film thickness decreases.)

8.2 Field-applied coatings are characteristically subject to short-range thickness variability resulting from rough substrates and erratic application. The magnitude of this variability will be sensitively reflected by the range or standard deviation of thickness determinations.

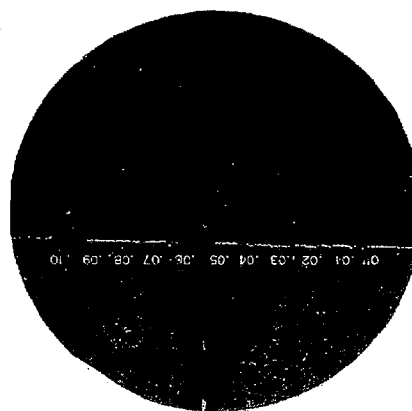


FIG. 3 Typical View through Microscope of Tooke Inspector Gage Showing Reticle

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Standard Practice for Conducting Accelerated Outdoor Exposure Tests of Coatings¹

This standard is issued under the fixed designation D 4141; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Footnote 3 was editorially changed in November 1987.

Scope

1.1 This practice covers three accelerated outdoor exposure procedures for evaluating the exterior durability of coatings applied to metal substrates.

1.2 The three procedures are as follows:

1.2.1 *Procedure A*—Black Box Exposure.

1.2.2 *Procedure B*—Heated Black Box Exposure.

1.2.3 *Procedure C*—Fresnel Reflector Rack Exposure.

1.3 The durability rankings of coatings provided by these three procedures may not agree when coatings differing widely in composition are compared.

1.4 The acceleration of degradation rates of coatings induced by the three procedures of exposure are discussed.

NOTE 1—Other exposure procedures may provide accelerated results on some types of products.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

ASTM Standards:

D 523 Test Method for Specular Gloss²

D 659 Test Method for Evaluating Degree of Chalking of Exterior Paints²

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²

D 714 Test Method for Evaluating Degree of Blistering of Paints²

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²

This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Coatings and Materials and is the direct responsibility of Subcommittee D-1.7 on Accelerated Tests for Protective Coatings.

Present edition approved June 25, 1982. Published August 1982.

Annual Book of ASTM Standards, Vol 06.01.

E 838 Practice for Performing Accelerated Outdoor Weathering Using Concentrated Natural Sunlight³

G 7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials⁴

3. Summary of Practice

3.1 Several procedures that will provide accelerated rates for the degradation of coatings are described. The procedures appear in the following order:

3.1.1 *Procedure A*—Exposure on a black box panel rack facing the equator at 5° from horizontal.

3.1.2 *Procedure B*—Exposure on a heated black box panel rack facing the equator at 5° from horizontal.

3.1.3 *Procedure C*—Exposure on a Fresnel reflector panel rack that provides a high intensity of sunlight irradiation by following the sun and focusing the sunlight on the test panels by means of mirrors. The panels are wet periodically by deionized water spray.⁵

3.2 Each of these procedures requires that coated test panels be placed on racks of specified design and be exposed under specified conditions of weathering.

3.3 The selection of a procedure for producing accelerated degradation is dependent on the intended end use of the coatings, limitations on the time available for the exposure tests, and the degree of reliability required for predictions of durability performance.

4. Significance and Use

4.1 The procedures described in this practice are designed to provide greater degradation rates of coatings than those provided by fixed angle outdoor exposure racks that optimize the annual incident solar radiation. For many products, fixed-angle exposures accelerate the normal end-use rate.

4.2 Following are comments regarding the relative rates of degradation that can be expected for coatings exposed by the procedures described in this practice.

4.2.1 *Procedure A (Black Box)*—For many coatings, this procedure provides greater rates of degradation than those provided by 5°, equator-facing, open-rack exposures because the black box produces higher panel temperatures during irradiation by the sun and longer time of wetness. The black box panel temperatures are comparable to those encountered

² Annual Book of ASTM Standards, Vols 12.02.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Fresnel reflector panel racks are located at DSET Laboratories, Inc., Box 1850, Black Canyon Stage 1, Phoenix, AZ 85029 and South Florida Testing Service Inc., Desert Site, 31818 N. 203rd Ave., Whittman, AZ 85361.

on the hoods, roofs, and deck lids of automobiles parked in direct sunlight.

4.2.2 *Procedure B (Heated Black Box)*—This procedure is most useful for exposures conducted in the late fall, winter, and early spring when it produces significantly higher panel temperatures than those produced by the 5°, equator facing, black box. Therefore, this procedure produces greater rates of degradation than those produced by Procedure A, particularly in the case of coatings with rates of degradation that are very temperature dependent.

NOTE 2—The relative rates of gloss loss and color change produced in some automotive and coil coatings by exposures in accordance with Procedures A and B are given in ASTM Special Technical Publication 781.⁶

4.2.3 *Procedure C (Fresnel Reflector Rack)*—This method provides greater rates of degradation of coatings than those provided by Procedure A or Procedure B. This high acceleration is produced by very high sunlight intensity and high panel temperature.

4.2.4 The degradation rates produced by any of the three procedures depend on the seasons of exposure, geographical location, and type of coating. The rates are higher in late spring, summer, and early fall when the intensity of sunlight irradiation is the greatest and when panel temperatures are higher. Also, the rates are highest for exposures in warm, humid environments.

4.2.5 Because outdoor weather conditions vary from season to season and year to year, these procedures are not reliable for establishing absolute performance ratings for coatings. The procedures should be used only for comparing the relative performance of coatings exposed at the same time at the same location.

5. Test Specimens

5.1 Each test specimen and control specimen shall consist of a uniform coating applied to the surface of a rigid panel. Suitable application procedures are given in Test Methods D 823.

5.2 Use surfaces of good planarity because warpage, waviness, or curvature may seriously affect the measurements of gloss and color and may produce a poor air seal on the black box rack.

5.3 Control specimens shall be prepared for inclusion in each exposure series to act as comparison standards and to provide a means for determining the severity of the exposure conditions encountered by the series. For best results, there should be at least two controls differing significantly in their durability performance.

5.4 Using Test Methods D 1186, measure the dry film thickness of the coatings at several different positions on the test and control specimens.

PROCEDURE A—BLACK BOX EXPOSURE

6. Apparatus

6.1 *Black Box Rack* of materials and construction as described in Practice G 7, or its equivalent.

⁶ Symposium on Performance of Organic Coatings, ASTM STP 781, ASTM, 1982.

7. Procedure

7.1 Position the rack so that the surfaces of the test specimens are 5° from the horizontal, facing the equator.

7.2 If change in gloss is to be measured, determine the specular gloss value for each unexposed specimen using a properly calibrated glossmeter in accordance with Test Method D 523.

7.3 If change in color is to be measured, determine the color coordinates for each unexposed specimen using Method D 2244. Unless otherwise agreed upon, use the CIE Lab Instrument Color Scale. The color-measuring instrument shall be stable and properly calibrated.

NOTE 3—As an alternative procedure, reserve unexposed duplicate specimen panels of each coating as reference for color difference measurements to be made on the exposed specimens. To minimize color drift, store these panels in a refrigerator.

7.4 Mount and fasten the specimens on the exposure rack. All empty spaces on the rack should be covered by dark gray or black panels so that the entire surface of the rack is covered.

NOTE 4—The predominant color of the specimen panels on the black box as well as the temperature of a standard panel should be noted. A panel on a black box will attain a lower temperature if all the other panels are white than if all the other samples are black.

7.5 Expose the test and control specimens for a specified period of time on the basis of one of the following:

7.5.1 Exposure for a specified number of days, months, or years with respect to an agreed upon starting date,

7.5.2 Exposure to a specified quantity of irradiation,

7.5.3 Exposure until a specified physical change has occurred in the test specimens, or

7.5.4 Exposure until a specified change has occurred in a control specimen exposed with the test specimens.

7.6 Remove the specimens from the exposure rack and gently wash a portion of the specimen surfaces to remove loose dirt. A suitable procedure consists of gentle rubbing with a sponge wet with water or a 0.05 % solution of a nonionic detergent. Water droplets should be gently removed from the specimen surfaces to prevent water spotting.

7.7 Unless otherwise agreed upon, perform the following on the washed portion of each washed specimen:

7.7.1 Measure the specular gloss by Test Method D 523.

7.7.2 Measure the color coordinates by Method D 2244, using the same color scales as used for measuring the unexposed specimen.

7.7.3 Checking rating by Test Method D 660.

7.7.4 Cracking rating by Test Method D 661.

7.7.5 Blistering rating by Test Method D 714.

7.8 Unless otherwise agreed upon, perform a chalk rating by Method D 659 on an unwashed area of the specimen.

PROCEDURE B—HEATED BLACK BOX EXPOSURE

8. Apparatus

8.1 *Black Box Rack* of materials and construction as described in Practice G 7, or its equivalent. The rack shall be equipped with a device that can heat the air in the interior of the box to the specified temperature.

8.2 *Safety Precautions*—The heated black box rack must be properly grounded to prevent possible electrical shock.

The heating elements in the heated black box must be installed so that, in normal use, a person cannot come in contact with a hot surface.

9. Procedure

9.1 Position the rack so that the surfaces of the test specimens are 5° from the horizontal, facing the equator.

9.2 If change in gloss is to be measured, determine the specular gloss value for each unexposed specimen using a properly calibrated gloss-meter in accordance with Test Method D 523.

9.3 If change in color is to be measured, use the procedure given in 7.3.

9.4 Mount and fasten the specimens on the exposure rack. All empty spaces on the rack shall be covered by dark gray or black panels so that the entire surface of the rack is covered.

9.5 Set the air temperature controller inside the heated black box to maintain an air temperature of approximately 140°F (60°C) or other agreed upon temperature.

9.6 The heater shall be turned on at approximately 9 a.m. each day and turned off at approximately 3 p.m. each day.

9.7 Expose the specimens for a specified period on the basis of one of the procedures outlined in 7.5.1 to 7.5.4.

9.8 Remove the specimens from the exposure rack and gently wash a portion of the specimen surfaces to remove loose dirt. Use the procedure recommended in 7.6.

9.9 Unless otherwise agreed upon, perform the following on the washed portion of each specimen:

9.9.1 Measure specular gloss by Test Method D 523.

9.9.2 Measure the color coordinates by Method D 2244, using the same color scales as used for measurements of the unexposed specimen.

9.9.3 Checking rating by Test Method D 660.

9.9.4 Cracking rating by Test Method D 661.

9.9.5 Blistering rating by Test Method D 714.

9.10 Unless otherwise agreed upon, perform chalk rating by Method D 659 on an unwashed portion of the specimen.

PROCEDURE C—FRESNEL REFLECTOR RACK EXPOSURE

10. Apparatus

10.1 *Fresnel Reflector Exposure Rack* that follows the sun, concentrates sunlight irradiation on the test specimens by means of mirrors, and sprays deionized water on the surfaces of the test specimens at specified intervals. The rack shall have provisions for cooling the test specimens while they are irradiated. Refer to Practice E 838 for detailed descriptions of the apparatus.

11. Procedure

11.1 If change in gloss is to be measured, determine the specular gloss value for each unexposed specimen. The gloss-meter used shall be properly calibrated.

11.2 If change in color is to be measured, use the procedure given in 7.3.

11.3 Mount and fasten the specimens on the exposure rack.

11.4 Set the water spray control to provide a deionized water spray on the specimens during the night at a frequency of 4 per hour, each spray period to be of 3-min duration. Refer to Procedure B of Practice E 838 for detailed operating procedures.

11.5 Expose the specimens for a specified period on the basis of one of the procedures outlined in 7.5.1 to 7.5.4.

11.6 Remove the specimens from the exposure rack and gently wash a portion of the specimen surfaces to remove loose dirt. Use the procedure recommended in 7.6.

11.7 Unless otherwise agreed upon, perform the following on the washed portion of each specimen:

11.7.1 Measure specular gloss by Test Method D 523.

11.7.2 Measure the color coordinates by Method D 2244, using the same color scales as used for measurements of the unexposed specimen.

11.7.3 Checking rating by Test Method D 660.

11.7.4 Cracking rating by Test Method D 661.

11.7.5 Blistering rating by Test Method D 714.

11.8 Unless otherwise agreed upon, perform a chalk rating by Method D 659 on an unwashed portion of the specimen.

12. Evaluation of Results

12.1 Express the change in gloss of each specimen either in terms of units of gloss loss or in percent gloss loss relative to the initial gloss value.

12.2 Express the change in color of each specimen in terms of total color difference, ΔE , using one of the procedures given in Method D 2244.

12.3 Express the amounts of chalking, checking and cracking on a scale of 10 to 0 as outlined in Method D 659, Test Method D 660 and Test Method D 661, respectively.

13. Report

13.1 Report the following information:

13.1.1 The method of exposure used and its geographical location.

13.1.2 The duration of the exposure and the date of the beginning of the test.

13.1.3 The heated black box air temperature used (for Procedure B).

13.1.4 The type of control specimens used and the severity of their degradation.

13.1.5 The evaluation measurements performed on each of the exposed specimens:

13.1.5.1 Units of gloss loss or percent gloss loss.

13.1.5.2 Units of ΔE color change. Denote the color space and color scales used.

13.1.5.3 Ratings for chalking, checking, cracking, and blistering.

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Standard Method for Estimating Package Stability of Coatings for Ultraviolet Curing¹

This standard is issued under the fixed designation D 4144; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This method covers procedures for testing the package stability of coatings intended to be cured by ultraviolet radiation. One procedure is given for clear coatings and another for opaque fillers.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Summary of Method

2.1 Specimens are placed in several containers, some of which are subjected to an elevated temperature while others are stored at room temperature. At specified intervals a specimen is checked for evidence of gelling. Clear materials are held in glass containers so they can be examined visually without opening to prevent contact with air which might inhibit polymerization. Opaque materials are checked by opening one can, probing the contents with a spatula to determine the extent of any polymerization, and then recording that specimen.

Significance and Use

3.1 Coatings intended to be cured by ultraviolet radiation, especially those involving free radical chemistry, tend to polymerize during storage. It is of interest to determine how well a formulation resists this effect. Many factors influence the storage stability of a composition. The procedures described here are intended to improve the precision of determining this property. Because the effects of resins, monomers, photoinitiators, synergists, stabilizers, or pigments can alter the relation between elevated and room temperature stabilities, any correlation of performance at different temperatures is possible only with a given formulation and, therefore, is useful only for quality control.

Apparatus

- 4.1 *Oven*, maintained at $60 \pm 2^\circ\text{C}$.
- 4.2 *Glass Jars*, wide-mouth, 4-oz (115-mL), with 38-mm flanges.
- 4.3 *Cans*, lined, 4-oz (115-mL), friction top, with lids.

- 4.4 *Spheres*, glass or porcelain, 7 to 10-mm diameter.

5. Procedure

5.1 Clear Coatings:

5.1.1 Fill three 4-oz (115-mL) wide-mouth jars to $\frac{1}{4}$ in. (6 mm) from the top. Add a small glass or porcelain sphere to each container and put the lids on tightly.

5.1.1.1 The amount of head space in a jar or can is critical because the volume of air in contact with the sample has an effect on the rate of polymerization. The stability is also related to the ratio of the area of liquid-air interface to the volume of liquid.

5.1.2 Put two jars in an oven at $60 \pm 2^\circ\text{C}$. Retain the third at ambient temperature, $25 \pm 2^\circ\text{C}$, and in the dark.

5.1.3 Check an oven jar daily but do not open or invert. Rather, tip slightly, no more than 30° , to determine the extent of polymerization by noting the mobility of the sphere. When gelling is noticed, check the second jar to confirm.

5.1.4 Record the duration of the test in days. Indicate the last day the sphere is mobile followed by the first day it is immobile, and if the days are not consecutive, why the interval occurred.

5.1.5 Check the jar stored at room temperature every week but do not open or invert. Tip slightly, no more than 30° , to determine if the sphere is immobile.

5.1.6 Record the number of weeks not gelled followed by the first week the sphere is immobile.

5.2 Pigmented (Opaque) Coatings:

5.2.1 Fill twelve 4-oz (115-mL) lined cans to $\frac{1}{4}$ in. (6 mm) from the top and put the lids on tightly.

5.2.1.1 See 5.1.1.1.

5.2.2 Put six of the cans in an oven at $60 \pm 2^\circ\text{C}$. Retain six cans at ambient temperature, $25 \pm 2^\circ\text{C}$.

5.2.3 After one day remove one can from the oven, open, and probe to the bottom to determine if gelling is beginning. Discard the can after the test. Check one of the remaining cans on the 2nd, 4th, 8th, 16th, and 32nd days and discard after testing. By starting on a Monday all the testing will fall on normal working days.

5.2.4 Record the condition of the specimen each day tested, indicating the fractional amount of any gelled material present.

5.2.5 After one week check one of the cans held at ambient temperature by opening and probing to the bottom to determine if gelling is beginning. Discard the can after the test. Check one of the remaining cans after 2, 4, 8, 16, and 32 weeks.

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D-1.2 on Factory-Coated Wood Products.
Current edition approved June 25, 1982. Published September 1982.

5.2.6 Record the condition of the specimen after each test and indicate the fractional amount of any gelled material present.

6. Report

6.1 Report whether the material was clear or pigmented and the length of time it was stable as indicated by the occurrence of gelation at both ambient and elevated temperatures. Report the age of the material when the test began, if it is known.

7. Precision

7.1 Clear Coatings:

7.1.1 At the elevated temperature, four of five cooperators reported a clear coating without inhibitor gelled on the 8th or 9th day. Four of five cooperators reported a coating with inhibitor gelled on the 32nd day; the fifth reported gelation on the 18th day.

7.1.2 At room temperature four of four cooperators reported no gelling at 32 weeks, with or without inhibitor.

7.2 Pigmented Coatings:

7.2.1 At the elevated temperature six of six cooperators reported a filler gelled at 8 days.

7.2.2 At room temperature four of four cooperators reported various degrees of gelation after 32 weeks.

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Standard Test Method for Coating Flexibility of Prepainted Sheet¹

This standard is issued under the fixed designation D 4145; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Section 2 was deleted and editorial changes were made throughout in May 1990.

1. Scope

1.1 This test method describes a procedure for determining the flexibility and adhesion of organic coatings (paints) on metallic substrates that are deformed by bending when the sheet is fabricated into building panels or other products.

1.2 The metal substrate must be capable of passing this test without fracturing and with no excessive grain development.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 *Description of Term Specific to This Standard:*

2.1.1 *T-bend*—a description of the severity of a bend in terms of the thickness (T) of the sheet to which the coating has been applied; *as used in this test method*, the T-bend rating is the minimum number of thicknesses of metal around which the coated sheet is bent (Fig. 1), or if bent around a die, the number of thicknesses of metal equivalent to the diameter of the die to achieve no fracture or removal of the coating.

3. Summary of Test Method

3.1 Prepainted panels are bent 180° around progressively more thicknesses of metal or larger diameter dies, the end point being when failures no longer occur. The panels are examined at low magnification (5 to 10 $\times$) after each bend for fracture of the coating (cracking) and, for loss of adhesion (pickoff), by means of a tape pull-off test.

4. Significance and Use

4.1 Organic coatings on precoated sheet are subjected to stresses when fabricated into products by roll forming, brake bending, or other deformation processes. These stresses can exceed the flexibility or adhesive strength of the coating, resulting in fracture of the coating, exposing the substrate, or loss of adhesion to the substrate. This test is a means of evaluating the ability of a coating system to withstand the stresses of fabrication.

4.2 The bend direction, whether the axis of bend is across or along the rolling diameter of the metal, and the temperature of the specimen when it is bent affect the results of this test and should be agreed upon between the supplier and the user.

5. Apparatus

5.1 A means of holding one end of the specimen rigidly while making the bend is needed. A bench vise with smooth jaws or with smooth inserts, preferably of a soft metal to avoid damage to the coating, has been found to be satisfactory.

5.2 If the metal is not bent around itself as in Fig. 1, a series of bending dies with smoothly rounded ends may be used as guides around which the specimen is bent (Fig. 2).

5.3 *Magnifier*, capable of 5 to 10 $\times$ magnification for examining the specimen for coating fracture and pickoff after testing. A stereomicroscope has been found useful for this purpose.

6. Procedure

6.1 Bending the Coated Specimen Around Itself.

6.1.1 The coated specimen shall be at least 2 in. (50 mm) across the bend direction, by a length sufficient to make the required number of bends. A 2 by 6-in. (50 by 150-mm) size is convenient. The specimen shall be at $\pm 2^\circ\text{F}$ of the temperature agreed on.

NOTE—A triangle-shaped specimen (Fig. 3) has been found convenient for making T-bend tests. This shape leaves a portion of each bend exposed for later examination and for a permanent record.

6.1.2 Secure about 0.5 to 0.75 in. (13 to 20 mm) of one end of the specimen in a vise or holding jig as shown in Fig. 1. Bend the free end of the specimen 90° in a smooth and uniform manner so that the coating is on the outside of the specimen after it is bent.

6.1.3 Continue to bend the specimen until the bent end can be inserted in the jaws of the vise. Tighten the vise to complete the 180° bend, which is called an OT bend. Take care to tighten the vise sufficiently so that the apex of the bend is as flat as can be reasonably achieved.

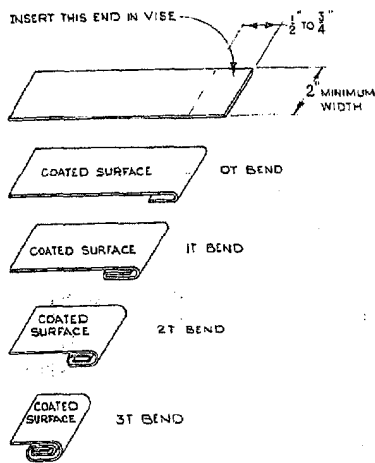
6.1.4 Examine the bend using a hand lens or low-power microscope for cracks in the coating.

6.1.5 Apply pressure-sensitive tape² along the bend. Rub the tape flat; then, holding the specimen firmly, remove the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.53 on Factory-Precoated Strip Metal.

Current edition approved March 25, 1983. Published July 1983.

² Scotch Brand #610 tape manufactured by 3M Co., St. Paul, MN or equivalent, or as agreed upon between the supplier and the user.



NOTE—The above bends are expanded for clarity. They are actually flat against themselves in the test.

FIG. 1 T-Bend Test in Which the Coated Specimen is Bent Around Itself

tape with a rapid movement at an angle of 180° to the bend surface. Examine the tape for coating removed from the surface of the specimen (that is, pickoff). In the case of a coated steel specimen (for example, galvanized steel), examine the tape to determine whether pickoff is between the organic coating and the metallic surface or between the metallic coating and the base steel.

6.1.6 Secure the bent end of the specimen in the vise and bend the free end 90° . Continue to bend the free end around the first (OT) bend to complete a 180° bend, and tighten in the vise as before. This makes a 1T bend (Fig. 1). Examine under magnification and by tapping for cracking and pickoff, as with the OT bend.

6.1.7 Repeat this procedure, making a 2T, 3T, etc., bends until no cracking or pickoff occurs.

6.2 Bending the Coated Specimen Around a Die:

6.2.1 The coated specimen shall be at least 2 in. (50 mm) across the bend direction.

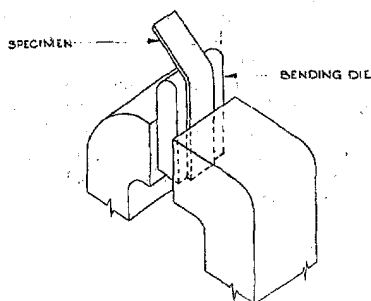
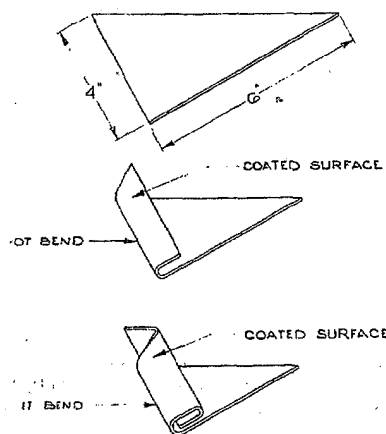


FIG. 2 T-Bend Test Using a Die Around Which the Specimen is Bent



NOTE—The above bends are expanded for clarity. They are actually flat against themselves in the test.

FIG. 3 Triangular Specimen for T-Bend Test

6.2.2 Secure the specimen and bending die in a vise or holding jig (Fig. 2). Bend the specimen 180° around the die in a smooth and uniform manner.

6.2.3 Examine the bend for cracking as in 6.1.4 and for loss of adhesion as in 6.1.5.

6.2.4 Express the T-bend to no cracking or no pickoff as the number of thickness around which the metal is being bent equal to the thickness of the die.

7. Report

7.1 Report the following information:

7.1.1 Identification of the material, such as coil number and location within the coil if the sample was coated on a coil line.

7.1.2 The coating system, coating thickness, and application and cure conditions.

7.1.3 The temperature at which the specimen is bent.

7.1.4 The bend direction—whether across or along the rolling direction of the sheet.

7.1.5 The minimum T-bend to no paint fracture, and

7.1.6 The minimum T-bend to no pickoff.

8. Precision³

8.1 On the basis of an interlaboratory study of this test method, in which two operators in each of five laboratories tested panels coated with paints of different flexibility and adhesion, the standard deviation was found to be:

	Standard Deviation	
	Aluminum	Galvanized Steel
T-bend to no fracture	0.55	1.33
T-bend to no pickoff	0.76	2.09

8.2 Based on these standard deviations, the following

³ Supporting data are available from ASTM Headquarters. Request RR:D01-1034.

Criteria should be used to judge the acceptability of results at the 95% confidence level:

8.2.1 *Repeatability*—Measurements obtained by two operators in a single laboratory should be considered suspect if they differ by more than:

	Aluminum	Galvanized Steel
T-bend to no fracture	0.4	1.8
T-bend to no pickoff	0.9	3.3

8.2.2 *Reproducibility*—Two measurements obtained in different laboratories, each the mean of measurements made by two operators within a laboratory, should be considered suspect if they differ by more than:

	Aluminum	Galvanized Steel
T-bend to no fracture	2.5	10.1
T-bend to no pickoff	1.8	10.6

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Standard Test Method for Formability of Zinc-Rich Primer/Chromate Complex Coatings on Steel¹

This standard is issued under the fixed designation D 4146; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Editorial changes were made throughout in March 1989.

1. Scope

1.1 This test method covers the evaluation of the formability and adhesion of factory applied zinc-rich primer/chromate complex coatings on steel with thicknesses of 0.025 to 0.05 in. (0.65 to 1.25 mm) typical of those used in the coil coating industry.

1.2 The degree of oil removal prior to forming, the techniques of taping, and differences in adhesive strength of the tape can affect the adhesion rating.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

D 3330 Test Method for Peel Adhesion of Pressure-Sensitive Tape at 180-Deg Angle²

2.2 Other Document:

Pictorial Standards of Coating Defects³

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *dome height*—the height of the dome formed in the test.

3.1.2 *indenting ball diameter*—the diameter of the spherical-ended penetrator (ball) used to deform the specimen.

3.1.2.1 *Discussion*—The 1½-in. (41-mm) diameter ball was selected because it minimizes the variability in the localized peak strain. This size ball is commercially available and is as large as most testing machines can accommodate.

3.1.3 *percent strain*—the percent elongation of scribed gage lengths after forming. For the draw height of 0.512 in.

(13 mm), using a 1½-in. diameter ball, the localized peak strain is about 19 % (see Fig. 1).

4. Summary of Test Method

4.1 A coated specimen is biaxially stretched a given distance in an appropriate machine, adhesive tape is applied to the deformed area (dome) and then pulled off, and the amount of coating removed is compared with a photographic standard to determine the coating adhesion rating.

5. Significance and Use

5.1 The results of the combined deformation and tape test are related to the ability of the coated metal to withstand stamping in factory applications.

5.2 This test can be used to control the manufacturing process or in development work to improve the product.

5.3 It should be recognized that variability in the results persist due to the test conditions and forming machine variations.

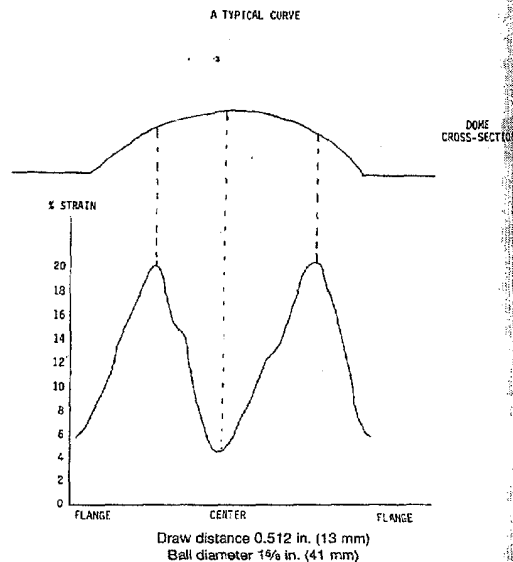


FIG. 1 Survey of Strain Over Biaxially Stretched Dome

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.53 on Coil Coated Metal.

Current edition approved Oct. 28, 1983. Published January 1984.

² Annual Book of ASTM Standards, Vol 15.09.

³ Copies of the pictorial photographic reference standard are contained in the publication *Pictorial Standards of Coating Defects* and may be obtained from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422. The silver halide gelatin photographs are intended to be the only primary reference standards for this test method. The reproductions of them in this test method are for the purpose of illustration only.

Apparatus and Materials

6.1 **Forming Machine**, equipped with a spherical-ended penetrator to deform the specimen with appropriate dies, until the required height is obtained, and clamping arrangement to hold the specimen with a minimum pressure of 2000 lbf (8.9 kN).

6.1.1 The speed of forming shall be between 0.2 and 1.0 in./min (4.8 and 25 mm/min). The speed is usually slowed to measure more accurately the final height as shown on the dial indicator, but the final punch speed shall be controlled at 0.24 in. (6 mm)/min maximum.

6.2 **Indenting Ball**, with a diameter of 1½ in. (41 mm), a Rockwell hardness number not less than 61 on the Rockwell C scale (HRC), and a smooth surface finish not exceeding 60 µin. (4 µm). The indenting ball shall not deform during the test. The ball and holder shall move through the centerline of the top and bottom dies. The ball shall be clean and free of oil, grease, oxide buildup, rust, dirt, nicks, or damage.

6.3 **Ball Holder**, to hold the ball in its seat during the test. Most ball holders are removable so that other ball punch information tests can be done in the same machine.

6.4 **Upper and Lower Dies** with a hardness of 56 HRC or higher. The upper die shall have an approach radius of 0.0625 ± 0.002 in. (1.6 ± 0.05 mm). The diameter of the opening in the upper and lower die shall be 1.64 in. (42 mm). In most machines, these dies are removable.

6.5 **Dial Indicator**, riding on the surface of the forming sheet to monitor the movement of the penetrator (ball) and graduated so that the displacement, or ball height, can be measured at least to ± 0.0025 in. (60 µm) but preferably to ± 0.001 in. (25 µm).

6.6 **Adhesive Tape**, 1 in. (25 mm) wide, transparent, high-tack, pressure-sensitive tape⁴ with an adhesive strength of steel of 45 oz/in. (50 g/mm) of width when tested in accordance with Test Method D 3330, and a tensile strength of 28 lb/in. (500 g/mm) of width.

6.7 **Photographic Standard**⁵—Steps in rating from 10 (good, no removal) to 0 (poor, complete removal) to rate the adhesion of the coating after forming and tape removal by comparison with the adhesive-backed tape.

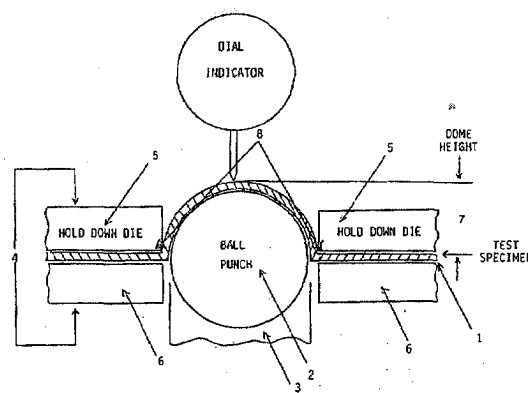
Hazards

7.1 There are possible hazards to personnel when performing tests on any hydraulic equipment. Stand free when machine is operating.

Sampling and Test Specimens

8.1 **Sampling**—The coated steel coil is cut, usually at the head or tail of the coil, into a sheet sample, which can be easily handled.

8.2 **Specimen Size**—The specimens may be square or rectangular but not narrower than 3.5 in. (90 mm) in either



Key	Dimensions	
	Inch-Pound	Metric
1 Test specimen		
2 Spherical-ended penetrator (ball)	1½ in.	41 mm
3 Ball holder		
4 Clamp force (minimum)	2000 lbf	8.9 kN
5 Upper die; bore diameter	1.64 in.	42 mm
6 Lower die; bore diameter	1.64 in.	42 mm
7 Dome height	0.512 in.	13 mm
8 Upper die corner radius	0.0625 in.	1.6 mm

FIG. 2 Tooling for Forming Test

dimension. Strips of full width of the coil can be tested if precautions are taken not to nick or damage the adjacent dome in the machine.

8.3 **Specimen Thickness**—Thicknesses should be 0.025 to 0.05 in. (0.64 to 1.25 mm). With thicknesses less than 0.025 in., fracture of the steel may occur, in which case the dome height can be reduced if agreed upon between the supplier and the user of the sheet product. When thicknesses greater than 0.05 in. are being tested, die modifications may be needed, but such modifications require agreement between the supplier and the user.

8.4 Test specimens shall be free of oil, grease, lubricants, rust, and burrs that may affect the holding pressures or dome height.

8.4.1 Typically a light spindle oil is applied to reduce corrosion on the uncoated side of the coil.

8.4.2 Remove oil, grease, and lubricants by one of two methods, as agreed upon between the supplier and the user. Either heat the specimens to 300°F (150°C) for 30 min or wash them in a cleaning solution⁶, rinse with tap water, and dry. As the solution can become contaminated with oil, do not wash more than 50 panels per gallon of solution.

9. Procedure

9.1 Condition the specimens for at least 24 h at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity before conducting the test, unless otherwise agreed.

⁴ Scotch Brand Transparent Tape No. 610, available from 3M Co., Commercial Tape Division, 3M Center, St. Paul, MN 55101, has been found satisfactory for this purpose. Equivalent material may be used.

⁵ The photographic reference standards that are part of this test method and are provided in the *Pictorial Standards of Coating Defects Handbook*³ are representative of the adhesion ratings. The photograph shown in Fig. 3 is for illustration purposes only and should not be used for evaluation.

⁶ This is one cleaning solution that is used. Add 10 g of Car Wash Concentrate, a product of Borden Inc., Chemical Div., 180T E. Broad St., Columbus, OH 43215 (product formerly called DuPont No. 7 Car Wash), per gallon (2.6 g/L) of water at a temperature of 100°F (40°C).

9.2 Place the specimen in the machine so that the coating to be tested will be on the convex side of the dome away from the punch.

9.3 With the ball touching the uncoated side of the specimen and the dial gage indicating zero penetration at the start of the test, indent the specimen with the ball to a distance of 0.512 in. (13 mm), ensuring that the hold-down pressure does not decrease during forming of the dome and that there is no evidence of slippage of the specimen under the hold-down dies during the test. The speed of forming the domes is done in accordance with 6.1.1.

9.4 Return the ball to the original position, remove the specimen from the machine, and lay it on a flat surface.

9.5 Apply the tape over the peak of the dome, press firmly, and then pull the tape from the dome at a fast, steady rate. The 1-in. (25-mm) wide tape will not fully cover the dome and will wrinkle at the edges.

9.6 Place the tape on a piece of white paper, compare the amount of coating on the tape with the photographic standard, and estimate coating adhesion to the nearest whole number. In Fig. 3, areas of darkness are coating removal so that a tape rating of No. 10 is perfect adhesion and No. 0 is complete removal of the coating. Wrinkles in the tape can be seen in the photographic standards as light areas.

9.7 Typically, a full-width specimen is cut from each coated coil and tested at three areas along the strip. Usually, domes are made at both edges and the center of the coated

full width strip.

10. Report

10.1 Report the following information:

10.1.1 Identification of the material such as coil number and location of the specimen within the coil,

10.1.2 Specimen thickness,

10.1.3 Number of tests or domes evaluated, and

10.1.4 Adhesion rating including mean, range, or standard deviation, if applicable.

11. Precision

11.1 On the basis of an interlaboratory study of this test method, in which one operator in each of three laboratories tested five separate panels cut from the same coated steel, the within-laboratory standard deviation was 0.61 units with 12 df and the between-laboratory standard deviation was 0.35 with 2 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 2 rating numbers.

11.1.2 *Reproducibility*—Two results, each the mean of two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 2.9 rating numbers.



FIG. 3 Photographic Standard Adhesion Rating on Tape After Deformation

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Standard Practice for Applying Coil Coatings Using The Wire-Wound Drawdown Bar¹

This standard is issued under the fixed designation D 4147; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure for applying a coating film of uniform thickness on a flat panel using the wire-wound drawdown bar.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Practice

2.1 A flat panel is secured on a firm horizontal surface. An ample amount of coating is poured across one end of the panel and the drawdown bar placed behind the coating. The bar is then drawn uniformly along the length of the panel toward the operator to apply a uniform film. The dry film thickness obtained is dependent on the combination of the bar used, the volume solids of the coating, and the speed of the drawdown motion.

3. Significance and Use

3.1 This practice is useful in the laboratory to simulate roll-coated films.

4. Apparatus

4.1 *Wire-Wound Drawdown Bars*²—A set of calibrated stainless steel wire-wound drawdown bars at least 1/2 in. (13 mm) in diameter to prevent bowing during application. These bars are calibrated such that the diameter of the wire wound on the bar controls thickness of the wet film applied. The choice of specific drawdown bar is dependent on the dry film thickness required and the volume solids of the coating under test.

4.2 Devices for Securing Panels:

4.2.1 *Magnetic Chuck*³ for securing steel panels to eliminate bowing.

4.2.2 *Vacuum Plate*⁴ for securing very lightweight nonmagnetic panels.

4.2.3 *Tape* for securing the top of thin-gage panels.

5. Safety Precautions

5.1 The flash points of most solvents used in coatings and related products are low enough that adequate ventilation is needed to avoid exceeding 25 % of the lower explosive limits of the solvent when preparing and baking the panels. As these solvents could be toxic, care should be taken to avoid inhalation of the vapors and unnecessary contact of the solvents with the skin.

6. Procedure

6.1 Deburr panels so edges are smooth. Secure a flat panel to be coated on a firm, uniform, level surface. To provide sufficient testing surface pour an ample amount of coating across the end of the panel. Place the drawdown bar behind the coating and pull into the coating. Rotate the drawdown bar 30 to 60° in the coating to ensure complete wetting of the coating in the threads of the bar. Apply moderate pressure equally to both ends of the drawdown bar and hold it firmly without allowing rotation. Using only one pass, draw the bar along the length of and off the panel at a uniform speed so that a continuous film is produced. The drawdown speed is dependent on the rheology and type of the coating being used. Remove the coated panel and bake, force dry, or air dry the coating in accordance with its type so its dry film thickness can be measured. A heavier film thickness is obtained along the edges of the panel and possibly at the top of the panel where the coating was poured. For a two-coat system, the side edges must be trimmed to eliminate the heavier areas on the first coat before applying the second coat.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.53 on Factory-Precoated Strip Metal.

Current edition approved June 25, 1982. Published September 1982.

² Wire-wound drawdown bars may be obtained from R.D. Specialties, P.O. Box 206, Webster, N.Y. 14580, Paul N. Gardner Co., Inc., 218 Commercial Blvd., Lauderdale-by-the-Sea, Fla. 33308, or the Leneta Co., P.O. Box 86, Ho-Ho-Kus, N.J. 07423, or equivalent may be used.

³ Magnetic chucks may be obtained from Brown & Sharpe Manufacturing Co., Precision Park, North Kingstown, R.I. 02852, or equivalent may be used.

⁴ Vacuum plate may be obtained from Paul N. Gardner Co., Inc., 218 Commercial Blvd., Lauderdale-by-the-Sea, Fla. 33308, or equivalent may be used.

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Standard Test Method for Sustained Burning of Liquid Mixtures by the Setaflash Apparatus (Open Cup)¹

This standard is issued under the fixed designation D 4206; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

This test method may be used in conjunction with a flash point determination. If the flash point of a mixture of flammable and nonflammable liquids or liquids of widely different flash points is below the upper limit of a flammability classification (for example, 100°F specified by the U.S. Department of Transportation), this test may be conducted to determine the sustained burning characteristics of the mixture.

This test method is a modification of the test for combustibility now incorporated as Schedule 2 of the "Highly Flammable Liquids and Liquified Petroleum Gases Regulation, 1972" of the United Kingdom under The Factories Act, 1961, which is also issued as British Standard BS-3900, Part A-11, Small Scale Test for Combustibility. This sustained burning test was studied and proposed by the ASTM Coordinating Committee for Flash Point and Related Properties. The major purpose of this test is similar to that of the British test—to provide a method for determining the sustained burning characteristics by directly observing this property rather than by deducing them from the flash point.

Scope

1.1 This test method² describes a procedure for determining the sustained burning characteristics of mixtures of flammable and nonflammable liquids and also mixtures containing liquids with widely different flash points.

1.2 This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Referenced Documents

2.1 British Standards:³

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.22 on Health and Safety.

Current edition approved April 28, 1989. Published June 1989. Originally published as D 4206 - 82. Last previous edition D 4206 - 82⁴.

² McKelvie, A. N., "A Test for Ability to Support Combustion for Liquids Including Paints and Allied Products," *Journal of Oil Co. Chemical Assoc.*, 1972, Vol. 55, pp. 1086-1093.

³ Available from British Standards Institute, 2 Park St., London, England W1A 2BS.

BS-3900, Part A-11, Small Scale Test for Combustibility

3. Summary of Test Method

3.1 A block of aluminum alloy, or other nonrusting metal of suitable heat conductivity, with a concave depression (called the well) is heated to the required temperature of 120°F (49°C). A standard source of flame, capable of being swung over the center of the well and at a given distance from it, is attached to the metal block.

3.2 Two millilitres of the product under test are transferred to the well. After the product has reached the stated temperature, the flame is passed over the well, held there for a specified time, and then removed. The time of sustained burning is then noted.

4. Significance and Use

4.1 Mixtures of flammable liquids and nonflammable liquids, such as an alcohol and water mixture, are classified by the U. S. Government by the definition of flammable liquid based on a closed-cup flash point method. Thus, mixtures may be classed as flammable even though they do not sustain burning. This test method determines the ability of a liquid mixture to sustain burning and, when used with a closed-cup flash point method, indicates the flammability characteristics of the mixture.

5. Apparatus

5.1 *Setaflash Open-Cup Tester*,⁴ or an equivalent tester consisting of a 316 stainless steel block or other nonrusting

⁴ Suitable apparatus is available from Erdco Engineering Corp., 721 Custer Ave., Evanston, IL 60204 or Stanhope-Seta Limited, Park Close, Englefield Egham, Surrey TW20 0XD, England.

block of suitable heat conductivity fitted with a concave depression or well (Note). The metal block has a thermometer embedded in it. A small gas jet on a swivel is attached to the metal block. The exact dimensions of the metal block and its well, the gas jet and its positioning, and the embedded thermometer are shown in Fig. 1 and are specified in Annex A1.

NOTE—While the Setaflash Open Cup Tester is generally made of an aluminum alloy, continued use of it for sustained burning tests may produce a carbon deposit in the well which may be difficult to remove without damaging the aluminum well. The stainless steel well may be cleaned with crocus cloth.

5.1.1 *Gage*—A metal strip, capable of checking the height of the gas jet above the top of the well lip (2.2 ± 0.1 mm).

5.1.2 *Hot Plate*, fitted with a temperature-controlling device or other means of heating the metal block.

5.1.3 *Stopwatch*, or other suitable timing device.⁴

5.2 *Thermometer*,⁴ 32 to 230°F (−5 to 110°C), traceable to the National Institute of Standards and Technology.⁴

5.3 *Graduated Pipet or Hypodermic Syringe*,² capable of delivering 2 ± 0.1 mL.

6. Preparation of Sample

6.1 Obtain a representative sample of the product under test, mixing it thoroughly, especially if it has a tendency to layer, and keep in an airtight container.

6.2 Because of the possibility of loss of volatile constituents, the sample should receive only the minimum treatment

to assure uniformity. After removing the specimen, immediately close the sample container tightly to assure that no volatile flammable components escape from the container. (Otherwise a new sample may be necessary if further testing is required.)

6.3 Do not open containers unnecessarily. Do not make transfers unless the sample temperature is at 100°F (37.8°C) or below, except for samples that are too viscous to be handled at that temperature. In these cases, transfer the samples at the lowest possible temperature at which the sample can be accurately measured into the cup.

6.4 Discard samples in leaky containers.

6.5 Do not store samples in plastic (polyethylene, polypropylene, etc.) bottles, since volatile material may diffuse through the walls of the bottles.

7. Correction for Barometric Pressure

7.1 When the barometric pressure differs from 760 mm Hg (101.3 kPa), determine the temperature at which the test should be conducted by the following equations:

$$F = 120^\circ\text{F} - 0.06(760 - P)$$

$$C = 49^\circ\text{C} - 0.03(760 - P)$$

$$F = 120^\circ\text{F} - 0.42(101.3 - B)$$

$$C = 49^\circ\text{C} - 0.23(101.3 - B)$$

where:

F, C = temperatures to obtain the equivalent of 120°F (49°C) at standard pressure,

P = barometric pressure, mm Hg, and

B = barometric pressure, kPa.

8. Procedure

8.1 Set up the apparatus (see 5.1) in a draft-free area. Place the metal block on the hot plate fitted with a temperature-controlling device or heat the metal block by other suitable means so that the temperature is maintained within $\pm 2^\circ\text{F}$ ($\pm 1^\circ\text{C}$) of the temperature 120°F (49°C) corrected, if necessary, for the difference in pressure from 760 mm Hg (101.3 kPa).

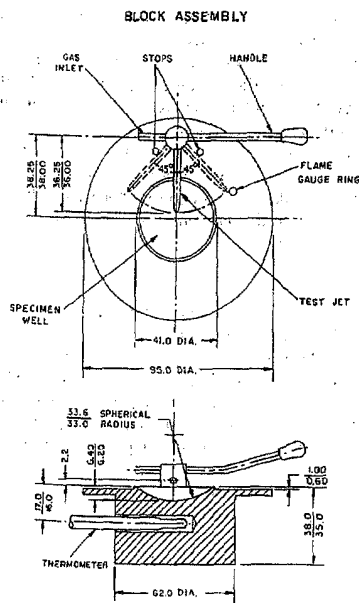
8.2 After the cup is within $\pm 2^\circ\text{F}$ ($\pm 1^\circ\text{C}$) of the correct test temperature, withdraw from the sample container a 2-mL specimen using the graduated syringe or pipet. Transfer the specimen to the well of the tester and immediately start the timing device.

8.3 Light the test flame with the jet in the "off" position away from the well. Adjust the size of the flame so that it is spherical and approximately $\frac{3}{32}$ in. (4 mm) in diameter by matching it to the $\frac{3}{32}$ -in. diameter circle engraved on the surface of the tester.

8.4 After exactly 1 min (0 to +1 s) at which time the test portion will be deemed to have reached the test temperature as indicated by the thermometer embedded in the metal block, swing the test flame into a position exactly over the well. Hold it in this position for exactly 15 s and then return it to the "off" position.

8.5 Observe and time the duration of the burning of the specimen following the return of the flame to the "off" position.

8.6 Repeat the test with a fresh specimen and determine the mean time of burning.



ALL DIMENSIONS IN MILLIMETERS

FIG. 1 Setaflash Tester (Open Cup)

Interpretation of Results

10.1 A product is considered to sustain burning if the specimen (a) ignites when the flame is over the well and burning is sustained for more than 15 s after it is removed, or (b) flashes and burns when the test flame is in the "off" position prior to swinging it over the well.

Report

10.1 Report the following information:

- 10.1.1 Identification of the material under test,
- 10.1.2 Test temperature and barometric pressure in millimetres of mercury (kilopascals),
- 10.1.3 If the specimen burns less than 15 s, the actual time of burning, and
- 10.1.4 Statement of sustained burning characteristics.

Precision

11.1 The precision of this test method will be determined.

ANNEX

A1. Dimensions of Tester

(a) Metal Block Details	Dimensions, mm
Diameter of block	61.5-62.5
Height of block	35.0-38.0
Diameter of flange	94.5-95.5
Flange thickness	3.0 approx.
Height of well "lip" above flange	0.6-1.0
Diameter of well "lip"	41.0 approx.
Spherical radius of well	33.0-33.5
Depth of well	6.2-6.4
Distance from top of block to thermometer hole	16.0-17.0
Thermometer hole diameter	7.0 approx.
(b) Test Gas Jet Details	Dimensions, mm
Outside diameter of jet	3.0-4.0
Jet end tapered to	1.7-2.3
Bore of jet	0.6-0.8
Length of jet (from center of axis to tip)	36.00-36.25
Distance of axis from center of wall	38.00-38.25
Flame gage ring diameter	4.0 ± 0.1
Height of jet above top of well "lip" ^A	2.2 ± 0.1
"Swing" of jet (from stop to stop)	90 ± 1°

^A Adjust with suitable gage.

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Standard Test Method for Viscosity by Dip-Type Viscosity Cups¹

This standard is issued under the fixed designation D 4212; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of viscosity of paints, varnishes, lacquers, and related liquid materials by dip-type viscosity cups. This test method is recommended only for viscosity control work within one plant or laboratory and should not be used to check compliance with specifications.

1.2 Viscosity cups are designed for testing of Newtonian and near-Newtonian liquids. If the test material is non-Newtonian, that is, shear-thinning or thixotropic, another method, such as Test Methods D 2196, should be used.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1200 Test Method for Viscosity by Ford Viscosity Cup²

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer²

D 4287 Test Method for High Shear Viscosity Using the ICI Cone/Plate Viscometer²

E 1 Specification for ASTM Thermometers³

3. Terminology

3.1 Definitions:

3.1.1 *near-Newtonian liquid*—a liquid in which the variation of viscosity with shear rate is small and the effect on viscosity of mechanical disturbances such as stirring is negligible.

3.1.2 *Newtonian liquid*—a liquid in which the viscosity is independent of the shear stress or shear rate. If the ratio of shear stress to shear rate is not constant, the liquid is non-Newtonian.

4. Summary of Test Method

4.1 The cup is completely immersed in the material to be tested, withdrawn, and the time for the material to flow through a hole in the base of the cup is measured.

5. Significance and Use

5.1 Viscosity is a measure of the fluidity of a material. Viscosity data are useful in the determination of the ease of stirring, pumping, dip coating, or other flow-related properties of paints and related fluids.

5.2 This type of cup is used to measure viscosity because it is easy to use, robust, and may be used in tanks, reservoirs, and reactors.

5.3 There are other types of apparatus for measuring viscosity in the laboratory that provide better precision and bias, including the Ford viscosity cup (Test Method D 1200), and the Brookfield viscometer (Test Methods D 2196).

5.4 Certain higher shear rate devices such as cone/plate viscometers (Test Method D 4287) provide more information about sprayability, roll coatability, and other high-shear rate related properties of coatings.

6. Apparatus

6.1 *Zahn Viscosity Cup*⁴—No. 1 through No. 5 Zahn viscosity cups made of corrosion- and solvent-resistant materials. The capacity of the cup is nominally 44 mL, but may vary from 43 to 49 mL, depending on the manufacturer. A diagram of a Zahn cup is given in Fig. 1. The dimensions, including orifices, are only approximate because the cups are not made to one given specification. Each manufacturer produces a different cup and considerable variation between batches from a given manufacturer has been noted in the past. This is a major reason why Zahn cups should not be referenced in specifications between producer and user. (See Appendix X1 for additional information on Zahn Cups.)

6.1.1 Nominal Zahn cup orifice diameters are listed in Table X1.1. Cup No. 1 with the smallest orifice is used for determining the viscosity of thin-bodied materials. Cup No. 2 is for use with clears, lacquers, enamels, and press-side adjustment of flexographic inks; cups Nos. 3 and 4 are for use with more viscous paints and inks (No. 3 for manufacturing of flexographic inks); and cup No. 5 is used for silk screen inks.

6.2 *Shell Viscosity Cup*⁵—No. 1 through No. 6 Shell viscosity cups made of stainless steel with a capacity of 23 mL and a 1-in. (25-mm) long capillary in the bottom and

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

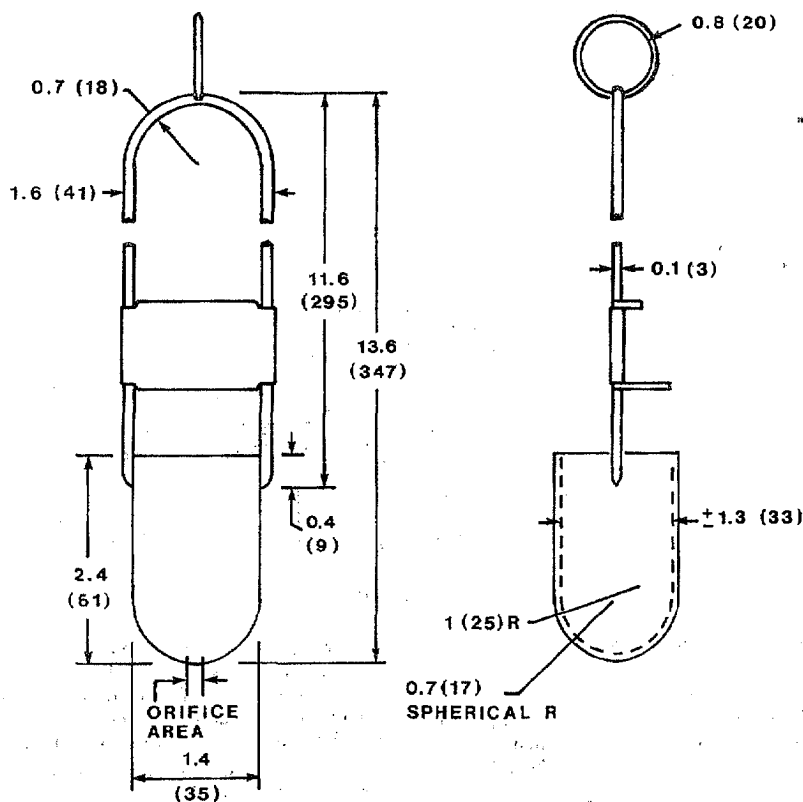
Current edition approved Dec. 30, 1988. Published April 1989. Originally published as D 4212 - 82. Last previous edition D 4212 - 82.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.03.

⁴ Zahn cups may be obtained from Paul N. Gardner Co., P.O. Box 10688, Pompano Beach, FL 33061 and from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910. It should be pointed out that cups from these sources are not the same. Paul N. Gardner "Zahn signature" cup viscosities should not be compared to Pacific Scientific or G.E. cup viscosities or to Paul N. Gardner "EZ" cup viscosities.

⁵ Shell cups may be obtained from the Norcross Corp., 255 Newtonville Ave., Newton, MA 02158.



DIMENSIONS IN INCHES
(MILLIMETERS IN PARENTHESES)

NOTE—Dimensions are approximate only and may vary with the manufacturer and from batch to batch

FIG. 1 Zahn Cup

conforming to the dimensions shown in Fig. 2.

6.2.1 Nominal Shell cup orifice diameters are listed in Table X1.1. Cup Nos. 1 through 2½ are recommended for use with reduced rotogravure inks; No. 2 is for use with flexographic inks; Nos. 3 through 4 are used for industrial enamels, lacquers, flexographic, and gravure inks; Nos. 5 and 6 are used for heavy materials.

6.3 Calibration Thermometer—ASTM Saybolt Viscosity Thermometer 17F having a range of 66 to 80°F and subdivisions of 0.2°F, or 17C having a range of 19 to 27°C and subdivisions of 0.1°C, both conforming to the requirements of Specification E 1.

6.3.1 For general operations, any thermometer with 1°C subdivisions may be used.

6.4 Timer—Any timing device may be used provided that the readings can be taken with a discrimination of 0.1 s or better.

7. Test Materials

7.1 The material to be tested should be visibly homogeneous and free from any foreign material or air bubbles.

8. Temperature of Testing

8.1 Measurements should be made at 77°F (25°C) unless otherwise specified. Temperature drift during the test should be kept to a minimum. The viscosities of paints and related materials are highly dependent on temperature. Differences in temperature between measurements can give substantially different viscosities (up to 5 % per °F). For careful work, the temperature should be taken in the efflux stream, but for process control (such as monitoring a dip tank), this is not necessary.

8.2 A temperature correction curve may be constructed for each liquid by plotting viscosity (seconds) against temperature over the expected temperature range. With this curve, a viscosity determined at one measured temperature may be converted quickly to a viscosity at another temperature.

NOTE 1—When dip cups are used for original purposes, that is thinning or monitoring of materials in tanks, coaters, etc., then temperature is not important. This is because the key to good operation is to maintain the fluid within a certain range of dip cup-seconds

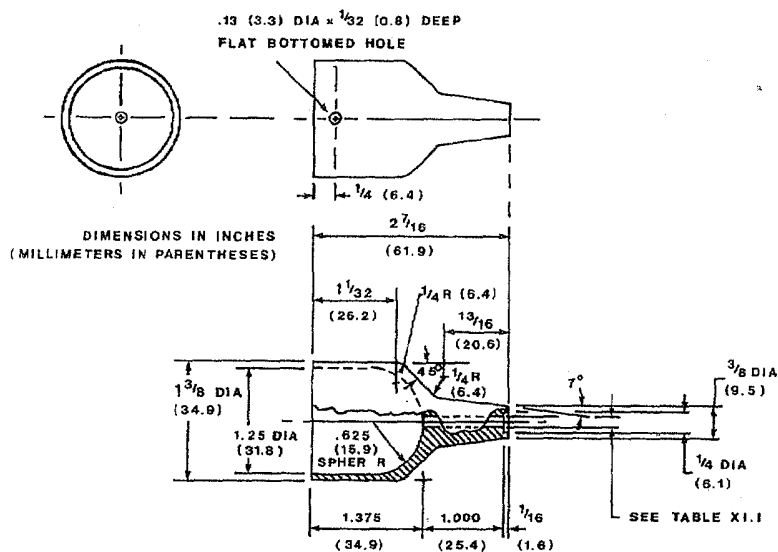


FIG. 2 Shell Cup

regardless of the temperature of the plant or lab.

9. Checking and Calibration of Cups

9.1 Cups should be checked in accordance with the procedure described in Appendix X2. The frequency of this depends upon the amount of use and care that the individual cup receives.

9.2 Cups may be calibrated with standard oils according to the procedure in Appendix X3. However, because of the great temperature dependence of the viscosity of standard oils and the lack of adequate temperature control with dip cups, calibration is a difficult and often inaccurate procedure that must be done with great care. Because of the wide variability in Zahn cups, checking rather than calibration is recommended.

10. Procedure

10.1 Choose the proper cup so that the time of efflux will be between 20 and 80 s. See Table 1 for viscosity ranges for the various cups.

10.2 Immerse the cup in the container, which may be a can or beaker, but is more likely to be a thinning or mixing

tank or even a resin reactor. Stir or agitate the fluid well to give uniform temperature and density. Allow the cup to remain in the fluid for 1 to 5 min to attain thermal equilibrium. (Because of their greater mass, Shell cups should remain in the fluid for the full 5 min.)

NOTE 2—Dip cups are not recommended for use with thixotropic (time dependent) materials but if used for them (such as gravure or flexographic inks), more vigorous agitation will be necessary to break up the structure before the measurement is made.

10.3 Lift the cup vertically out of the material in a quick, steady motion. As the top edge of the cup breaks the surface, start the timer. During the time of flow hold the cup vertically no more than 6 in. above the level of the liquid. Stop the timer at the first definite break in the stream at the base of the cup. The efflux time in seconds constitutes the viscosity. It is common to make only a single measurement, but for greater precision and accuracy the mean of two or more measurements should be taken.

11. Care of Cups

11.1 Following each determination, clean the cup with a suitable solvent and a soft brush. Use no metal tools in contact with the instrument as nicks or wear of the drilled orifice affect the accuracy of the cup.

12. Report

12.1 Report the efflux time to the nearest 0.2 s for Zahn or Shell cup No. ____, manufactured by ____, the temperature of the fluid (and where measured, and whether the result is from a single measurement or the mean of two or more measurements).

13. Precision and Bias

13.1 Dip cups are not precision instruments and should

TABLE 1 Approximate Viscosity Ranges, cST (Roughly Corresponding to 20 to 80 s Flow Time)

Cup Number	Zahn Cup	Shell Cup
1 ^A	5-60	2-20
2	20-250	10-50
2 1/2	...	20-80
3	100-800	30-120
3 1/2	...	40-170
4	200-1200	70-270
5	400-1800	125-520
6	...	320-1300

^A The lower limit for the Zahn No. 1 cup is 35 s rather than 20 s.

used for viscosity control at a given location only. However, if comparisons are made, cups from the same manufacturer *must* be used. The following criteria can be used for judging the acceptability of results at the 95 % confidence level:

13.1.1 *Zahn Cups*—Precision was determined on the basis of an interlaboratory test in which six laboratories used new Zahn cups (all from the same set from the same manufacturer) to test eight paints covering a broad range of viscosities. The within-laboratory coefficient of variation was 3.7 % and the between-laboratories coefficient of variation was 11.5 %. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.1.1 *Repeatability*—Two results, each the mean of two measurements, obtained by the same operator should be considered suspect if they differ by more than 11 % of their mean value.

13.1.1.2 *Reproducibility*—Two results, each the mean of two measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 33 % of their mean value.

NOTE 3—Since the precision values were obtained under ideal conditions (a single set of cups), reproducibility in practice probably is poorer than that given (perhaps as bad as 50 %).

13.1.2 *Shell Cups*—Precision was determined on the basis of an interlaboratory test in which four laboratories tested seven paints covering a broad range of viscosities. The within-laboratory coefficient of variation was 3.2 % and the between-laboratories coefficient of variation was 6.3 %. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.2.1 *Repeatability*—Two results, each the mean of two measurements, obtained by the same operator should be considered suspect if they differ by more than 9 % of their mean value.

13.1.2.2 *Reproducibility*—Two results, each the mean of two measurements, obtained by operators in different laboratories should be considered suspect if they differ by more than 18 % of their mean value.

14. Index Terms

14.1 This test method is indexed under the following terms: viscosity; dip cup(s); Zahn cup(s); Shell cup(s); flow cup(s).

APPENDICES

(Nonmandatory Information)

X1. ZAHN CUP DESIGN, MANUFACTURE, AND USE

X1.1 Zahn cups were designed (and made by General Electric) as simple flow property devices for use in dip tanks, flow coat reservoirs, etc. They never were meant to be viscometers or to be used as such. Zahn cup use usually involves thinning or maintaining a coating, adhesive, or ink at a certain consistency (so many Zahn-seconds) regardless of temperature. This is the beauty of dip cups. The operator knows that his or her tank, bath, or coater runs well over a certain range of Zahn-seconds whether the plant temperature is 50°F or 100°F. The operator does whatever is necessary to keep the fluid within the range. He or she does not care what the viscosity is at 25.0°C (77.0°F).

X1.2 Because the Zahn cup patents have long since run out, the cups can be made by anyone. Each manufacturer makes cups that are somewhat different from those made by others. Considerable batch-to-batch difference from any given manufacturer has been seen in the past. Cup variations are not a problem for control of a bath or tank as long as new cups are compared to old ones for the Zahn cup-seconds operating window and a new window is established if necessary. However, cup differences cause great difficulty if cups are used to set producer-user specifications. Comparisons are almost impossible under such conditions.

X2. DIP CUP CHECKING PROCEDURES

X2.1 Because Zahn cups vary with the manufacturer and batch, it is not possible to set up calibration equations that are applicable to all cups. Therefore, the only procedures that are recommended checking a cup against its original efflux time or comparing one cup against another. However, if a cup owner has an absolute compulsion to calibrate a Zahn cup, there are equations available (see X3). Shell cups may be checked by the same procedures as Zahn cups or may be calibrated.

X2.2 *Monitoring Cup Characteristics*—A useful checking technique is to measure the efflux time for a new cup with a given standard oil, then check the cup periodically with the same oil at the same exact temperature to see whether the efflux time has changed. If the time changes drastically (greater than 20 %), the cup should be replaced. If the change is small, the cup can be used as is or a correction can be made in subsequent efflux times of paints or other materials. The correction factor is equal to original oil efflux time divided by the current one. Recommended oils for such tests are given in Table X2.1. The viscosity of oils is very sensitive to temperature variations and, therefore, the temperature of the oil must be controlled closely during testing. It is recommended that the oil be held at the test temperature for at least 15 min prior to testing.

X2.3 *Comparing Cups*—It sometimes is necessary to compare one Zahn cup with another in order to settle a dispute, determine whether a new cup will give similar times to an old one, etc. The most usual technique is to dip both

TABLE X2.1 Viscosity Standards Recommended for Checking Cups

Cup Number	Nominal Diameter ^A of Orifice, mm	Standard Oil Number ^B	Approximate Oil Viscosity ^C at 77°F (25°C), cST
Zahn			
1	2.0	S10	20
2	2.7	S60	120
3	3.8	S200	480
4	4.3	S200	480
5	5.3	S350, S600	900, 1600
Shell			
1	1.8	S6	9
2	2.4	S6, S10	9, 20
2½	2.7	S20	35
3	3.1	S20, S60	35, 120
3½	3.5	S60	120
4	3.8	S60	120
5	4.6	S60, S200	120, 480
6	5.8	S200	480

^A Information based on literature of cup manufacturers.

^B Certified kinematic viscosity standards (1-pt samples only) are available from the Cannon Instrument Co., P.O. Box 16, State College, PA 16801. Oils from other sources, having known kinematic viscosities, may also be used.

^C Actual viscosity is noted on the label of the standard.

cups into the same container of standard oil at the same time and pull them out together, timing both of them. The efflux times are compared and a correction factor can be calculated. Sometimes paint or another material is substituted for the oil.

X3. CALIBRATION OF DIP CUPS

X3.1 Select the appropriate standard oil for the cup to be checked (see Table X2.1).

X3.2 Bring the cup and the standard oil to a constant temperature as close as possible to 77.0°F (25.0°C). Determine the time of efflux to the nearest 0.2 s using the procedure detailed in Section 10. Record the temperature of the efflux stream. If it is not 77°F, correct the viscosity of the standard oil to the actual temperature.

X3.3 Convert the time of flow in seconds to kinematic viscosity as follows:

$$V = K(t - c)$$

where:

V = kinematic viscosity, cST

t = efflux time, s, and,

K, c = appropriate constants (from Table X3.1).

X3.3.1 These equations represent linear or relatively linear portions of the overall viscosity-time curves for the cups. The linear equations have been chosen because it is much more straightforward to make time corrections based on linear equations than on nonlinear ones. Zahn cup nonlinear equations that better fit viscosity curves for some

TABLE X3.1 Constants ^A for Use with Viscosity Formulas

Cup	K	C
Zahn		
1	1.1	29
2	3.5	14
3	11.7	7.5
4	14.8	5
5	23	0
Shell		
1	0.226	13
2	0.576	5
2½	0.925	3
3	1.51	2
3½	2.17	1.5
4	3.45	1
5	6.5	1
6	16.2	0.5

^A Cup constants from Palton, T. C., *Paint Flow and Pigment Dispersion*, second edition, John Wiley & Sons, New York, 1979, p. 82.

cups better may be found in the literature.^{6,7} No such equations for Shell cups have been published.

X3.4 Calculate the correction factor by dividing the true kinematic viscosity of the standard oil by the kinematic

⁶ Euverard, M., *ASTM Bulletin*, Vol 162, No. 67, October 1950.

⁷ Pierce, P. E., *Journal of Paint Technology*, Vol 42, No. 533, 1969, p. 383.

viscosity calculated from the efflux time. This factor may then be used to correct viscosity readings taken with the cup.

The product of the factor and an efflux time gives a corrected viscosity in Zahn or Shell seconds.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Wet Abrasion Resistance of Interior Paints¹

This standard is issued under the fixed designation D 4213; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers an accelerated procedure for determining the resistance of interior paints to erosion caused by scrubbing.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer²
- D 1193 Specification for Reagent Water³
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products²
- D 2369 Test Method for Volatile Content of Coatings²
- D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints²
- D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings²
- D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings²
- D 3450 Test Method for Washability Properties of Interior Architectural Coatings²
- D 3980 Practice for Interlaboratory Testing of Paint and Related Materials²
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴

3. Summary of Test Method

3.1 The test paint is applied to a black plastic panel. After conditioning, the coated panel is placed over a glass plate in a washability machine and scrubbed with a fine pore cellulosic sponge and an abrasive medium. The weight of the film eroded is determined by weighing the panel before and after the test, and the volume loss per stated number of cycles is calculated.

4. Significance and Use

4.1 Interior paints often become soiled especially near doorways, windows, and in work and play areas. This test

method covers determination of the relative resistance of different interior paints to erosion when scrubbed.

4.2 Results obtained by this test method do not necessarily represent the scrub resistance of aged paint films, since scrub resistance may change with age.

4.3 Results obtained by this test method also do not necessarily relate to relative ease of soil removal.

5. Apparatus

5.1 Wet Abrasion Test Apparatus.⁵

5.1.1 Accessory Apparatus (Fig. 1).⁵

5.1.1.1 *Sponge Holder*,⁵ sponge and sheet lead weights (total weight 500 ± 10 g).

5.1.1.2 *Glass Plate*, measured to fit.

5.1.1.3 *Gasketed Frame and Clamps*.

5.2 *Film Caster*,⁷ having a 10 mil (250 μ m) clearance and 5.5-in. (140-mm) path width.

5.3 *Balance*, weighing to 1 mg.

5.4 *Oven*, adjusted to 120 to 130°F (50 to 55°C).

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. Unless otherwise indicated, it is intended that all reagents shall conform to the Specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁸ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

6.3 *Black Plastic Panels*.⁹

6.4 *Masking Tape*.

6.5 *Flannel Cloth*.

⁵ The Gardner Abrasion Tester Model AG 8100 and accessory apparatus, available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910 have been found satisfactory for this purpose. Other straight line wash testers may be adapted to meet the requirements of this test method.

⁶ This holder is also used as a brush holder in Test Method D 2486. It should not be confused with the sponge holder employed in Test Method D 3450.

⁷ The Dow film caster, available from the Gardner Laboratory Div., has been found satisfactory for this purpose.

⁸ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

⁹ Leneta P-121-10N dull black plastic panels, 6½ by 17 in. by 10 mils (165 by 432 by 0.25 mm) in size obtainable from the Leneta Co., P. O. Box 86, Ho-Ho-Kus, NJ 07423, are suitable for the purpose. An equivalent may be used.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Dec. 14, 1987. Published February 1988. Originally published as D 4213 - 83. Last previous edition D 4213 - 83.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁴ Annual Book of ASTM Standards, Vol 15.05.

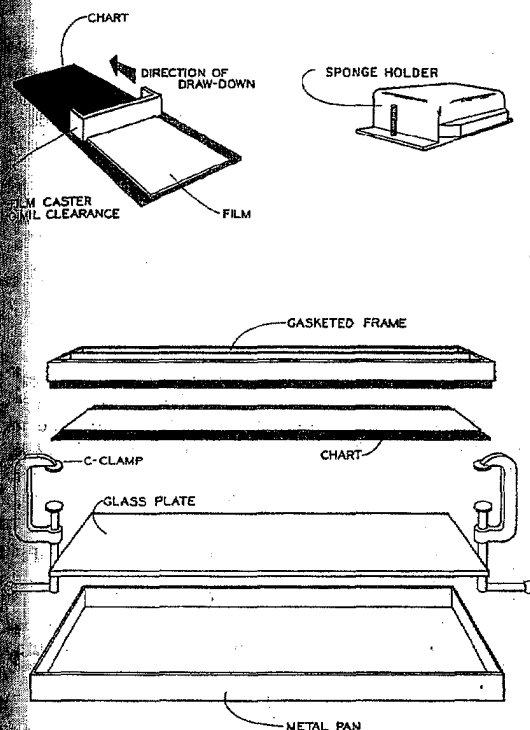


FIG. 1 Accessory Apparatus for Scrub Test

6.6 Detergent.¹⁰

6.7 Sponge.¹¹ fine pore, uncompressed sponge conforming to U.S. Federal Specification L-S-626, Type II, Fine Pore, Uncompressed.

6.8 Scrub Medium.¹² (Note 1), consisting of the following:

Water, reagent, Type IV	49.4 ^a
Hydroxyethylcellulose ¹³	0.8
Ammonium hydroxide, 28 %	...
Detergent ¹⁰	2.0
Trisodium phosphate, anhydrous	2.0
Silica ¹⁴	45.0
Acetic acid, glacial	0.7 ^b
Preservative ¹⁵	0.1
	100.0

^a Vary to achieve a viscosity from 110 to 120 Krebs Units before use, as measured in accordance with Test Method D 562.

^b Vary to achieve a pH from 9.5 to 10.0 as measured in accordance with Test Method E 70.

¹⁰ Octyl phenoxy polyethoxyethanol containing approximately ten moles of ethylene oxide per mole has been found satisfactory. Triton X-100, obtainable from Rohm and Haas Co., Independence Square, Philadelphia, PA, is representative of this product. An equivalent may be used.

¹¹ Super-Cel Cellulose Sponge, Fine Pore #85, obtainable from American Sponge and Chamois Co., Inc., 47-100 34th St., Long Island City, NY 11101, has been found satisfactory for this purpose. An equivalent may be used.

¹² Laboratory standardized scrub medium is available from the Leneta Co. An equivalent may be used.

NOTE 1—When a referee test is made, prepare fresh medium or use standardized scrub medium from a previously unopened container that is no more than one year old.

6.8.1 Slowly add the hydroxyethylcellulose to the water while stirring mechanically. Stir until uniform and then slowly add 2 to 3 drops of 28 % ammonium hydroxide solution while mixing and continue mixing until the solution turns clear. In the order shown, add the other ingredients separately, stirring continuously. Be sure each item is uniformly dispersed before adding the next one. Add the silica slowly to ensure uniform dispersion. Finally, add the preservative and adjust the pH with glacial acetic acid.

7. Preparation of Apparatus

7.1 *Scrub Test Apparatus*—The scrub machine should be leveled before use and operated at 37 ± 1 cpm. Before each test check the tightness of the motor belt or chain drive and alignment of the cables in both horizontal and vertical planes. Adjust tautness of the cables with the accessory spring scale to approximately 5 lb.

7.2 Cut the sponge to fit the standard brush holder, $3\frac{3}{4}$ by $1\frac{1}{2}$ by $1\frac{1}{2}$ in. (95 by 38 by 40 mm). New sponges should be saturated with reagent water, wrung dry by hand and allowed to equilibrate in a constant temperature and humidity room for 24 h before being cut.

7.3 *Brush Holder*—Add sufficient weight to the holder in the form of sheet lead or other flat weights to total 500 ± 10 g including the sponge.

8. Procedure

8.1 Stir the material under test thoroughly and strain to remove all skins and particles. Clean the top of the glass plate (or preferably suction plate) and both sides of the black plastic panel to be sure they are free of specks. Place the black panel on the plate and tape one end to the plate. Smooth the panel along the plate by rubbing with flannel, creating static electricity to improve adhesion to the plate.

8.2 Drawdown the paint on the panel using the 10-mil (0.25-mm) side of the film caster, starting as close to the taped end of the panel as possible (should be less than $\frac{1}{2}$ in. (13 mm) from the taped end of the panel). The time of application should be fairly slow—3 to 4 s from end to end—to prevent formation of pinholes or holidays in the film. Air dry in a horizontal position for seven days in an open room, preferably kept at $73 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and 50 ± 5 % relative humidity.

8.3 After the specified drying time, cut off the top $\frac{3}{4}$ in. (20 mm) of the panel, preferably with a paper cutter to

¹³ Hydroxyethylcellulose having a molar substitution (MS) value of 1.8 to 2.5 and a viscosity of a 2 % solution in the range of 4400 to 6500 cPs. Cellosize QP-4400, obtainable from Union Carbide Co., Chemicals Div., 270 Park Ave., New York, NY 10017, has been found satisfactory for this purpose, as well as Natrosol 250 MR obtainable from Hercules Inc., 910 Market St., Wilmington, DE 19899.

¹⁴ This silica is an amorphous grade of 99+ % silicon dioxide giving a pH of 6.8 to 7.2 in reagent water. Average particle size is $2.95 \mu\text{m}$ with 92 % being less than $50 \mu\text{m}$ and surface area of $7600 \text{ cm}^2/\text{g}$. Silica No. 22 obtainable from Whittaker, Clark and Daniels, Inc., 100 Church St., New York, NY 10007, has been found satisfactory for this purpose. An equivalent may be used.

¹⁵ 1,3,5 triethyl hexahydro-sym-triazine (Vancide TH) obtainable from R. T. Vanderbilt Co., 30 Winfield St., Norwalk, CT 06855, has been found satisfactory. An equivalent may be used.

ensure a smooth straight edge. The film should now cover the panel uniformly along its entire length, exactly 16 1/4 in. (413 mm). The panel may now be cut in half, *length-wise*, so that duplicate tests can be run. Roll the test panels into the shape of a cylinder to fit the balance pan, fasten the ends together with a paper clip and weigh to 1 mg. Record this as Weight A. Remove the clip and carefully unroll the panel. Save the paper clip for later weighings. Avoid any damage to the film after weighing.

8.4 Clean the glass plate and set it in the pan of the scrub test apparatus. Place the test panel, with painted side up, over the glass plate so that the test area is level. Pre-wet the gasket of the frame (if frame is to be used), and wipe dry before use. Place the frame over the drawdown and clamp in place. Clamps should be tight enough to ensure close contact but not tight enough to cause warping of the panel.

8.5 Place the sponge in reagent water and allow to remain for 30 min. Remove, hand squeeze dry, and insert in the weighted holder. Stir the scrub medium and spread 10 g evenly over the face of the sponge. Place the sponge at the end of the panel and wet the path of the sponge on the panel with 5 mL of water.

8.6 Note counter reading on scrub test apparatus and start the test. Make sure the sponge travels only over the test panel and only over the coated portion of the panel, and that sponge and holder are level during run. If the sponge and carriage tend to tilt sidewise, twist the cable in the direction in which the holder is tilting to impart an opposing torque to the sponge and holder enabling them to ride upright.

8.6.1 **Caution**—Cables should not be twisted more than three or four times. Excessive twisting in the direction opposite to that used to form the cable can cause unraveling of cable strands.

8.7 After each 100 cycles examine the panel for erosion through to the substrate. If cut-through is evident, stop the test and record the number of cycles. Otherwise continue until 400 cycles, at which time add 10 g of stirred medium to the sponge again and 5 mL of water to the panel, and continue testing to a maximum of 1000 cycles. Add 10 g scrub medium and 5 mL deionized water at the end of every 400 cycles.

8.7.1 In instances where cut-through is observed, repeat the test using the other half of the test chart and stop at least 100 cycles short of the previously noted cut-through point. It is important not to erode the film to the point where the substrate shows through since this will cause an erroneous result.

8.8 Rinse off the panel with tap water making sure all scrub medium has been removed, brushing gently with a soft camel hair brush to facilitate removal of the scrub medium. Blot the panel (do not rub) with paper towels, allow to air dry for 30 min, and then place flat in the oven at 120 to 130°F (50 to 55°C) for 30 min. Remove and allow to equilibrate for 15 to 20 min preferably in a conditioned room, or if necessary at ambient conditions surrounding the balance. Roll the panel into a cylinder, clip the edges together with the paper clip used in the initial weighing, and weigh to 1 mg. Record this as Weight B. Then calculate the Erosion Rate C in accordance with 9.1.

8.9 Between runs, rinse the sponge thoroughly to remove as much detergent as possible, soak the sponge in reagent

water before the next test, and then proceed as in 8.5.

8.10 Make three drawdowns from each sample. Test two panels (four tests), then calculate the mean value of C and report this value unless any of the results differ by more than 25 % of the mean. If they do, test the third panel and include the two results in the mean unless one of the results is obviously discrepant, in which case it should be discarded.

NOTE 2—See Table 3 of Practice D 3980 for limits used in rejecting extreme values.

8.11 Determine the density (D) of the paint in g/mL, in accordance with Test Method D 1475.

8.12 Determine the percent nonvolatile by weight, (W), of the paint, in accordance with Test Method D 2369.

9. Calculation

9.1 Determine the total weight loss in milligrams for each panel by subtracting B from A. Divide the weight loss by the number of cycles and multiply by 100 to determine the erosion rate C expressed as milligrams lost per 100 cycles, thus:

$$C = \frac{(A - B) \times 100}{\text{number of cycles}}$$

9.2 Calculate V, the percent nonvolatile by volume of the paint as follows:

9.2.1 *Water-borne Paints*— $V = 100 - D(100 - W)$

9.2.2 *Solvent-borne Paints*— $V = 100 - 1.28D(100 - W)$

9.3 Calculate the erosion rate in terms of wet film volume, thus:

$$\frac{\text{microlitres}}{100 \text{ cycles}} = \frac{100 C}{WD}$$

NOTE 3—This value relates to the whole paint and is thus analogous, though inversely, to the cycles-to-failure values of Test Method D 2486.

9.4 Calculate the erosion rate in terms of dry film volume, thus:

$$\frac{\text{microlitres}}{100 \text{ cycles}} = \frac{CV}{WD}$$

NOTE 4—This value relates to the nonvolatile portion of the paint. The film volume in the equation is exclusive of interstitial air, whether due to vesiculated beads or ordinary porosity.

10. Report

10.1 Report the following information:

10.1.1 Erosion rate of the equivalent wet film in microlitres/100 cycles, as calculated in 9.3.

10.1.2 Erosion rate of the dry film in microlitres/100 cycles, as calculated in 9.4

10.1.3 Number of tests.

10.1.4 Number of cycles at which any erosion to the substrate initially occurred.

10.1.5 Any deviations from the standard procedure.

11. Precision

11.1 In an interlaboratory study of this test method in which operators in six laboratories tested three latex coatings covering a range of pigment volume concentrations, the within-laboratory coefficient of variation was found to be 5.6 % and the between-laboratories coefficient 9.4 %. Based

When these coefficients of variation the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results, each the mean of two repeat tests, obtained by the same operator, should be considered suspect if they differ by more than 18 % of the mean.

11.1.2 *Reproducibility*—Two results, each the mean of two repeat tests, obtained by operators in different laborato-

ries, should be considered suspect if they differ by more than 30 % of the mean.

12. Indexing Terms

12.1 This test method is indexed under the following terms: abrasion (of paints/related coatings); interior paints/coatings; resistance—abrasion (of paints/related coatings); resistance—scrubbing; weight—weight loss.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films¹

This standard is issued under the fixed designation D 4214; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the evaluation of the degree of chalking on white or lightly tinted exterior paint films. It describes the procedures recommended for transferring the chalk to a fabric or fingertip, which is then compared to photographic reference standards, or in the case of adhesive tapes, compared to a reflectance table or photographic reference standards, to determine the degree of chalking.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 659 Method of Evaluating Degree of Chalking of Exterior Paints²

D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints²

D 1150 Single and Multi-Panel Forms for Recording Results of Exposure Tests of Paints^{2,3}

E 97 Test Method for Directional Reflectance Factor, 45-Deg 0-Deg, of Opaque Specimens by Broad-Band Filter Reflectometry²

2.2 Other Document:

Pictorial Standards of Coating Defects⁴

3. Terminology

3.1 Definition:

3.1.1 *chalking*—the formation on a pigmented coating of a friable powder evolved from the film itself at or just beneath the surface.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved March 31, 1989. Published June 1989. Originally published as D 4214 - 82. Last previous edition D 4214 - 82 (1987)¹.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ These record sheets may be obtained from ASTM, 1916 Race St., Philadelphia, PA 19103 (request Adjunct Nos. 12-411500-11 and 12-411500-21) and from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

⁴ Copies of the pictorial photographic reference standards applicable to Test Method D 659 are contained in the publication *Pictorial Standards of Coatings Defects* and may be obtained from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

4. Significance and Use

4.1 The procedures provide a broader range of techniques and photographic references to evaluate chalking of exterior paints than that in Method D 659 and are thus an extension of that method.

5. Type of Chalking

5.1 Only one type of chalking is recognized, as defined in Section 3.

6. Use of Photographic Reference Standards

6.1 The photographic reference standards that are part of this test method are representative of the degrees of chalking on a paint film. The photographs shown in Figs. 1 and 2 are for illustration purposes only and should not be used for evaluation.

6.2 The use of photographic reference standards illustrated in Figs. 1, 2, 3, and 4 requires the following precautions:

6.2.1 The degree of chalking will vary over any given area. Therefore, an average portion of the coating should be evaluated. On large surfaces, it is recommended that the rating be made at several locations and the mean and range reported.

6.2.2 It is difficult to make readings on a windy day and making readings at such time should be avoided. It should also be noted that rain, snow, or moisture in any form will remove chalk so that readings should be made after a period of clear weather and when the surface is dry.

6.2.3 Chalking and erosion (Note 1) are closely related and erosion is a result of chalking failure. However, the rate of chalking as measured by these test methods, and the rate of erosion may not be comparable because some pigment combinations tend to retain chalk on the surface while other pigment combinations exert a self-cleaning action by natural means.

NOTE 1—For the evaluation of erosion, see Test Method D 662.

6.3 Records may be kept on forms such as shown in Fig. 3, according to Standard D 1150, or other inspection forms. The reporting of the results shall include the information given in Section 10.

6.4 When this test method is referenced in specifications for performance, the permissible degree of chalking is established between the producer and the user.

7. Recommended Procedures—Wood Substrates

7.1 Test Method A—Method D 659:

7.1.1 *Material*—Fabric, as agreed upon between the producer, user, or other interested parties, to rub against the

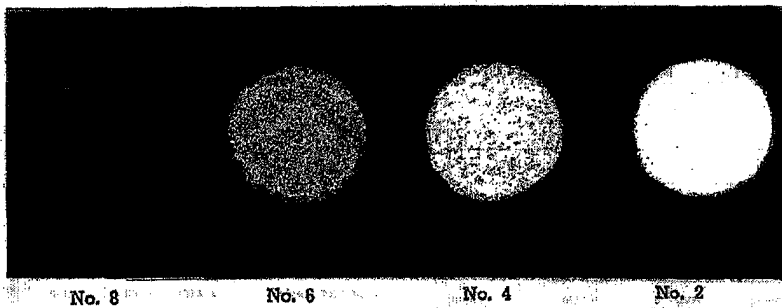


FIG. 1 Photographic Reference Standard No. 1—Test Method D 659

surface being tested. Black wool felt, velvet, and velveteen have proven particularly effective.

3.1.2 Procedure—Wrap the fabric around the index finger, then apply it with medium pressure to the coating under observation. Rotate the finger through an angle of 90° holding the fabric so it also rotates. Remove the fabric and compare the spot of chalk on it with Photographic Reference Standard No. 1.

7.2 Test Method B—Stroke Method:

7.2.1 *Material*—Fabric as in 7.1.1.

2.2.2 Procedure—Wrap the fabric around the index finger, then make a 3-in. (75-mm) stroke with medium pressure on the coating under observation. Remove the fabric and compare the spot of chalk on it with Photographic Reference Standard No. 1.

3 Test Method C—Wet Finger Method:

7.3.1 Procedure—Moisten a fingertip and with medium pressure make one continuous rub 2 to 2½ in. (50 to 65 mm) in length on the surface under test. Compare the rub-off on the finger with Photographic Reference Standard No. 1. This test method can be used quite effectively by experienced operators and is recommended mainly for on-the-field evaluation when use of one of the other methods is impractical.

Recommended Procedures—Metallic Substrate—Industrial, Automotive, and Coil Coatings

8.1 *Test Method C—Wet Finger Method—See 7.3.*

8.2 Test Method D—Transparent Tape Method:⁵

8.2.1 Materials:

8.2.1.1 *Cellulose Adhesive Tape*,⁶ 1/2 in. (13 mm) wide, pressure-sensitive.

8.2.1.2 *Eraser*, $\frac{3}{4}$ in. (20 mm), wrapped with cellophane

8.2.1.3 *Masking Tape*, ½ in. (13 mm) wide.

8.2.1.4 *Plastic Sheet Protector*, clear.

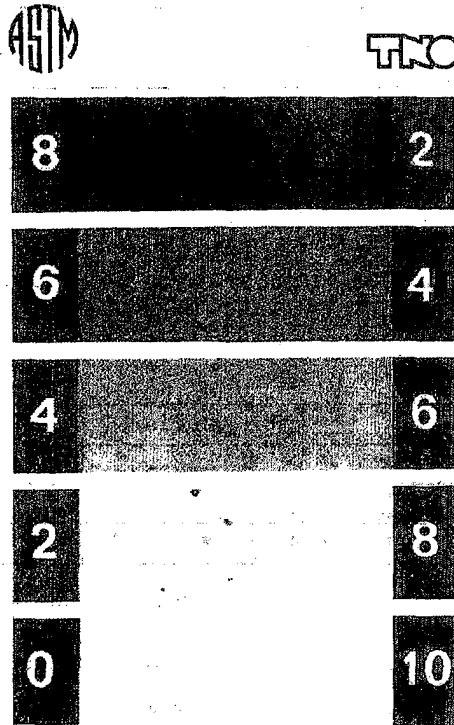


FIG. 2 Photographic Reference Standard No. 2—Verfinstituut
TNO

8.2.1.5 *Photographic Reference Standard No. 2, TNO.*⁷

8.2.1.6 *Reflectance Standard*, polished black glass.

8.2.1.7 *Reflectance Standard*, white tile.

Permission to include this method is provided as a courtesy of NL Chemicals,
Croff Mills Rd., Hightstown, NJ 08520.

Permacel 404 manufactured by Permacel, Inc., Route 1, New Brunswick, NJ 08903 and Scotch Brand tape No. 600 manufactured by The 3M Company, Packaging Systems, 3M Center, Bldg. 230F267, St. Paul, MN 55144, have been found suitable for the purpose.

⁷ The TNO Method and photographic reference standard are provided as a courtesy of *Verinstituut TNO Paint Research Institute* TNO, Schoemakerstraat 97, Delft, Nederland. The original source of the photographic reference standard illustrated in Fig. 2 is the Paint Research Institute, TNO. The ASTM numerical rating of chalking shown on the photographic reference standard is opposite to the original TNO scale.

TAPE CHALK RATING WORKSHEET

Reflectance Method

Client Name _____

Order # _____

Radiation to date _____

	Measurements	Avg %	Corrected Value	Rating
Sheet				
Tape & Sheet				
1st Set	2nd Set			
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
Tape & Sheet				
Sheet				

Data _____

Inspected by _____

NOTE—Label sample numbers, apply initial blank tape, and proceed with tape specimens of the samples.

FIG. 3 Example of Worksheet

NOTE 2—The black reference standard is necessary as the background for this measurement, since the reflectance of black paper is too high. Reflectometers (tristimulus colorimeters), with 0 to 45 degree geometry, use the Y value.

8.2.2 Optional Materials:

8.2.2.1 China Marker, black.

8.2.2.2 Razor Blade.

8.2.3 Preparation:

8.2.3.1 Separately mount and apply two 11-in. (279-mm) pieces of masking tape along the right side of the clear plastic sheet cover leaving 1/4 in. (32 mm) of space between the pieces (see Fig. 3).

8.2.3.2 Remove a 2 in. (50 mm) long piece of 1/2 in. (13

TAPE CHALK RATING WORKSHEET SHEET

Reflectance Method

BLANK

Client Name ABC CompanyOrder # 164433Radiation to date 6/6 11/10 2/01

	Measurements	Avg %	Corrected Value	Rating
Sheet	0.84	0.0046		
Tape & Sheet	2.38	0.0289	0.0194	4.0
1st Set	2nd Set			
1	0.40	0.14	0.2380	2.0
2	0.46	0.15	0.1664	4.5
3	0.47	0.17	0.1611	4.5
4	0.45	0.16	0.1484	5.0
5	0.44	0.14	0.103	3.5
6	0.43	0.10	0.1024	3.5
7	0.41	0.08	0.1068	3.0
8	0.34	0.06	0.0862	2.5
9	0.29	0.03	0.2419	0.5
10	0.26	0.01	0.0540	0.0
Tape & Sheet	2.40	0.0289	0.0194	
Sheet	0.46	0.0046		

Date 6-20-01Inspected by John R. Doe

NOTE—The cut-out section is for illustration purposes only. The labeling (china marker) may be removed by rubbing the sheet with a clean tissue or cloth.

FIG. 4 Completed Worksheet

mm) cellulose, pressure-sensitive adhesive tape from the roll, place it across the masking strips, and adhere it to the sheet using a 3/4-in. (20-mm) eraser, wrapped with cellophane tape. Label this tape "blank" on the clear plastic sheet cover. A black china marker has been found useful for this purpose.

NOTE 3—The average reflectance measurements of the initial and ending "blank" tapes less the correction value for the clear plastic sheet divided by 100 are used to verify a rating of 10 using Table 1.⁸

8.2.4 Procedure:

8.2.4.1 Apply a 2 in. (50 mm) long piece of 1/2 in. (13 mm) wide tape to the surface being rated. Rub ten times with moderate pressure using the covered eraser, to remove all bubbles and prevent scratching. Remove the tape from the surface and adhere it to the sheet by rubbing with the eraser. Label specimens using a black china marker. Place successive tapes vertically adjacent to previous tapes, separated by 1/8 in. (3 mm). Follow the instructions given in 8.2.3.2, and place the final "blank" tape across the masking tape strips and label "blank" on the clear sheet. When completed, use a razor blade to cut along the inside edges of the masking tape, cutting through the adhesive tapes. The removal of the masking tape will leave only the tapes to be measured and evaluated with the sample number of each tape listed on the

⁸ Kronos-Titan Table for Chalk Rating from Reflectance Reading using the Transparent Tape Method is provided as a courtesy of Kronos-Titan GmbH, Leverkusen, West Germany. The original source of Table 1 is Kronos-Titan GmbH.

TABLE 1 Chalk Rating from Reflectance Reading^a

Reflectance Range	Chalk Rating
0-0.038	10
0.0381-0.044	9.5
0.0441-0.054	9.0
0.0541-0.062	8.5
0.0621-0.072	8.0
0.0721-0.082	7.5
0.0821-0.095	7.0
0.0951-0.105	6.5
0.1051-0.120	6.0
0.1201-0.131	5.5
0.1311-0.150	5.0
0.1501-0.165	4.5
0.1651-0.190	4.0
0.1901-0.210	3.5
0.2101-0.235	3.0
0.2351-0.260	2.5
0.2601-0.285	2.0
0.2851-0.310	1.5
0.3101-0.340	1.0
0.3401-0.366	0.5
>0.366	0

^aThis table is based upon a correlation between tape reflectance measurements and visual evaluations of the same tape compared to the photographic standards prepared by Kronos-Titan.⁸

sheet (see worksheet example in Fig. 4). Before proceeding, check to ensure all sample numbers have been recorded on the sheet.

8.2.4.2 Insert the 1/2-in. (13-mm) aperture and calibrate the reflectometer according to the manufacturer's instructions, setting the reflectometer for zero reflectance using the black reflectance standard and standardizing with the white reflectance standard and record the values. Refer to Test Method E 97 should there be any question on the correct procedure to follow in the calibration of the instrument.

8.2.5 Reflectometer Measurements:

8.2.5.1 Leave the transparent tapes mounted on the clear plastic sheet. Remove the black paper that may have been inserted behind the sheet and fold back the unused portion of the sheet. Measure the reflectance of the clear plastic sheet using the black reflectance standard of the instrument (Note 1) as a backing or background and record its value. Move the sheet until the first tape is exposed to the light source with the adhesive side toward the light and the black reflectance standard behind the area being measured, and record the value.

8.2.5.2 Continue this procedure until ten tapes have been measured, then check reflectance values for the white and black standards. If no changes have occurred, proceed with measurements. If values have changed, restandardize and record values before proceeding to the remaining tapes. Following the final tape measurement, record reflectance values of the clear plastic sheet cover and the white and black reflectance standards.

8.2.5.3 Subtract the mean reflectance value of the sheet from each reading, enter on worksheet form (Fig. 4), or other form used, and determine from Table 1⁸ the chalk rating value of each tape to the nearest 0.5 unit. Record the rating on the worksheet or other form. The worksheet form (Fig. 4),

inserted into the plastic sheet protector with a black background gives a clear permanent record of these measurements and evaluations.

8.2.6 These tape chalks may also be compared to Photographic Reference Standard No. 2 as an alternative procedure.

8.3 Test Method E-TNO Method:

8.3.1 Materials:

8.3.1.1 *Photographic Reference Standard No. 2* for the determination of chalking, consisting of a photograph of five strips of tape mounted on a black background, numbered 0, 2, 4, 6, and 8, and varying in this order from white to almost black. The numerical rating of chalking shown on the photographic reference standard is opposite of the original TNO Scale.

8.3.1.2 *Polyethylene Tape*,⁹ transparent, 1-in. (25-mm) wide.

8.3.1.3 *Black Velvet*, dull black with a short pile and without a tendency to crush, size approximately 8 by 12 in. (200 by 300 mm), mounted on a flat substrate. Place adjacent to the standard for the ratings.

8.3.2 *Procedure*—Apply a piece of tape, approximately 4-in. (100-mm) long, to the coating by uniform gentle pressure of the finger, remove the tape, and lay it with the adhesive side on the piece of velvet. Under diffused light compare the tape on the black velvet with Photographic Reference Standard No. 2, and determine which of the five grades most closely matches the whiteness of the adhering pigment. If the degree of chalking is obviously between two adjacent grades, select the intermediate odd number as the chalk rating.

8.3.3 Chalk ratings may also be determined by following the procedures of 8.2.3 and comparing to the values shown in Table 1. The use of the worksheet form shown in Figs. 3 and 4 may be used as a permanent record.

9. Recommendations

9.1 The procedures recommended for various substrates and coatings are based upon the results obtained in interlaboratory testing. The selection of the method to be used is subject to agreement between producer and user.

10. Report

10.1 A record of the test method used, the rating, panel number, and other pertinent information must be clearly shown on the inspection report for each evaluation. The report form may be in accordance with Standard D 1150 or other form agreed upon between the producer and the user.

10.2 The pertinent information should include: date of inspection, date of exposure start, purchase order number of testing organization, duration, remarks about unusual weather, etc., the name of the person making the inspection, and other information agreed upon between the producer and the seller.

⁹Sellotape 1401, Transparent Polythene Electrical, manufactured by Sellotape Products Ltd., Edgware, Middlesex, England has been found suitable for this purpose.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Practice for Qualification of Journeyman Painters for Application of Coatings to Concrete Surfaces of Safety-Related Areas in Nuclear Facilities¹

This standard is issued under the fixed designation D 4227; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This practice provides a standard qualifying method for journeyman painters to verify their proficiency and to attain the required quality for application of specified coatings to concrete surfaces in safety-related areas in a nuclear facility.

1.2 Variations or simplifications of the practice set forth herein may be appropriate for special coating work such as maintenance. It is not the intent of this practice to mandate a regular basis for all qualifications.

1.3 Evaluation of the journeyman painter being qualified in accordance with this practice, shall be by qualified agents as specified in 4.1. Reports shall be prepared as specified in Section 5, and qualification as specified in Section 6.

1.4 It is the intent of this practice to judge only the ability of the journeyman painter to apply specified coatings with proper tools and equipment.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Definitions

2.1 *journeyman painter*—an individual who has worked in the painting trade sufficiently long enough to master the use of all applicable tools and the materials being applied.

2.2 *qualifying agents*—the designated representatives of the owner or of the coating organization, or both, who have sufficient experience in the practical application and evaluation of coating applied to concrete surfaces of a nuclear facility.

2.3 *governing documents*—technical specifications, jobsite procedures, and reference documents.

Application of Coatings

3.1 This practice requires the journeyman painter to apply the specified coating in conformance to the governing documents to a test area similar to that detailed in Figs. 1 and 2.

3.1.1 *Ceiling Section*—Beginning at the wall line, one 5 by 5-ft (1.5 by 1.5-m) coating.

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.07 on Application. Current edition approved April 6, 1983. Published June 1983.

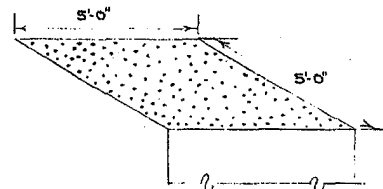


FIG. 1 Concrete Ceiling Test Area

3.1.2 *Wall Section*—Beginning at the floor line, one 5 by 5-ft (1.5 by 1.5-m) coating.

3.1.3 *Floor Section*—Beginning at the wall line and connecting with the wall section, one 5 by 5-ft (1.5 by 1.5-m) coating.

3.2 This practice requires the journeyman painter perform the application using the proper technique and application equipment consistent with the governing documents.

3.3 The surfaces of the concrete test area shall be prepared in accordance with the governing documents.

3.4 The journeyman painter shall demonstrate the ability to apply the specified coating to a uniform dry film thickness in accordance with the governing documents, as evaluated by the qualifying agents.

3.5 When desired by the owner, one of the test surfaces may include embedded steel.

3.6 If a coating system specified requires a primer, intermediate, and finish coat, treat each coat as a separate application, allowing a specified drying time before applying each succeeding coat.

3.7 The journeyman painter shall be provided with the following:

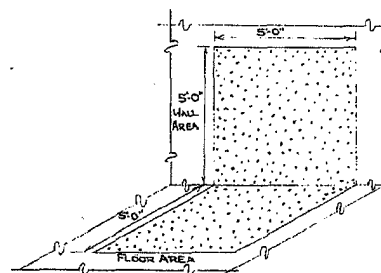


FIG. 2 Concrete Wall and Floor Test Area

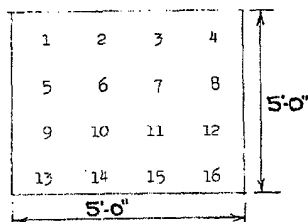


FIG. 3 Location of Wet-Film Thickness Readings

3.7.1 Information regarding the specified coating material(s), including wet and dry film thickness required, and all other information contained in the governing documents for the coating system being applied.

3.7.2 Coating materials properly mixed in accordance with the governing documents and ready for application.

3.7.3 The necessary equipment for the proper application of the specified coating.

3.7.4 Miscellaneous equipment, brushes, and thinners required for cleaning the equipment after completion of the test.

3.7.5 A practice area to adjust and test the equipment prior to performing the test.

3.7.6 All necessary safety equipment.

3.7.7 Wet-film thickness gage for verifying the coating thickness during application.

3.8 The journeyman painter shall use the wet-film thickness gage furnished to determine coating thickness buildup during application. Material thickness is one of the criteria for evaluation.

4. Evaluation of Coating Application

4.1 Evaluation of the journeyman painter shall be made

by two qualifying agents. Only one qualifying agent can be production-related.

4.2 The qualifying agents shall be capable of answering technical questions requested by the journeyman painter relating to the application of the specified coating material(s). The qualifying agents shall be thoroughly familiar with the specified coating material(s) and acceptance criteria and shall be aware of any difficulties in applying the coating to any surface.

4.3 The qualifying agents shall have a wet-film gage of the same type used by the journeyman painter.

4.4 The qualifying agents shall take approximately 16 wet-film thickness readings on the test areas. These readings shall be recorded as specified in Section 5. The wet-film thickness readings shall be used to verify the specified dry-film thickness requirements and uniformity of application. The number and location of readings shall be as indicated on Fig. 3.

4.5 The journeyman painter and the qualifying agents shall understand the required dry-film thickness range requirements of the governing documents before any coatings are applied.

4.6 The qualifying agent shall inspect the finished surface to verify that it conforms to the requirements of the governing documents. A description of the appearance of the completed applied coating shall be recorded on the form shown in Fig. 4.

5. Report

5.1 The qualifying agents shall use a report form similar to that in Fig. 4 to record wet-film thickness readings specified in 4.4, and the appearance of the completed coating surface as specified in 4.6.

5.2 The qualifying agents and journeyman painter shall sign the report form.

Test Application of Coating to Standard Concrete Test Areas by Journeyman Painter for Qualification for Coating of Concrete Surfaces in Safety-Related Areas of Nuclear Facilities

Type of Recording Instrument for Wet-Film (WFT) Thickness: _____

WFT Reading No.	Floor Test Area	Wall Test Area	Ceiling Test Area
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			
15.			
16.			

Description of Materials: _____

Qualifying Agent: _____
 Qualifying Agent: _____
 Date: _____ Journeyman: _____

FIG. 4 Record Form for Test Application

Representing: _____
Representing: _____

ing System:
Manufacturer:

Manufacturer: _____
Type of Application Equipment: _____
Comments: _____

Comments:

The above applicant has been qualified as a Journeyman Painter for the application of the above coatings for concrete surfaces of safety-related areas of nuclear facilities.

Qualifying Agent: _____

Qualifying Agent: _____

Journeyman: _____

FIG. 5 Qualification Form

3. A copy of the report shall become a permanent part of the quality assurance file for the nuclear project for which the journeyman painter is qualifying.

4. A copy of the report shall be given to the qualifying journeyman painter.

Initial Qualification

7.4.1 The qualifying agents shall prepare a qualification form similar to Fig. 5. The qualification form shall state the coating materials used and the application equipment used in the test.

5.2 The qualifying agents and the journeyman painter shall sign the qualification form.

6.3. A copy of the qualification form shall become a permanent part of the quality assurance file for the nuclear project for which the journeyman painter is qualified.

6.4 A copy of qualification form shall be given to the journeyman painter.

7. Regualification

7.1 The owner or his designated representative may determine the degree of requalification to be permitted, as well as the acceptance of previously qualified journeyman painters based on time interval since the date of the qualification and provided the same coating materials and application procedures are used. Note the acceptance of previous qualification in the quality assurance file for the project.

8. Limited Qualifications—Touch Up and Repair

8.1 Limited qualifications can be accomplished using only areas representative of the actual plant surfaces, even though they do not include all areas represented by Figs. 1 and 2.

9. Limited Qualifications—Brushes, Rollers, Squeegees, or Other Special Tools

9.1 Limited qualifications for special application tools may be required on certain projects. When such limited qualification is performed, the limitations shall be noted on the records for qualification and application.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Qualification of Journeyman Painters for Application of Coatings to Steel Surfaces of Safety-Related Areas in Nuclear Facilities¹

This standard is issued under the fixed designation D 4228; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice provides a standard qualifying method for journeyman painters to verify their proficiency and ability to attain the required quality for application of specified coatings to steel surfaces in safety-related areas in a nuclear facility.

1.2 Variations or simplifications of the practice set forth herein may be appropriate for special coating work such as maintenance or qualifications of equipment suppliers shop personnel. It is not the intent of this practice to mandate a singular basis for all qualifications.

1.3 Evaluation of the journeyman painter being qualified in accordance with this practice, shall be by qualified agents as specified in 4.1. Reports shall be prepared as specified in Section 5, and certification as specified in Section 6.

1.4 It is the intent of this practice to judge only the ability of the journeyman painter to apply specified coatings with the proper tools and equipment.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Definitions

2.1 *journeyman painter*—an individual who has worked in the painting trade sufficiently long enough to master the use of all applicable tools and the materials being applied.

2.2 *qualifying agents*—the designated representatives of the owner or of the coating organization, or both, who have sufficient experience in the practical application and evaluation of coatings applied to steel surfaces of a nuclear facility.

2.3 *governing documents*—technical specifications, jobsite procedures, and reference documents.

3. Application of Coatings

3.1 This practice requires the journeyman painter to apply the specified coating in conformance to the governing documents to a test panel similar to that detailed in Fig. 1. This panel is typical of the panel used for qualification, and may be modified to suit site configurations.

3.2 This practice requires the journeyman painter to

perform the application using the proper technique and application equipment consistent with the specified coating materials.

3.3 The surfaces of the test panel shall be prepared in accordance with the governing documents.

3.4 Place the test panel approximately 12 in. (305 mm) above ground level and at approximately 30° from the vertical plane, with the complex side up. This will simulate the types of difficult coating situations encountered by journeyman painters in a nuclear facility.

3.5 The journeyman painter shall demonstrate his ability to apply the specified coating to a uniform dry-film thickness in accordance with the governing documents, as evaluated by the qualifying agents.

3.6 Allow required drying as prescribed by the governing documents prior to taking the dry-film thickness reading of the applied coating. If a coating system specified requires a primer, intermediate, and finish coat, treat each coat as a separate application, allowing a specified drying time before inspecting each completed coat.

3.7 The journeyman painter shall be provided with the following:

3.7.1 Information regarding the specified coating material(s), including wet and dry film thickness required, and all other information contained in the governing documents for the coating system being applied.

3.7.2 Coating materials properly mixed in accordance with the governing documents and ready for application.

3.7.3 The necessary equipment for the proper application of the specified coating.

3.7.4 Miscellaneous equipment, brushes, and thinners required for cleaning the equipment after completion of the test.

3.7.5 A practice area to adjust and test the equipment prior to performing the test.

3.7.6 All necessary safety equipment.

3.7.7 Wet-film thickness gage for testing the coating thickness during application, where applicable.

4. Evaluation of Coating Application

4.1 Evaluation of the journeyman painter shall be made by two qualifying agents. Only one qualifying agent can be production-related.

4.2 The qualifying agents shall be capable of answering technical questions requested by the journeyman painter relating to the application of the specified coating material(s). The qualifying agents shall be thoroughly familiar with the specified coating material(s) and acceptance criteria and shall

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.07 on Application.

Current edition approved April 6, 1983. Published June 1983.

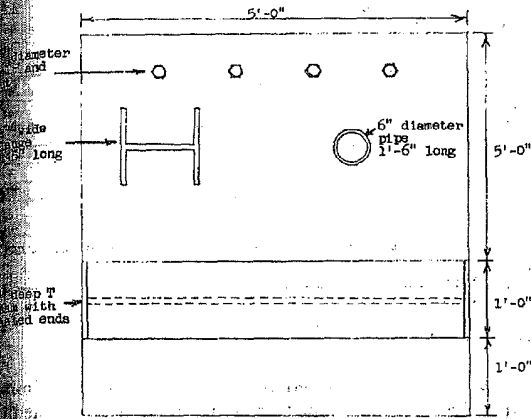


FIG. 1 Test Panel—Complex Side

aware of any difficulties in applying the coating to any surface.

4.3 The qualifying agents shall have a wet-film gage of the same type used by the journeyman painter, as well as a calibrated magnetic-type dry-film thickness gage.

4.4 The qualifying agents shall take dry-film thickness readings on all areas of the test panel, except nuts and bolts. These readings shall be recorded as specified in Section 5. Dry-film readings may be used to check the journeyman painter's progress during application. The dry-film thickness reading shall be used to verify the specified dry-film thickness requirements and uniformity of application. The number and location of readings shall be as indicated on Fig.

4.5 The journeyman painter and the qualifying agents shall understand the required dry-film thickness range requirements of the governing documents before any coatings are applied.

4.6 The qualifying agent shall inspect the finished surface to verify that it conforms to the requirements of the governing documents. A description of the appearance of the

completed applied coating and dry film thickness shall be recorded on form Fig. 3.

5. Report

5.1 The qualifying agents shall use a report form similar to that in Fig. 2 to record dry-film thickness readings specified in 4.4, and the appearance of the completed coating surface as specified in 4.6.

5.2 The qualifying agents and journeyman painter shall sign the report form.

5.3 A copy of the report shall become a permanent part of the quality assurance file for the nuclear project for which the journeyman painter is qualifying.

5.4 A copy of the report shall be given to the qualifying journeyman painter.

6. Initial Qualification

6.1 The qualifying agents shall prepare a form similar to Fig. 3. The qualification form shall state the coating materials used and the application equipment used in the test.

6.2 The qualifying agents and the journeyman painter shall sign the qualification form.

6.3 A copy of the qualification form shall become a permanent part of the quality assurance file for the nuclear project for which the journeyman painter is qualified.

6.4 A copy of the qualification form shall be given to the journeyman painter.

7. Requalification

7.1 The owner or his designated representative may determine the degree of requalification to be permitted, as well as the acceptance of previously qualified journeyman painters based on time interval since the date of the qualification form and provided the same coating materials and application procedures are used. Note the acceptance of previous qualification in the quality assurance file or the project.

8. Limited Qualifications—Touch Up and Repair

8.1 Limited qualifications can be accomplished using only areas representative of the actual plant surfaces, even though they do not include all areas represented in Fig. 1.

Test Application of Coating to Standard Steel Test Panel by Journeyman Painter for Qualification for Coating of Steel Surfaces in Safety-Related Areas of Nuclear Facilities

Journeyman:	Readings	Wet	Dry
Outside:			
Complex Side:			
Top:			
Beam:			
Bar:			
Date:			
Description of Finished Surface:			
Date:		Qualifying Agent:	
Material:		Qualifying Agent:	
DFT Range:		Journeyman:	

FIG. 2 Record Form for Test Application



D 4228

Journeyman Painter Qualification for Application of Coatings to Steel Surfaces of Safety-Related Areas of Nuclear Facilities

Journeyman's Name: _____
Location (Site): _____

Qualifying Agents:
1. _____ Representing: _____
2. _____ Representing: _____

Coating System: _____
Manufacturer: _____
Type of Application Equipment: _____
Comments: _____

The above applicant has been certified as a Journeyman Painter for the application of the above coatings for steel surfaces of safety-related areas of nuclear facilities.

Date: _____ Qualifying Agent: _____
Journeyman: _____ Qualifying Agent: _____

FIG. 3 Qualification Form

9. Limited Qualifications—Brushes, Rollers, or, Other Special Tools

may be required on certain projects. When such limited qualification is performed, the limitations shall be noted on the records for qualification and application.

9.1 Limited qualifications for special application tools

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Labeling Art Materials for Chronic Health Hazards¹

This standard is issued under the fixed designation D 4236; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Uninformed or careless use of some art material products can give rise to health hazards, either acute or chronic, or both. Specific and readily available warnings are needed to help protect users of any age. One way to disseminate such information is to provide appropriate precautionary labeling on art material products.

Labeling for acute health hazards, including those associated with art materials, is being addressed by such requirements as the U.S. Consumer-Product Safety Act, the Federal Hazardous Substances Act, and the like. There are presently no specific national standards for labeling art materials with respect to chronic health hazards.

This practice is intended to provide a standard for developing precautionary labels concerning chronic health hazards related to the use of art materials. It is further intended to have the adaptability necessary to keep labels current with existing scientific and medical knowledge, as well as in conformity with other precautionary labeling requirements, both acute and chronic, thereby avoiding unnecessary confusion by users with respect to other precautionary labeling.

Scope

1.1 This practice describes a procedure for developing precautionary labels for art materials and provides hazard and precautionary statements based upon knowledge that exists in the scientific and medical communities. This practice concerns those chronic health hazards known to be associated with a product or product component(s), when the component(s) is present in a physical form, volume, or concentration that in the opinion of a toxicologist (see 2.1.11) has the potential to produce a chronic adverse health effect(s).

1.2 This practice applies exclusively to art materials packaged in sizes intended for individual users of any age or those participating in a small group.

1.3 Labeling determinations shall consider reasonable foreseeable use or misuse. The responsibility for precautionary labeling rests with the producer or repackager who markets the materials for art or craft use.

1.4 This practice does not specify test methods for determining whether a substance or product presents chronic health hazards.

1.5 This practice does not apply to products appropriately labeled for known chronic health hazards in accordance with chemical substance labeling standards and practices, such as another national consensus standard, existing labeling statutes, regulations, or guidelines.

1.6 Since knowledge about chronic health hazards is incomplete and warnings cannot cover all uses of any product, it is not possible for precautionary labeling to ensure

completely safe use of an art product.

1.7 Manufacturers or repackagers may wish to determine individually or collectively precautionary labeling for art materials in accordance with this practice. Compliance may be certified by a certifying organization. Guidelines for a certifying organization are given in Appendix X1.

1.8 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 *Descriptions of Terms Specific to This Standard:*

2.1.1 *analytical laboratory*—a laboratory having personnel and apparatus capable of performing quantitative or qualitative analyses of art materials, which may yield information that is used by a toxicologist for evaluation of potentially hazardous materials.

2.1.2 *art material or art material product*—any raw or processed material, or manufactured product, marketed or represented by the producer or repackager as intended for and suitable for users as defined herein.

2.1.3 *bioavailability*—the extent that a substance can be absorbed in a biologically active form.

2.1.4 *chronic adverse health effect(s)*—a persistent toxic effect(s) that develops over time from a single, prolonged, or repeated exposure to a substance. This effect may result from exposure(s) to a substance that can, in humans, cause sterility, birth defects, harm to a developing fetus or to a nursing infant, cancer, allergenic sensitization, damage to the nervous system, or a persistent adverse effect to any other organ system.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 4236 - 83. Last previous edition D 4236 - 89.

2.1.5 *chronic health hazard(s)* (hereafter referred to as "*chronic hazard*")—a health risk to humans, resultant from exposure to a substance that may cause a chronic adverse health effect.

2.1.6 *label*—a display of written, printed, or graphic matter upon the immediate container of any art material product. When the product is unpackaged, or is not packaged in an immediate container intended or suitable for delivery to users, the label can be a display of such matter directly upon the article involved or upon a tag or other suitable labeling device attached to the art material.

2.1.7 *producer*—the person or entity who manufactures, processes, or imports an art material.

2.1.8 *repackager*—the person or entity who obtains materials from producers and without making changes in such materials puts them in containers intended for sale as art materials to users.

2.1.9 *sensitizer*—a substance known to cause, through an allergic process, a chronic adverse health effect which becomes evident in a significant number of people on re-exposure to the same substance.

2.1.10 *toxic*—applies to any substance that is likely to produce personal injury or illness to humans through ingestion, inhalation, or skin contact.

2.1.11 *toxicologist*—an individual who through education, training, and experience has expertise in the field of toxicology, as it relates to human exposure, and is either a toxicologist or physician certified by a nationally recognized certification board.

2.1.12 *users*—artists or crafts people, of any age, who create, or recreate in a limited number, largely by hand, works which may or may not have a practical use, but in which aesthetic considerations are paramount.

3. Requirements

3.1 To conform to this voluntary practice the producer or repackager of art materials shall submit art material product formulation(s) or reformulation(s) to a toxicologist for review, such review to be in accordance with Section 4 of this practice. The toxicologist shall be required to keep product formulation(s) confidential.

3.1.1 Unless otherwise agreed in writing by the producer or repackager, no one other than the toxicologist shall have access to the formulation(s); except that the toxicologist shall furnish a patient's physician, on a confidential basis, the information necessary to diagnose or treat cases of exposure or accidental ingestion.

3.2 To conform to this practice, the producer or repackager, upon advice given by a toxicologist in accordance with Section 4 of this practice, shall adopt precautionary labeling in accordance with Section 5 of this practice and based upon generally accepted, well-established evidence that a component substance(s) is known to cause chronic adverse health effects.

3.3 To conform to this practice, labeling shall be parallel to, conform to, and minimally include any labeling practices prescribed by U.S. federal and state statutes or regulations and shall not diminish the effect of required acute toxicity warnings.

3.4 To conform to this practice, the producer or repackager shall supply a poison exposure management informa-

tion source² the generic formulation information required for dissemination to poison control centers or provide a 24-h cost-free telephone number to poison control centers.

3.5 To conform to this practice, the producer or repackager shall have a toxicologist review as necessary, but at least every 5 years, art material product formulation(s) and associated label(s) based upon the then current, generally accepted, well-established scientific knowledge.

3.6 *Statement of Conformance*—"Conforms to ASTM Practice D 4236," or "Conforms to ASTM D 4236," or "Conforms to the health requirements of ASTM D 4236." This statement may be combined with other conformance statements. The purpose of the conformance statement is to inform the purchaser, at the time of purchase, of the product's compliance with the standard. To accomplish this purpose the conformance statement should appear whenever practical on the product; however, it shall also be acceptable to place the statement on one or more of the following: (a) the individual product package, (b) a display or sign at the point of purchase, (c) separate explanatory literature available on request at the point of purchase, (d) a response to a formal request for bid or proposal.

4. Determination of Labeling

4.1 An art material is considered to have the potential for producing chronic adverse health effects if any customary or reasonably foreseeable use can result in a chronic hazard.

4.2 In making the determination a toxicologist(s) shall take into account the following:

4.2.1 Current chemical composition of the art material, supplied by an analytical laboratory or by an industrial chemist on behalf of a manufacturer or repackager.

4.2.2 Current generally accepted, well-established scientific knowledge of the chronic toxic potential of each component and the total formulation.

4.2.3 Specific physical and chemical form of the art material product, bioavailability, concentration, and the amount of each potentially chronic toxic component found in the formulation.

4.2.4 Reasonably foreseeable uses of the art material product as determined by consultation with users and other individuals who are experienced in use of the material(s), such as teachers, or by market studies, unless such use information has previously been determined with respect to the specific art material(s) under review.

4.2.5 Potential for known synergism and antagonism of the various components of the formulation.

4.2.6 Potentially chronic adverse health effects of decomposition or combustion products, if known, from any reasonably foreseeable use of the hazardous art material product.

4.2.7 Opinions of various regulatory agencies and scientific bodies, including the International Agency for Research on Cancer and the National Cancer Institute, on the potential for chronic adverse health effects of the various components of the formulation.

² Two of the larger poison exposure management information sources are: The Rocky Mountain Poison Control Center, West 8th and Cherokee, Denver, CO 80204; and the National Poison Center Network, 125 De Soto St., Pittsburgh, PA 15213.

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3 Based upon the conclusion reached in conformance with review determinations set forth herein the toxicologist(s) shall recommend precautionary labeling consistent with Section 5 of this practice.

Labeling Practices

5.1 Signal Word:

5.1.1 When a signal word for an acute hazard(s) is mandated and a chronic hazard(s) exists, the signal word shall be thht for the acute hazard.

5.1.2 When only a chronic hazard(s) exists, the signal word WARNING shall be used.

5.1.3 The signal word shall be prominently visible and set in bold capitals in a size equal to or greater than the statement of potential chronic hazards.

5.2 *List of Potentially Chronic Hazards*—Potentially chronic hazards, as determined under the procedures of Section 4, shall be stated substantially in accordance with the statements listed in Annex A1 of this practice. Potentially chronic hazards noted shall be those that are clinically significant and that might be expected with any reasonably foreseeable use of the art material. The hazards should be grouped in the order of relative descending severity.

5.3 *Name of Chronically Hazardous Component(s)*—All components and known decomposition products of the formulation with a potential for chronic hazards, as determined under the procedures of Section 4, shall be listed prominently. Generically equivalent names may be used.

5.4 *Safe Handling Instructions*—Appropriate precautionary statements as to work practices, personal protection, and ventilation requirements shall be used substantially conforming with those listed in Annex A2 of this practice.

5.5 *List of Sensitizing Components*—To protect users from known sensitizers found within art materials, each label shall contain a list of those sensitizers present in sufficient amounts to contribute significantly to a known skin or respiratory sensitization.

5.6 *Combined Statements*—If an art material contains more than one component capable of causing a chronic adverse health effect, or if a single chemical can cause several different chronic adverse health effects, the potential effects may be combined into one statement.

5.7 *Information Sources*—In addition to an appropriate telephone number, the precautionary label shall contain a statement identifying a source for additional health informa-

tion substantially in conformance with one of the phrases listed below:

5.7.1 For more health information—(24-h cost-free telephone number), or

5.7.2 For further health information call a poison control center.

5.8 *Labeling Content, Product Size*—An art material product(s) in a container larger in size than one fluid ounce (30 mL) (if the product is sold by volume) or one ounce net weight (28 g) (if the product is sold by weight) shall have full precautionary labeling, as generally described in Section 5 of this practice. An art material product(s) in a container equal to or smaller than one fluid ounce or one ounce net weight shall have a label that includes a signal word in conformance with 5.1 of this practice and a list of potentially harmful or sensitizing components in conformance with 5.3 and 5.5 of this practice.

5.9 The information described in Section 5 must appear (1) on the outside container or wrapper, if any, unless it is easily legible through the outside container or wrapper and (2) on all accompanying literature where there are directions for use, written or otherwise. In a case where one or more individual product(s), which require warning labels under Sections 4 and 5, are packed within a point of sale package which obscures the warning statement(s), the point of sale package shall carry the signal word conforming to 5.1 of this practice and the following wording: "Contains: (list hazardous product(s)) that may be harmful if misused. Read cautions on individual containers carefully. Keep out of the reach of children."

5.10 Statements required under Sections 4 and 5 must be in the English language and located prominently in conspicuous and legible type in contrast by topography, layout, or color with other printed matter on the label.

5.11 *Supplemental Information*—Where appropriate, more detailed technical information that relates to chronic hazard(s), such as physical properties, decomposition products, detailed safety instructions, or disposal recommendations, shall be included in supplemental documents, such as Material Safety Data Sheets, technical brochures, technical data sheets etc.

6. Keywords

6.1 art materials; chronic toxicity; craft materials; health labeling; precautionary statements; warning statements

ANNEXES

(Mandatory Information)

A1. CHRONIC HAZARD STATEMENTS

MAY CAUSE STERILITY.
CONTACT MAY CAUSE PERMANENT EYE DAMAGE.
MAY BE HARMFUL BY BREATHING VAPORS/DUSTS.
MAY BE HARMFUL IF SWALLOWED.
MAY BE HARMFUL BY SKIN CONTACT.

MAY PRODUCE BIRTH DEFECTS IN THE DEVELOPING FETUS.
MAY BE EXCRETED IN HUMAN MILK.
MAY CAUSE HARM TO THE NURSING INFANT.
CANCER AGENT! EXPOSURE MAY PRODUCE CANCER.

CANCER AGENT BASED ON TESTS WITH LABORATORY ANIMALS.

POSSIBLE CANCER AGENT BASED ON TESTS WITH LABORATORY ANIMALS.

MAY PRODUCE ALLERGIC REACTION BY INGESTION/INHALATION/SKIN CONTACT.

MAY PRODUCE NUMBNESS OR WEAKNESS IN THE EXTREMITIES.

EXPOSURE MAY CAUSE (SPECIFY THE ORGAN(S)) DAMAGE.

HEATING/COMBUSTION MAY CAUSE HAZARDOUS DECOMPOSITION PRODUCTS.

MAY CAUSE (SPECIFIC EFFECT) OF (SPECIFY ORGAN).

CANCER AGENT! EXPOSURE BY (SPECIFIC ROUTE) MAY PRODUCE CANCER.

MAY CAUSE HARM TO THE DEVELOPING FETUS. EXPOSURE MAY RESULT IN NAUSEA, HEADACHE, CONFUSION OR INSTABILITY.

MAY CAUSE DAMAGE TO RED BLOOD CELLS WITH REDUCED ABILITY TO CARRY OXYGEN.

A2. PRECAUTIONARY STATEMENTS

Keep out of reach of children.
When using do not eat, drink, or smoke.
Wash hands immediately after use.
Avoid inhalation/ingestion/skin contact.
Avoid fumes from combustion.
Keep container tightly closed when not in use.
Store in well-ventilated area.
Wear protective clothing (specify type).
Wear protective goggles/face shield.
Wear NIOSH³-certified mask for dusts/mists/fumes.
Wear NIOSH-certified respirator with an appropriate cartridge for (specify).
Wear NIOSH-certified supplied-air respirator.

Use window exhaust fan to remove vapors and ensure adequate cross ventilation. (Specify explosion-proof if necessary.)

Do not heat above (specify temperature) without adequate ventilation.

Use (specify type) local exhausting hood.

Do not use/mix with (specify material).

Do not dry grind.

Do not spray apply.

If pregnant or contemplating pregnancy, use only under professional supervision.

Avoid using/do not use if pregnant (or contemplating pregnancy).

Not for use by children.

Avoid breathing dust.

Use in glove box.

³ U. S. National Institute of Occupational Safety and Health.

APPENDIX

(Nonmandatory Information)

X1. GUIDELINES FOR A CERTIFYING ORGANIZATION

X1.1 The term certifying organization, as used in these guidelines, refers to an organization or an institute that, after assuring that all provisions are met, certifies that an art material does conform to the labeling requirements of this practice.

X1.2 The certifying body may be funded by member manufacturers, but should include users or their representatives, as well as manufacturers' chemists, on its technical and certifying committees.

X1.3 Representative samples of art materials, labeled as conforming to this practice and bought at retail, should be analyzed at random and from time to time by an analytical laboratory to ensure they are the same as the formulation

used by the toxicologist(s) for determining labeling requirements.

X1.4 The methods used by the toxicologist(s) in review and determination of the need and content of precautionary labeling for potentially chronic adverse health effects should be periodically reviewed by an advisory board composed of not less than three or more than five toxicologists, at least one of whom is certified in toxicology by a nationally recognized certification board.

X1.5 In cases where there is disagreement by participating producers or participating users, with the determination of the toxicologist(s), there should be a method whereby the toxicologist's decision can be presented to the advisory board of toxicologists for arbitration.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



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Standard Test Method for Determination of the Decontaminability of Coatings Used in Light-Water Nuclear Power Plants¹

This standard is issued under the fixed designation D 4256; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

Scope

1.1 This test method establishes procedures for determining and ranking the ease of decontaminability of coatings for use in nuclear power plants.

1.2 This standard defines two different test procedures involving differing contaminant contact times and decontaminating media. Procedure A involves a relatively brief radionuclide solution contact time and decontamination using both demineralized water and acid solutions. Procedure B utilizes a seven-day contamination solution contact time and decontamination with ambient temperature water only. Procedure B is more in line with power plant decontamination practices.

1.3 Users of this standard should specify either Procedure A or Procedure B, or both, based upon specific power plant conditions and use requirements.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²
E 522 Method of Calibrating Germanium Detectors for Measurement of Gamma-Ray Emission Rates of Radionuclides³
- 2.2 *American National Standards Institute:*⁴
N512 Protective Coatings (Paint) for the Nuclear Industry

Terminology

- 3.1 *Definitions:*
3.1.1 *decontaminate*—the removal of radionuclides from test panel using prescribed procedures.
3.1.2 *decontamination factor (DF)*—the ratio of the original radioactivity (dpm) to the radioactivity detected after treatment.

¹ This test method is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.11 on Decontamination of Coated Surfaces.

² Current edition approved Feb. 24, 1989. Published June 1989. Originally published as D 4256 - 83. Last previous edition D 4256 - 83.

³ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁴ Discontinued—see 1983 *Annual Book of ASTM Standards*, Vol 12.02.

⁵ Available from American National Standards Institute, 11 W. 42nd St., 13th floor, New York, NY 10036.

3.1.3 *disintegration of atomic nuclei*—the nuclear transformation characterized by the emission of one or more particles or photons from the nucleus.

3.1.4 *disintegrations per minute (dpm)*—the number of disintegrations occurring in a time period of 1 min.

4. Significance and Use

4.1 Day-to-day operation or a loss-of-coolant accident at a nuclear power plant can give rise to a significant level of radioactive contamination on the walls, floors, and attendant equipment. The ease with which the coated surfaces can be decontaminated could result in considerable reduction in the level of radioactive contamination, thereby mitigating the exposure to plant operating personnel. It is recognized that this test method does not duplicate actual field decontamination procedures, but it does represent comparative test procedures that have been in use for many years. Other tests, designed to be more meaningful in terms of real-life conditions, are under development.

5. Apparatus

5.1 *Radiation Detection Equipment*⁵—Use a gamma-sensitive system composed of the following items:

5.1.1 *Detector*—Use a germanium crystal detector with a resolution of 2.5 keV at 1332 keV or better.

NOTE—See Method E 522 for calibration methods.

5.1.2 *Photomultiplier-Amplifier*.⁶

5.1.3 *Analog-to-Digital Converter (ADC)*.⁷

5.1.4 *Pulse-Height Analyzer*, capable of resolving simple gamma-ray peaks shall be used.

5.2 Mount test specimens so as to give an optimum scanning geometry of the contaminated surface.

5.3 Shield the detector to eliminate excessive background counts.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chem-

⁵ The most up-to-date information on gamma-ray detectors and accompanying electronics is found in manufacturers' literature. A list of vendors of this equipment can be found in compilations such as the *Guide to Scientific Instruments*, published by the American Association for the Advancement of Science, Washington, DC.

⁶ Teknec Model TC-205, or equivalent, has been found satisfactory for this purpose.

⁷ Nuclear Data Model ND570, or equivalent, has been found satisfactory for this purpose.

ical Society, where such specifications are available.⁸ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent grade water conforming to Specification D 1193.

6.3 Contaminating Solution:

6.3.1 Use a radioactive contaminant solution of mixed fission products of cobalt-60 and cesium-137, prepared in 8 M nitric acid (HNO₃) and stored in a polyethylene bottle.

6.3.2 The solution shall have a minimum beta-gamma activity of 5×10^6 disintegrations per minute (dpm) per 0.1 mL.

6.4 *Decontaminating Solutions*—The following decontaminating solutions shall be utilized, based upon the procedure selected (Procedure A or Procedure B):

6.4.1 An aqueous mixture of 0.4 M oxalic acid (COOH)₂, 0.05 M sodium fluoride (NaF), and 0.3 M hydrogen peroxide (H₂O₂).

6.4.2 Demineralized water.

7. Preparation of Test Specimens

7.1 *Steel Panels*—Prepare panels in accordance with ANSI N512.

7.2 *Concrete Blocks*—Prepare blocks in accordance with ANSI N512.

8. Test Specimens

8.1 Use a minimum of two test specimens for each coating system.

9. Specimen Contamination

9.1 Degrease the test specimens using the coating manufacturer's approved solvent; place on a level surface inside a laboratory hood and at $24 \pm 3^\circ\text{C}$ until the solvent has evaporated. Maintain a minimum air velocity of 150 ft/min at the face of the hood.

9.2 Transfer a portion of the contaminating solution to a clean polyethylene bottle and immediately adjust to pH 4 with 8 M ammonium hydroxide (NaOH).

9.3 Contamination Procedure A, Evaporate to Dryness:

9.3.1 Immediately transfer a 0.2-mL portion of the pH-adjusted solution to the center of the lower 2-in. section of the test specimen so as to cover an area about 1 cm in diameter.

9.3.2 Allow the specimen to air dry as in 9.1, and place it in a plastic bag approximately 1.5-mil thick.

9.3.3 Position the specimen and bag in the detector chamber and scan, using identical geometry as in 8.2. Record all counts.

9.4 Contamination Procedure B, Seven-Day Contact Time:

9.4.1 Perform contamination using the assembly shown in Fig. 1.

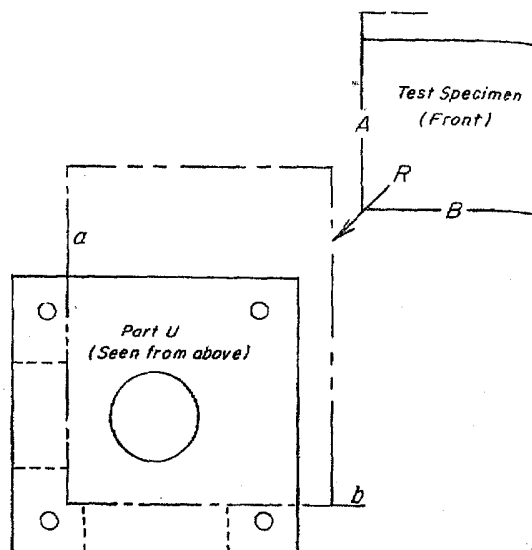


FIG. 1 Top View of Holder for Contamination of Test Specimen in Seven-Day Exposure (Procedure B)

9.4.2 A test specimen is pushed into the positioning aid with its reference corner *R* as shown in Fig. 2. Edges *A* and *B* of the test specimen are brought into close contact with Lines *a* and *b* of the positioning aid, the Contact *B-b* having priority. The test specimen is then pressed against Part *U* containing the silicone rubber ring by fastening the screws until the test specimen is in contact with Part *U* along all of its edges.

9.4.3 The test specimens are contaminated by applying 1 mL of the contaminant solution to the middle of the circle visible through Part *U*.⁸ The holder shall be kept in a horizontal position. If, in the case of highly hydrophobic surface materials, the contaminant does not immediately cover the test circle, the holder may be tilted to enforce sufficient spread of the liquid.

9.4.4 A small petri dish is placed over the test circle on top of Part *U* to prevent evaporation of the contaminant solution.

9.4.5 After seven-days contact time the contaminant solution is sucked off as completely as possible using a pipet. For this purpose it is necessary to tilt the holder. The holder is then inverted. After removing the nuts and Part *L*, lift the test specimen vertically so that distribution of contaminant solution to uncontaminated parts of the test specimen is prevented.

9.4.6 To avoid evaporation effects immediately place the test specimen in a plastic bag. The whole procedure of removing the specimens from their holders and packaging them shall not take more than 12 min.

9.4.7 Position the contaminated specimens and scan as in 9.3.3.

10. Decontamination Procedure

10.1 The decontamination process for Procedure A will

⁸ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

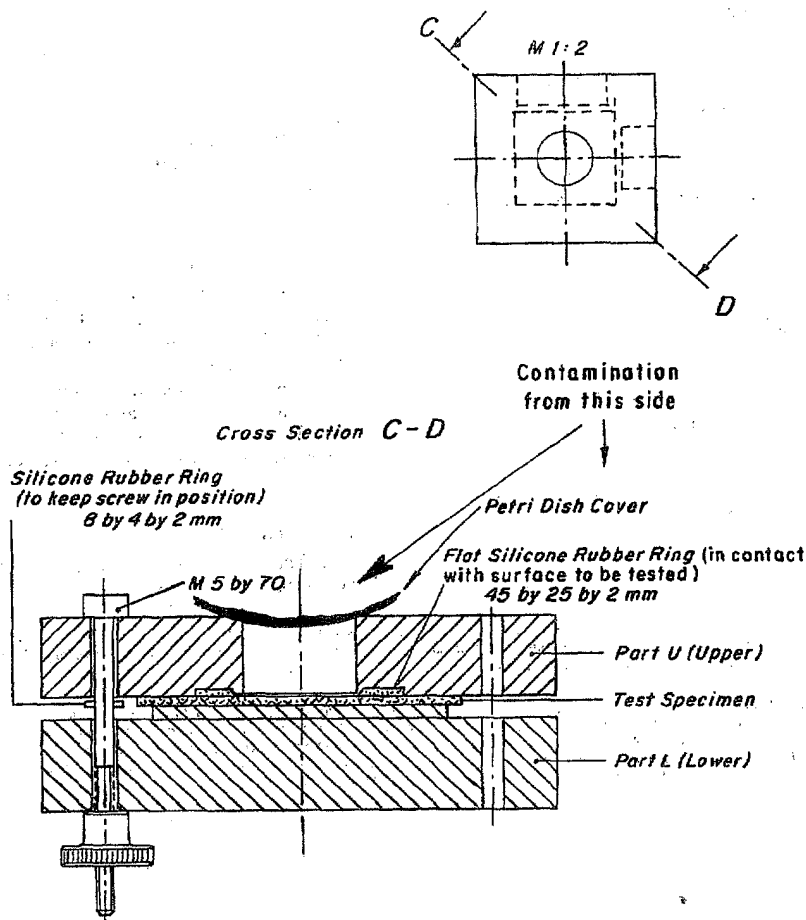


FIG. 2 Cross Section of Holder for Contamination in Seven-Day Exposure (Procedure B)

incorporate ambient water (10.2), ambient acid (10.3), and hot acid (10.4). The decontamination process for Procedure B will incorporate only ambient water (10.2).

10.2 Decontamination With Water:

10.2.1 Place a 600-mL polyethylene beaker containing 400 mL of demineralized water ($24 \pm 3^\circ\text{C}$) and a plastic-covered stirring bar on a magnetic stirrer.

10.2.2 Rigidly suspend the sample in the water to a depth of 2 in. with the contaminated side facing the center of the beaker.

10.2.3 Adjust the speed of the stirrer until the water vortex touches the stirring bar; stir for 10 min.

10.2.4 Remove the sample, rinse the back (uncontaminated side) with water, and air dry the contaminated face as in 9.1.

10.2.5 After the sample has dried, transfer it to a clean plastic bag and scan as before.

10.2.6 The ratio of the original radioactivity (counts per

minute) to the radioactivity detected after the water wash is recorded as the decontamination factor (DF) for the water wash.

10.3 Decontamination With Ambient Acid:

10.3.1 Using the same sample as for 10.2, perform a second decontamination as described in 10.2, except use the acid solution described in 6.4.1 at a temperature of $24 \pm 3^\circ\text{C}$.

10.3.2 The ratio of radioactivity after the water wash to radioactivity after the acid wash is recorded as the DF for the cold acid wash.

10.4 Decontamination With Hot Acid:

10.4.1 Continuing to use the same sample as in 10.3, perform a third decontamination as described in 10.3, except heat the acid to $80 \pm 3^\circ\text{C}$.

10.4.2 The ratio of radioactivity after the cold acid wash to radioactivity after the hot acid wash is recorded as the DF for the hot acid wash.

10.5 The ratio of radioactivity before the water wash to radioactivity after the hot acid wash, with a possible correc-

tion for natural radioactive decay described in 11.1, is recorded as the overall DF.

10.6 Control Sample:

10.6.1 Scan a contaminated, untreated control sample at the time the original and final counts are made on the test specimen.

10.6.2 Record any decrease in radioactivity.

10.6.3 Add this decrease to the value for the radioactivity after the final wash to calculate a corrected DF. The final wash will be with hot acid for Procedure A and with water for Procedure B.

11. Calculation

11.1 Calculate the decontamination factor (DF) (in consistent units) as follows:

$$DF = \frac{\text{original activity}}{\text{activity after treatment}}$$

11.2 Calculate percent removed as follows:

% removed

$$= \frac{\text{original activity} - \text{activity after decontamination}}{\text{original activity}} \times 100$$

$$= \left(1 - \frac{1}{DF}\right) \times 100$$

12. Report

12.1 The report shall include the following:

12.1.1 Procedures and conditions relating to the test specimen preparation,

12.1.2 Decontamination factors for each procedure,

12.1.3 Percent activity removed for each procedure, and

12.1.4 Any deterioration of the coating due to the test procedure.

13. Testing Laboratory

13.1 Testing shall be conducted by an independent testing laboratory. This laboratory shall not be affiliated in any manner with the coating manufacturer without the prior approval of the purchaser.

13.2 The testing laboratory shall be responsible for the documentation, reporting, and certification of all test results.

13.3 The testing laboratory shall be responsible for meeting the quality assurance requirements of the owner.

14. Precision and Bias

14.1 The overall precision and bias of this test method have not been established.

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**Standard Practice for
Design and Use of Safety Alert System for Hazardous Work
Locations in the Coatings and Lining Industry**

This practice covers a safety alert system for hazardous work locations and materials for the coatings and lining application industry. This practice is designed for multi-employer work sites.
Formerly under the jurisdiction of Committee D-33 on Protective Coatings and Lining Work for Power Generation facilities, this practice was discontinued in 1989.



Standard Practice for Surface Cleaning Concrete for Coating¹

This standard is issued under the fixed designation D 4258; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice includes surface cleaning of concrete to remove grease, dirt, and loose material prior to the application of coatings. Procedures include broom cleaning, vacuum cleaning, air blast cleaning, water cleaning, detergent water cleaning, and steam cleaning.

1.2 This practice is not intended to alter the surface profile of the concrete but to clean the surface.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 5.*

2. Referenced Documents

2.1 ASTM Standards:

D 4259 Practice for Abrading Concrete²

D 4260 Practice for Acid Etching Concrete²

D 4262 Test Method for pH of Chemically Cleaned or Etched Concrete Surfaces²

D 4263 Test Method for Indicating Moisture in Concrete by the Plastic Sheet Method²

D 4285 Test Method for Indicating Oil or Water in Compressed Air²

2.2 Other Standard:

ACI-308 Recommended Practice for Curing Concrete³

3. Summary of Practice

3.1 Surface cleaning is intended to provide a clean, contamination-free surface without removing concrete from intact, sound surfaces.

3.2 Acceptable surfaces shall be free of oil, grease, loosely adhering concrete, and other contamination. Fins and projections shall be corrected prior to surface cleaning.

3.3 Air, water cleaning, scrubbing, sweeping, or vacuuming are acceptable cleaning methods. Cleaning agents may also be used to remove oil and grease spots followed by a fresh water rinse.

3.4 Any one or a combination of the listed cleaning procedures may be used to achieve acceptably cleaned

surfaces.

4. Significance and Use

4.1 Surface cleaning is used to prepare concrete surfaces for applying coatings intended for light-duty service.

4.2 Use of this practice alone is not intended where protective systems will be used for continuous or intermittent immersion, mechanical loading, or for protective systems needing optimum bond for satisfactory performance (see Practices D 4259 and D 4260).

5. Cautions

5.1 New concrete shall be cured according to ACI-308 and sufficient additional time allowed for drying.

5.2 Moisture in the concrete may be detrimental to coating adhesion. Moisture content shall be in compliance with coating manufacturer's recommendation (see Test Method D 4263).

5.3 Concrete cure compounds, form release materials, or concrete hardeners not compatible with the coating may require removal by other surface preparation methods as in Practice D 4259.

6. Procedures

6.1 Broom Cleaning:

6.1.1 This procedure is intended to remove most surface dust and other loosely adherent solid contaminants.

6.1.2 Broom cleaning shall consist of sweeping the surface with a clean industrial stiff-bristled broom or similar device. Sweepings shall be removed from the immediate work area.

6.1.3 Broom-cleaned surfaces require additional cleaning using one or more of the surface preparation procedures specified in 6.2 through 6.6.

6.2 Vacuum Cleaning:

6.2.1 This cleaning procedure is intended to remove surface dust and other debris.

6.2.2 Vacuum cleaning shall consist of vacuuming the surface with a heavy-duty type industrial vacuum to provide an essentially dust-free surface.

6.3 Air Blast Cleaning:

6.3.1 This procedure is intended to remove debris, dust, dirt, loosely adherent laitance, and concrete from walls and ceilings and to provide an essentially sound dust-free surface.

6.3.2 Air blast cleaning shall consist of cleaning the surface with a compressed air stream at 80 to 100 psi through a blasting nozzle held approximately 2 ft from the surface.

6.3.3 The air stream shall be free of oil. This may be verified using Test Method D 4285.

6.3.4 Surface cleanliness is dependent upon carrying off air-borne dust before it is redeposited. Vacuum cleaning may be required to remove redeposited dust.

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.05 on Surface Preparation.

Current edition approved June 24, 1983. Published November 1983.

² Annual Book of ASTM Standards, Vol 06.01.

³ Available from American Concrete Institute, 22400 W. Seven Mile Rd., Detroit, MI 48219.

Water Cleaning:

6.4.1 This procedure is intended to remove dust, dirt, and water-soluble surface contaminants.

6.4.2 Water cleaning shall consist of cleaning the surface with a stream of clean potable water having sufficient pressure to remove dust, dirt, and loose material. When necessary, hand scrub with a stiff-bristled brush.

6.4.3 Prior to water cleaning, provisions shall be made for the removal of wash water and contaminants generated by this cleaning method.

6.4.4 The cleaned surface may be tested for moisture content in accordance with Test Method D 4263 prior to applying coatings.

Detergent Water Cleaning:

6.5.1 This procedure is intended to remove water-soluble surface contaminants and oils, grease, and other emulsifiable materials on the surface.

6.5.2 Detergent water cleaning shall consist of scraping off heavy deposits of grease or oil and cleaning the surface with a stiff-bristled brush using an aqueous solution of detergent or nonsolvent emulsifier. Immediately after treatment, before the surface dries, residues of the cleaning agent shall be removed by thoroughly flushing the surface with clean potable water. Repeat flushing until the pH of the surface water meets the acceptance criteria of Test Method D 4262.

6.5.3 Trisodium phosphate at not less than 4 oz/gal, or proprietary products intended for cleaning concrete, may be used following manufacturers instructions. The effectiveness of trisodium phosphate may be improved with the use of hot water.

6.5.4 Repeat 6.5.2 until water does not bead on the surfaces.

6.5.5 Prior to detergent water washing, provisions shall be made for the removal of wash water and contaminants generated by this cleaning method.

6.5.6 The cleaned surface may be tested for moisture content in accordance with Test Method D 4263.

Steam Cleaning:

6.6.1 This cleaning procedure is similar to detergent water washing, but is more effective for the removal of heavy deposits of grease and oil.

6.6.2 Steam cleaning shall consist of cleaning the surface with a jet of high-pressure steam sufficient to remove contaminants.

6.6.3 Detergents or nonsolvent emulsifying agents intended for use with steam cleaning equipment may be added where required for removal of oil and grease.

6.6.4 When detergents or other emulsifying agents are used, after cleaning and before the surface dries, the surface shall be thoroughly flushed with potable water. Repeat flushing may be necessary to remove cleaning residues.

6.6.5 The surfaces cleaned with detergent or nonsolvent emulsifying agents shall be tested for pH in accordance with Test Method D 4262 and may be tested for moisture content in accordance with Test Method D 4263 prior to applying coatings.

6.6.6 Prior to steam cleaning, provisions shall be made for the removal of water and contaminants generated by this cleaning method.

7. Inspection

7.1 Visually examine the prepared surface for the presence of debris, dust, dirt, oil, grease, loosely adherent concrete, and other contaminants.

7.2 Test surfaces cleaned with detergent or nonsolvent emulsifying agents for pH following Test Method D 4262.

7.3 Moisture content may be determined following Test Method D 4263.

8. Acceptance

8.1 Acceptable surfaces shall be free of oil, grease, loosely adhering concrete, and other contamination.

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Standard Practice for Abrading Concrete¹

This standard is issued under the fixed designation D 4259; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice includes surface preparation of concrete to prepare the surface prior to the application of coatings.

1.2 This practice is intended to alter the surface profile of the concrete and to remove foreign materials and weak surface laitance.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 5.*

2. Referenced Documents

2.1 ASTM Standards:

- D 4258 Practice for Surface Cleaning Concrete for Coating²
- D 4263 Test Method for Indicating Moisture in Concrete by the Plastic Sheet Method²
- D 4285 Test Method for Indicating Oil or Water in Compressed Air²
- D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers²
- 2.2 *Other Standard:*
ACI-308 Recommended Practice for Curing Concrete³

3. Summary of Practice

3.1 This practice is intended to provide a clean, contamination-free, and roughened surface.

3.2 Acceptable surfaces shall be free of laitance, form release agents, curing agents, oil, grease, and other penetrating contaminants. The surface shall be free of fins, projections and loosely adhering concrete, dirt, and dust particles.

3.3 For some applications, a minimum concrete surface strength may be required for proper coating performance.

3.4 Acceptable methods of preparation include abrasive blasting (wet or dry), mechanical abrading, water blasting, and other similar procedures that will alter the surface profile of the concrete.

4. Significance and Use

4.1 This practice is used to prepare concrete for coatings where optimum bond is desired for service conditions such as continuous or intermittent immersion, temperature cycling, or mechanical loading.

5. Cautions

5.1 New concrete shall be cured in accordance with ACI-308.

6. Mechanical Abrading Procedure

6.1 *Suitable Surfaces*—Mechanical abrading is suitable for use on formed surfaces and floors and for the removal of fins and projections.

6.2 *Pre-Surface Preparation:*

6.2.1 Remove grease, oil, and other penetrating contaminants. (See Practice D 4258.)

6.2.2 Concrete surfaces may be wet or dry as appropriate to the type of equipment to be used.

6.3 *Apparatus:*

6.3.1 Typical apparatus covered by this method are rotary impact, vertical impact, and circular grinding equipment.

6.3.2 Use the equipment in accordance with the manufacturer's instructions in an organized manner in order to thoroughly cover the entire surface to be prepared.

6.4 *Appearance of Prepared Surface:*

6.4.1 The intent is to remove sufficient material in order to achieve a sound concrete surface free of laitance, glaze, efflorescence and incompatible concrete curing compounds or form release agents.

6.4.2 The surface shall have a roughened, textured appearance. Aggregate may be exposed. A roughness standard may be established by mutual agreement.

6.4.3 The appearance will vary depending upon the equipment used and type of concrete.

6.5 *Post-Surface Preparation Cleaning*—Clean in accordance with Practice D 4258 to remove loose material.

7. Water Blast Cleaning

7.1 *Suitable Surfaces*—This method is suitable for use on formed surfaces and floors.

7.2 *Pre-Surface Preparation:*

7.2.1 Remove grease, oil, and other penetrating contaminants (see Practice D 4258).

7.2.2 Correct fins and protruding irregularities by mechanical means.

7.2.3 Concrete surface may be wet or dry.

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.05 on Surface Preparation.

Current edition approved Feb. 26, 1988. Published April 1988. Originally published as D 4259 - 83. Last previous edition D 4259 - 83.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Available from American Concrete Institute, 22400 W. Seven Mile Rd., Detroit, MI 48219.

8.3 Apparatus—Typical apparatus covered by this method is a piston-type positive displacement pump, high-pressure water blasting unit. The cleaning medium shall be potable water.

8.4 Appearance of Prepared Surface:

8.4.1 The prepared surface shall be clean and free of dust, laitance and efflorescence.

8.4.2 The surface shall have a roughened textured appearance. A roughness standard may be established by mutual agreement.

8.5 Post-Surface Preparation Cleaning—Clean in accordance with Practice D 4258 to remove loose material.

8.6 Abrasive Blast Cleaning Procedure

8.6.1 Pre-Surface Preparation:

8.6.1.1 Remove grease, oil, and other penetrating contaminants (see Practice D 4258).

8.6.1.2 Correct fins and protruding irregularities by mechanical means.

8.6.1.3 Concrete surface may be wet or dry as appropriate to the type of equipment to be used.

8.6.2 Suitable Surfaces—This method is suitable for use on prepared surfaces and floors.

8.6.3 Apparatus:

8.6.3.1 Typical methods are wet or dry open-blast cleaning with nozzles and self-contained recirculating blast-cleaning apparatus.

8.6.3.2 The air stream used for nozzle blast cleaning shall be free of oil. This may be verified using Test Method D 4285.

8.6.3.3 Use the equipment in accordance with the manufacturer's instructions.

8.6.4 Appearance of Prepared Surface:

8.4.1 The intent is to remove sufficient material in order to achieve a sound concrete surface free of laitance, glaze, efflorescence, and incompatible concrete curing compounds or form release agents.

8.4.2 The surface shall have a roughened textured appearance. A roughness standard may be established by mutual agreement.

8.4.3 Some aggregate may be exposed and bug holes shall be opened.

8.5 Post-Preparation Cleaning—Clean in accordance with Practice D 4258 to remove loose material.

9. Inspection

9.1 Visually examine the prepared surface for loose adhering concrete, thin crusts bridging voids, fins, and projections.

9.2 Visually examine the prepared surface for oil, grease, and markings.

9.3 If required, surface strength may be determined in accordance with Method D 4541 or other agreed upon method.

10. Acceptance

10.1 Acceptable surfaces shall be free of laitance, oil, grease, and other materials incompatible with the coating. The surface shall also be free of fins, projections and loosely adhering concrete, dirt, and dust particles.

10.2 The surface shall have a roughened, textured appearance. Aggregate may be exposed. Bug holes shall be opened.

10.3 The roughened textured appearance shall be similar to the roughness standard established by mutual agreement.

10.4 If specified, the treated surface shall meet the surface strength requirements.

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Standard Practice for Acid Etching Concrete¹

This standard is issued under the fixed designation D 4260; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice includes surface preparation of concrete to prepare the surface prior to the application of coatings.

1.2 This practice is intended to alter the surface profile of the concrete and to remove foreign materials and weak surface laitance.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 5.*

2. Referenced Documents

2.1 ASTM Standards:

D 4258 Practice for Surface Cleaning Concrete for Coating²

D 4259 Practice for Abrading Concrete²

D 4262 Test Method for pH of Chemically Cleaned or Etched Concrete Surfaces²

D 4263 Test Method for Indicating Moisture in Concrete by the Plastic Sheet Method²

D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers²

2.2 Other Standard:

ACI-308 Recommended Practice for Curing Concrete³

3. Summary of Practice

3.1 This practice is intended to provide a clean, contamination-free, and roughened surface.

3.2 Acceptable surfaces shall be free of laitance, form release agents, curing agents, oil, grease, and other penetrating contaminants. The surface shall be free of fins, projections, and loosely adhering concrete, dirt, and dust particles.

3.3 For some applications, a minimum concrete surface strength may be required for proper coating performance.

4. Significance and Use

4.1 This practice is used to prepare concrete for coatings where optimum bond is desired for service conditions such

as continuous or intermittent immersion, temperature cycling, or mechanical loading.

5. Cautions

5.1 New concrete shall be cured in accordance with ACI-308.

5.2 Concrete cure compounds, form release materials, or concrete hardeners may require abrading, in accordance with Practice D 4259, as acid etching may not be effective.

5.3 All oil and grease shall be removed in accordance with Practice D 4258 prior to mechanical abrading, abrasive blast cleaning, water blasting, or acid etching.

5.4 Use and disposal of materials should conform to established federal, state, local, and project requirements.

6. Acid Etching Procedure

6.1 Pre-Surface Preparation:

6.1.1 Remove grease, oil, and other penetrating contaminants (see Practice D 4258).

6.1.2 Remove fins and protruding surface irregularities by mechanical means.

6.1.3 Surfaces shall be free of standing water.

6.1.4 Some curing compounds may not be removed by acid etching and will require preparation by mechanical abrading, abrasive blasting, or water blasting in accordance with Practice D 4259.

6.2 Suitable Surfaces:

6.2.1 This method is primarily suited for use on horizontal surfaces.

6.2.2 Other methods of preparation may be more suitable for rough concrete surfaces and walls or overhead surfaces.

6.3 Acid Etching Solutions:

6.3.1 Typical solutions covered by this method are muriatic (hydrochloric), sulfamic, phosphoric and citric acids. Hydrochloric acid shall not be used where chlorides are prohibited.

6.3.2 The acid concentrations of etching solutions may vary, depending on the concrete texture and degree of etching required.

6.3.3 The concrete surface shall be pre-wet with water prior to applying etching solutions. Free-standing water shall be removed. Uniformly apply the etching solution to the wet surface. Polyethylene sprinkling cans are suitable for applying acid solutions. Bubbling should be uniformly evident. If not, this indicates the presence of grease or oil contamination, or both, curing compounds or sealers, or a need to increase the concentration of the acid solution. Scrub the acid wetted surface with a stiff bristle brush.

6.3.4 When the etching solution bubbling begins to subside, flush surfaces to remove reaction products, and inspect for uniform roughening and removal of laitance. Repeat

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.05 on Surface Preparation.

Current edition approved Feb. 26, 1988. Published April 1988. Originally published as D 4260 - 83. Last previous edition D 4260 - 83.

² Annual Book of ASTM Standards, Vol 06.01.

³ Available from American Concrete Institute, 22400 W. Seven Mile Rd., Detroit, MI 48219.

application of acid to obtain required surface.

6.3.5 After the desired roughening is achieved, thoroughly flush the surface with potable water. Repeated flushing and scrubbing with a stiff-bristled brush may be necessary to remove acid residues.

6.3.6 The acid etched surface shall be tested for pH in accordance with Test Method D 4262 and may be tested for moisture content in accordance with Test Method D 4263 prior to applying coating.

6.4 Appearance of Prepared Surface:

6.4.1 The intent is to remove sufficient material in order to achieve a sound concrete surface free of laitance, glaze, efflorescence, and incompatible concrete curing compounds or form release agents.

6.4.2 The acid etched surface shall be uniformly roughened to a degree similar in appearance to a medium to coarse grade sandpaper. A roughness standard may be established by mutual agreement.

7 Inspection

7.1 Visually examine the prepared surface for loose ad-

hering concrete, thin crusts bridging voids, fins, and projections.

7.2 Visually examine the prepared surface for oil, grease, and markings.

7.3 Test surfaces cleaned by acid etching for pH in accordance with Test Method D 4263.

7.4 If required, surface strength may be determined in accordance with Method D 4541 or other agreed upon method.

8. Acceptance

8.1 Acceptable surfaces shall be free of laitance, oil, grease, and other materials incompatible with the coating. The surface shall also be free of fins, projections, and loosely adhering concrete, dirt, and dust particles.

8.2 The surface shall have a roughened, textured appearance. Aggregate may be exposed. Bug holes shall be opened.

8.3 A roughness standard may be established by mutual agreement.

8.4 If specified, the treated surface shall meet the surface strength requirements.

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Standard Practice for Surface Cleaning Concrete Unit Masonry for Coating¹

This standard is issued under the fixed designation D 4261; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers surface cleaning of concrete unit masonry to remove dust, dirt, mortar spatter, oil, and grease prior to the application of coatings. Procedures include vacuum cleaning, air-blast cleaning, water cleaning, detergent water wash, steam cleaning, and mechanical cleaning.

1.2 This practice is not intended to alter the surface profile of the concrete masonry units but to clean the surface.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 5.*

2. Referenced Documents

2.1 ASTM Standards:

D 4258 Practice for Surface Cleaning Concrete for Coating²

D 4262 Test Method for pH of Chemically Cleaned or Etched Concrete Surfaces²

D 4263 Test Method for Indicating Moisture in Concrete by the Plastic Sheet Method²

2.2 Other Standard:

ACI Specification 531.7-76 Specification for Concrete Masonry Construction³

3. Summary of Practice

3.1 Surface cleaning is intended to provide a clean, contamination-free surface suitable for the application of coatings.

3.2 Acceptable surfaces shall be free of dust, dirt, mortar spatter, oil, grease, and other contaminants. Mortar joints shall be struck flush or tooled with a round jointer and shall be free of rough edges and nibs (see ACI 531.1-76).

3.3 Vacuum cleaning, air blast cleaning, water cleaning, detergent water wash, steam cleaning, hand tool, and mechanical cleaning are acceptable cleaning methods.

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.05 on Surface Preparation.

Current edition approved Aug. 17, 1983. Published November 1983.

² Annual Book of ASTM Standards, Vol 06.01.

³ Available from American Concrete Institute, 22400 W. Seven Mile Rd., Detroit, MI 48219.

3.4 Any one or a combination of listed cleaning procedures may be used to achieve acceptably cleaned surfaces.

4. Significance and Use

4.1 Surface cleaning is to be used to prepare concrete unit masonry surfaces for applying coatings intended for light duty service, splash and spillage of water and chemical solutions, radiation exposure, decontamination, and repeated washdowns with alkaline cleaners.

5. Cautions

5.1 Moisture in concrete unit masonry may be detrimental to coating adhesion. Moisture content shall be in compliance with the coating manufacturer's recommendations (see Test Method D 4263 for moisture test method).

6. Procedures

6.1 *Steam Cleaning*—This procedure is intended to remove heavy deposits of grease and oil, as well as other water soluble surface contaminants and emulsifiable materials, with a jet of high pressure steam, in accordance with Practice D 4258.

6.2 *Detergent Water Wash*—This procedure is intended to remove water soluble surface contaminants, oils, grease, and other emulsifiable materials with an aqueous solution of detergent or nonsolvent emulsifier in accordance with Practice D 4258.

6.3 *Water Cleaning*—This procedure is intended to remove dust, dirt, and water soluble contaminants with a stream of water under pressure, in accordance with Practice D 4258.

6.4 Mechanical Tool Cleaning:

6.4.1 The intent of this procedure is to remove mortar spatter, efflorescence, and similar dry, firmly adhering foreign material prior to performing either cleaning operation.

6.4.2 This procedure shall consist of scraping, abrading, brushing, or stoning to dislodge mortar and similar materials from the surface.

6.4.3 Clean in accordance with Practice D 4258 to remove dust and loose particles.

6.5 *Vacuum Cleaning*—This procedure is intended to remove surface dust and other debris by vacuum cleaning in accordance with Practice D 4258.

6.6 *Air Blast Cleaning*—This procedure is intended to remove debris, dust, dirt, and loosely adhering material by air blast cleaning in accordance with Practice D 4258.

7. Inspection

7.1 Visually examine prepared surface for the presence of debris, dust, dirt, oil, grease, loose particles, mortar spatter, and other contaminants.

7.2 Test surfaces cleaned with detergent or non-solvent emulsifying agents for pH in accordance with Test Method D 4262.

7.3 Moisture content may be determined in accordance

with Test Method D 4263.

8. Acceptance

8.1 Acceptable surfaces shall be free of dust, dirt, mortar spatter, oil, grease, and other contaminants.

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Standard Test Method for pH of Chemically Cleaned or Etched Concrete Surfaces¹

This standard is issued under the fixed designation D 4262; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the procedure for determining the acidity or alkalinity of concrete surfaces prepared by chemical cleaning or etching prior to coatings.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Definition

2.1 *pH*—a measure of the hydrogen ion concentration and indicates whether a solution is acidic, alkaline, or neutral.

3. Significance and Use

3.1 Chemical cleaning or etching is used to prepare concrete for coating.

3.2 Residual chemicals not removed by water rinsing may adversely affect the performance and adhesion of coatings applied over prepared concrete surfaces. It is the intent of this test method to determine that residual chemicals have been removed by measuring the acidity or alkalinity of the final rinsed surface.

4. Apparatus

4.1 *pH Test Paper*, with a minimum range from 1 to 11

pH units with a capability of measuring in increments of 0.5 pH units.

5. Materials

5.1 *Potable Water*, for rinsing chemically cleaned or etched concrete surfaces.

5.2 *Wet Concrete Surface*, following the final water rinse and before the rinse water has completely drained off the surface.

6. Procedure

6.1 Tear off a strip of test paper, wet with test water and after the color develops, compare with color chart to determine pH.

6.2 The pH of the water used for rinsing shall be determined to establish acceptance criteria. Readings shall be taken at the beginning and end of the final rinse cycle.

6.3 At least two surface pH readings shall be taken for each 500 square feet or portion thereof. Readings shall be taken at randomly selected locations immediately following the final rinse and before all the rinse water has drained off the surface.

6.4 Unless otherwise specified, tests shall be conducted in accordance with this procedure.

7. Acceptance Criteria

7.1 The pH readings following the final rinse shall not be more than 1.0 pH lower or 2.0 points higher than the pH of the rinse water (see 6.2) unless otherwise specified.

¹ This test method is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.05 on Surface Preparation.
Current edition approved Aug. 17, 1983. Published November 1983.

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Standard Test Method for Indicating Moisture in Concrete by the Plastic Sheet Method¹

This standard is issued under the fixed designation D 4263; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

⁶¹ NOTE—Editorial changes were made throughout in September 1988.

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Scope

1.1 This test method is used to indicate the presence of capillary moisture in concrete.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific caution statements, see Section 4.*

Significance and Use

2.1 Capillary moisture in the concrete may be detrimental to the performance of certain coating systems that cannot tolerate moisture on or within the surface boundary.

2.2 This test method is used prior to the application of coatings on concrete.

Materials

3.1 *Transparent Polyethylene Sheet*, commercially available, approximately 4 mils (0.1 mm) thick.

3.2 *Adhesive Tape* that will adhere to the substrate. (Duct tape 2 in. (50 mm) wide is suggested.)

Cautions

4.1 This test method shall be conducted when the surface

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Current edition approved Aug. 17, 1983. Published November 1983.

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temperature and ambient conditions are within the established parameters for application of the coating system.

4.2 Avoid direct sunlight, direct heat, or damage to the plastic sheet, as such treatment affects the reliability of the results.

5. Procedure

5.1 Tape a segment of plastic sheet, approximately 18 by 18 in. (457 by 457 mm), tightly to the concrete surface making sure that all edges are sealed.

5.2 Allow the plastic sheet to remain in place a minimum of 16 h.

5.3 After the allowed time has elapsed, remove the plastic sheet and visually inspect the underside of the sheet and the concrete surface of the patch for the presence of moisture.

5.4 Sampling:

5.4.1 *Floors*—One test area per 500 ft² (46 m²) or portion thereof, of surface areas unless otherwise specified.

5.4.2 *Walls and Ceilings*—One test area per 500 ft² (46 m²) or portion thereof, of surface area unless otherwise specified.

5.4.3 The recommended practice is a minimum of one test for each 10 ft (3 m) of vertical rise in all elevation starting within 12 in. (300 mm) of the floor.

6. Report

6.1 Report the presence or absence of moisture.

7. Precision and Bias

7.1 This test method indicates the presence of capillary moisture. No precision or bias has been established for this test method.



Standard Test Method for Indicating Oil or Water in Compressed Air¹

This standard is issued under the fixed designation D 4285; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is used to determine the presence of oil or water in compressed air used for abrasive blast cleaning, air blast cleaning, and coating application operations.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 4.*

2. Significance and Use

2.1 Clean compressed air is required to prevent contamination of coating materials and surfaces being prepared for coating. This test method is a visual examination technique for determining oil or water in compressed air. Other types of contamination may require additional analytical techniques for detection.

3. Apparatus and Materials

3.1 *Absorbent Collector*, such as white absorbent paper or cloth and rigid backing for mounting absorbent collector, or

3.2 *Nonabsorbent Collector*, such as rigid transparent plastic, approximately $\frac{1}{4}$ in. thick.

4. Cautions

4.1 To avoid false indications prior to testing:

4.1.1 Allow compressed air system to reach operating conditions, and

4.1.2 Allow air to discharge at operating conditions to remove accumulated condensation in the system.

4.2 Fasten the collector material to the rigid backing. Avoid personal contact with the air stream.

5. Sampling

5.1 Conduct the test on discharging air as close to the use point as possible and after the inline oil and water separators.

6. Procedure

6.1 Use either the absorbent or nonabsorbent collector.

6.2 Position the collector within 24 in. of the air-discharge point, centered in the air stream.

6.3 Adjust air discharge so that the collector remains intact during the test. Allow air to discharge onto the collector for a minimum of 1 min.

6.4 Visually examine the collector for the presence or absence of oil or water, or both.

7. Interpretation of Results

7.1 Any indication of oil discoloration on the collector shall be cause for rejection of the compressed air for use in abrasive blast cleaning, air blast cleaning, and coating application operations.

7.2 Any indications of water contamination on the collector shall be cause for rejection of the compressed air for use in those applications where water is detrimental, such as abrasive blast cleaning, air blast cleaning, and coating application operations.

8. Precision and Bias

8.1 No precision or bias statement has been established for this test method.

¹ This test method is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generating Facilities and is the direct responsibility of Subcommittee D33.05 on Surface Preparation. Current edition approved Sept. 30, 1983. Published December 1983.

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Standard Practice for Determining Coating Contractor Qualifications for Nuclear Powered Electric Generation Facilities¹

Scope

1.1 This practice provides a criteria guide and procedural method to assist utility owners, architects, engineers, contractors, and other selection agencies in determining the overall qualifications of a coating contractor to execute coating work for the primary containment and other safety-related facilities of light-water nuclear power plants.

1.2 The qualification criteria and requirements address the essential basic capability of a contractor to execute nuclear coating work. Obviously, the specific capability to execute those requirements unique to a given project must also be carefully considered. The evaluation procedure contained in this practice is designed to be adaptive to this detailed final qualification process. Variation or simplification of the practice is appropriate for non safety-related areas of nuclear power plants, fossil fueled facilities, and other industrial projects.

1.3 The overall capability of a contractor to successfully execute the varied and complex requirements of nuclear coating work is dependent upon competency in a variety of essential categories.

1.4 The nine evaluation categories described in Sections 3 through 12 detail the specific data to be provided by the contractor as essential to determining qualification status. In addition, a tenth untitled category has been provided on the evaluation work described in Section 12 for inclusion of other information pertinent to a specific project.

1.5 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 ASTM Standard:

D 3843 Practice for Quality Assurance for Protective Coatings Applied to Nuclear Facilities²

D 4537 Guide for Establishing Procedures to Qualify and Certify Inspection Personnel for Coating Work in Nuclear Facilities²

2.2 American National Standard:

N 45.2.6 Qualifications of Inspection, Examination, and Testing Personnel for Nuclear Power Plants³

2.3 Other Standard:

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.07 on Application.

² Current edition approved March 30, 1990. Published May 1990. Originally published as D 4286 - 84. Last previous edition D 4286 - 84.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Available from American National Standards Institute, 11 W. 42nd St., 13th floor, New York, NY 10036.

Nuclear Regulatory Agency 10 CFR 50 Appendix B, Quality Assurance Criteria for Nuclear Power Plants⁴

3. Personnel

3.1 *Home Office Management and Staff*—Resumes of the following types of personnel including educational background, duration and degree of involvement in industrial coating operations, qualifications, registrations, certifications, and specific experience in nuclear coating work.

3.1.1 *Principals and General Management.*

3.1.2 *Engineering Manager.*

3.1.3 *Project Engineers*—List specific nuclear coating work project involvement.

3.1.4 *QA/QC Manager*—List specific qualification/certification credentials per ANSI N45.2.6, or Guide D 4537, or both.

3.2 *General Field Supervision*—Resumes to include specific nuclear coatings work experience.

3.3 *On-Site Project Supervision:*

3.3.1 Total number of personnel qualified for nuclear coatings work, including specific work history.

3.3.2 Total number available in a specific time frame, if appropriate.

4. Contractor Work Experience

4.1 *General Experience*—List of comparable industrial projects, including type (construction or maintenance), form of contract, dollar volume, scope, and duration (start and completion dates) executed during the past five years. Accompanying this listing provide personnel references to include names, titles, locations, and telephone numbers.

4.2 *Nuclear Coating Work Experience:*

4.2.1 List of nuclear projects including type (construction or maintenance), form of contract, dollar volume, scope, and duration, materials, and equipment involved. Accompanying this listing provide personnel references to include names, titles, locations, and telephone numbers.

4.2.2 If the contractor has executed work comparable to the specific evaluation project, submit complete case histories of scope and functions performed to include personnel references for each case.

5. Equipment

5.1 List contractor-owned equipment in the categories listed in 5.1.1 through 5.1.9. Indicate those items required for the specific evaluation project.

5.1.1 Vehicles.

5.1.2 Surface preparation, all types.

5.1.3 Abrasive control and recovery.

5.1.4 Coatings mixing and application, all types.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

- 5.1.5 Environmental control and curing.
- 5.1.6 Rigging and enclosure.
- 5.1.7 Safety.
- 5.1.8 Quality control testing instrumentation.
- 5.1.9 Specialized equipment.
- 5.1.9.1 Unique equipment required for the specific project, if applicable.
- 5.1.9.2 Contractor to indicate and provide examples of capability to design and install highly specialized items of equipment, enclosure systems, devices, etc. which have been utilized on previous nuclear coating projects.
- 5.1.10 *Equipment Repair and Replacement Capability*—Contractor to describe standard procedure followed in the event of equipment failure to prevent lost time situations.

6. Quality Assurance

6.1 *Quality Assurance Program*—The contractor shall submit one copy of his quality assurance program that shall conform to 10 CFR 50 Appendix B, ANSI N 45.2.6, Guide D 4537, Practice D 3843, and other applicable documents. The program shall include implementation procedures and sample forms.

6.2 *Quality Assurance/Quality Control*—List number of personnel qualified in accordance with ANSI N 45.2.6 or Guide D 4537, or both, for each of the following:

- 6.2.1 LEVEL III Inspector,
- 6.2.2 LEVEL II Inspector, and
- 6.2.3 LEVEL I Inspector.

7. Training

7.1 Submit training manual for craft and supervisory personnel. If no formal training program exists, the contractor shall submit evidence of procedures and methods utilized.

8. Safety

8.1 Submit copy of corporate safety program. If no formal program exists, the contractor shall submit evidence of safety procedures and work rules.

9. Production/Cost Control

9.1 Submit explanation of methods and procedures utilized to monitor and regulate personnel and equipment productivity.

9.2 Submit sample of actual report forms utilized to record job progress, labor cost analysis, and equipment deployment efficiency.

10. Financial

10.1 Submit documentation to verify financial capability to manage and execute all project requirements. Include the following data:

- 10.1.1 Current financial statement,
- 10.1.2 Trade references, three or more,
- 10.1.3 Bank references, one or more,
- 10.1.4 Bonding capacity, name of bonding company, and name and address of agent, and
- 10.1.5 Current certificate of insurance.

11. Current and Projected Performance Capability (restricted to specific project considerations)

11.1 Submit a statement indicating current commitment status of personnel, equipment, and management capability. The statement shall include analysis of and ability to fulfill requirements in the following categories:

- 11.1.1 Craft personnel,
- 11.1.2 Staff engineering and supervisory personnel,
- 11.1.3 Management personnel,
- 11.1.4 Equipment, and
- 11.1.5 Materials.

12. Contractor Evaluation Work Sheet

12.1 The sample work sheet shown in Fig. 1 is suggested for consolidation of rating and comparison data. Note that there are nine basic rating categories corresponding to the criteria detailed in the text of this practice. Space for an optional tenth category has been provided for owner insertion of other rating data that may be unique to the specific project involved.

12.2 *Rating Values*—Numerical rating values are structured on the following format:

Rating	Value
Excellent	4
Good	3
Average	2
Poor	1
Unsatisfactory	0
No data	0

12.3 *Weighted Category Values*—Each of the ten (includes option) evaluation categories will be assigned a weighted value by the owner in accordance with the specific requirements of the project. The value scale will range from 1 (least critical) to 10 (most critical). The weighted value of each category will be used as a multiplier to determine the overall rating for each contractor in each category.

Project: _____ Date: _____

CONTRACTOR EVALUATION WORK SHEET

Evaluator: _____ Title: _____

	PERSONNEL (W.V. =)					EXPERIENCE (W.V. =)					EQUIPMENT (W.V. =)					Q.A./Q.C. (W.V. =)					TRAINING (W.V. =)					SAFETY (W.V. =)									
	Home Office	General Field	On Site	SUB-TOTAL	X W.V.	TOTAL	General	Ref. Qual.	Nuclear	Ref. Qual.	Case History	SUB-TOTAL	X W.V.	TOTAL	Inventory	Design Capacity	Repair Capacity	SUB-TOTAL	X W.V.	TOTAL	Manual	Personnel	Case History	SUB-TOTAL	X W.V.	TOTAL	Manual	Other	SUB-TOTAL	X W.V.	TOTAL	Manual	Other	SUB-TOTAL	X W.V.
Contractor "A"																																			
Contractor "B"																																			
Contractor "C"																																			
Contractor "D"																																			
Contractor "E"																																			
Contractor "F"																																			
Contractor "G"																																			

	P/C CONTROL (W.V. =)					FINANCIAL (W.V. =)					LIMITATIONS (W.V. =)					OPTION (W.V. =)					SUMMARY														
	Procedures	Report Forms	Other	SUB-TOTAL	X W.V.	TOTAL	Statement	Ref. Qual.	Bond	Insurance	SUB-TOTAL	X W.V.	TOTAL	Craft	Staff	Management	Equipment	SUB-TOTAL	X W.V.	TOTAL															
Contractor "A"																																			
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Contractor "F"																																			
Contractor "G"																																			

Rate Value: Excellent = 4, Good = 3, Average = 2, Poor = 1, Unsatisfactory = 0, No Data = 0.
 Weighted Value: 1 (Least Critical) through 10 (Most Critical)

FIG. 1 Contractor Evaluation Work Sheet

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Standard Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer¹

This standard is issued under the fixed designation D 4287; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the viscosity of paints, varnishes, and related products at a rate of shear of $12\,000\text{ s}^{-1}$.

1.2 Paints and varnishes that dry very rapidly may not give reproducible results with this test method. Measurements made at elevated temperatures may also give poor precision due to loss of volatiles and to drying.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability or regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems²

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²

3. Summary of Test Method

3.1 The material to be tested is placed between the cone and plate of an ICI cone/plate viscometer, then subjected to a high shear rate while the viscosity is determined.

4. Significance and Use

4.1 The viscosity value obtained by this test method gives information about the flow properties of the material under application conditions: brushing, spraying, electrostatic disk, or roll coating.

4.2 This test method is suitable for all paints and varnishes whether they are Newtonian in behavior or not. However, due to the narrow gap between the stationary and rotary parts of high-shear viscometers, this test method is more reproducible for paints having finer pigment dispersions as determined by Test Method D 1210.

5. Apparatus

5.1 *ICI Cone/Plate Viscometer*³ (see Fig. 1) with 0 to 10-P

(0 to 1 Pa·s) or 0 to 5-P cone producing a rate of shear of $12\,000\text{ s}^{-1}$. With higher viscosity materials, other cones (0 to 20 P, 0 to 40 P, 0 to 100 P) may be used on agreement between the producer and the user, but it should be noted that these give lower shear rates ($\sim 3000\text{ s}^{-1}$) not truly representative of application conditions. Other cone/plate viscometers may be used on agreement between the producer and the user as long as the same rate of shear is measured and it is approximately $12\,000\text{ s}^{-1}$. However, results may differ from those produced with an ICI cone/plate viscometer.

NOTE 1—The SI units for viscosity are pascal-seconds (Pa·s, 1 Pa·s = 10 P, 1 mPa·s = 1 cP).

6. Reagents and Materials

6.1 *Water or Solvent*—Water or a low viscosity solvent such as xylene or mineral spirits to be used for zeroing the instrument.

6.2 *Mineral Oils*—Three standard mineral oils with known viscosities (certified by an approved laboratory) lying between 10 and 90 % of full scale.⁴

7. Sampling

7.1 Take a representative sample of the product to be tested in accordance with Practice D 3925. If the sample has a tendency to settle or separate on standing, it must be stirred or shaken until homogeneous before a test specimen is taken from it. The specimen must be free of any foreign matter or air bubbles and its volume must be sufficient to cover the portion of the viscometer plate under the cone when the latter is brought into contact with the plate.

8. Preparation of Apparatus

8.1 Zero the apparatus daily when in regular use, or otherwise before use, by following the procedure in Section 9, but using a low viscosity liquid. If the pointer does not indicate zero, it may be adjusted by means of a lever on the left-hand side of the upper part of the instrument housing. If the instrument cannot be zeroed, it should be returned to the supplier for adjustment.

8.2 Verify the calibration of the apparatus by following the procedure in Section 9, but using standard refined mineral oils having Newtonian characteristics and known viscosities.⁴ If the viscometer pointer reads the correct viscosity (or within 5 % of that value) with two or more oils whose viscosities bracket those of specimens to be tested, then the viscometer readings may be used as is. If the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 4287 - 83. Last previous edition D 4287 - 83.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ ICI cone/plate viscometers are available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910 and from Research Equipment (London) Ltd., 64 Wellington Rd., Hampton Hill, Middlesex TW12 1JX, England.

⁴ Such oils are available from The Cannon Instrument Co., P.O. Box 16, State College, PA 16801.

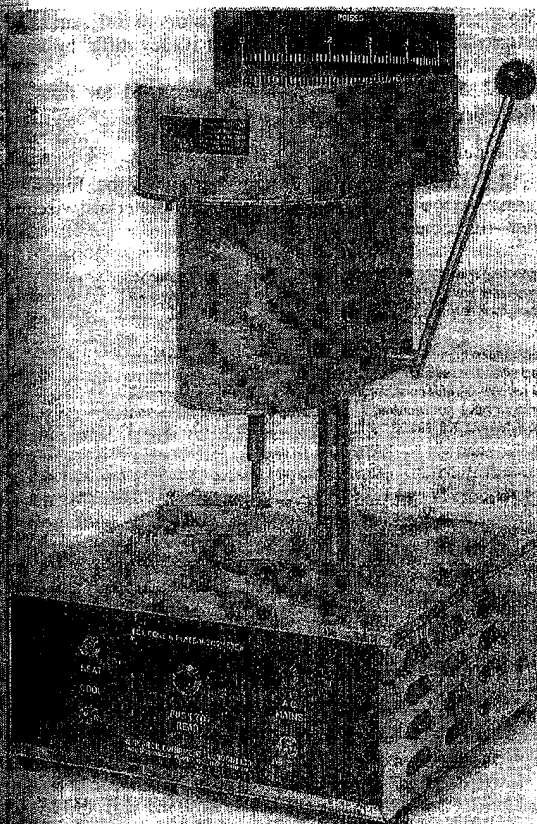


FIG. 1 ICI Cone/Plate Viscometer

viscometer readings do not give the correct viscosities for the oils, then a calibration curve must be constructed by taking ICI viscometer readings for three oils and plotting measured viscosity versus specified (correct) viscosity for the oils. Subsequent measurements are corrected to true viscosities through use of the curve.

9.3 Check the cones periodically for wear. Replace any cone that shows a definite flattening of the apex. Some users have found it necessary to replace cones every year.

9.4 The determination must be made at a closely controlled temperature of $25 \pm 0.3^\circ\text{C}$ unless otherwise agreed. In order to check the temperature control, carry out the test as outlined in 9.1 with the standard refined mineral oil of the nearest viscosity. Allow the viscometer to run with this oil for 10 min and determine whether the reading decreases. If the decrease is more than 10 %, the apparatus is unsuitable for the determination of viscosities at high rates of shear in accordance with this test method.

NOTE 2—Multitemperature ICI cone/plate viscometers have heating, but not cooling, capabilities. Therefore, runs with these viscometers at 25°C should only be done at room temperatures at or below 22°C to ensure that the plate temperature does not go above 25°C . The

instruments that operate at 25°C only do have cooling capabilities and give better temperature control.

9. Procedure

9.1 With the cone in the down position, turn the instrument on and allow it to warm up for at least 5 min. For a multitemperature instrument, set the dial at 25°C or to an alternatively agreed upon temperature. Raise the cone to the up position. Transfer a suitable amount of the product to be tested to the plate, taking care to avoid the inclusion of air bubbles, and again lower the cone to the down position. Wait for 30 s to allow the specimen to attain the agreed upon temperature.

9.2 Start the cone rotating and record the reading on the scale (Note 3) when the pointer becomes steady.

NOTE 3—Whether the reading gives a direct indication of the viscosity or not, depends on the cone and scale used.

NOTE 4—In some cases it is difficult to judge whether a constant reading has been obtained. However, if the pointer does not become steady after 15 s, record the reading at 15 s and mention the lack of a constant reading in the test report. If highly accurate readings are required, make the readings below 90 % of the scale.

9.3 If the reading does not directly indicate the viscosity, multiply the reading by the appropriate conversion factor or use the appropriate calibration curve to obtain the viscosity.

9.4 Clean both the cone and the plate carefully, employing a cloth or tissue and a suitable solvent. Take care to remove all of the test material and cleaning solvent. Do not use cleaning utensils that may damage the apparatus. *Metal cleaning tools must never be used.*

9.5 Repeat the determination with a second specimen. If the two viscosity determinations differ by less than 7 %, calculate their mean and report as the high shear viscosity for the material. If they differ by more than 7 %, make a third determination. If no two readings are within 7 % of each other, then the material is not suitable for testing by this test method.

10. Report

10.1 Report the following information:

- 10.1.1 Reference to this test method,
- 10.1.2 Type and identification of the product under test,
- 10.1.3 Type of cone used (0 to 5 P, 0 to 10 P, etc.),
- 10.1.4 Rate of shear at which the determination was made (in reciprocal seconds),
- 10.1.5 Temperature at which the determination was made,
- 10.1.6 Test results in poises, reported to the nearest 1 % of the total range, that is, 0.05 P for 0 to 5 P-cones, 0.1 P for 0 to 10 P-cones, etc.,
- 10.1.7 Any deviation, by agreement or otherwise, from the test procedures described, and
- 10.1.8 Date of the test.

11. Precision and Bias⁵

11.1 *Precision*—On the basis of an interlaboratory test of this test method in which eight operators in four laboratories tested six paints ranging in viscosity from 0.8 to 7.9 P, the

⁵ Supporting data are available from ASTM Headquarters. Request RR-D01-1035.

within-laboratory coefficient of variation was found to be 2.2 %, at 40 df. The between-laboratory coefficient of variation was found to be 6.9 % at 34 df. Based on these results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two test results, each the mean of two determinations obtained by the same operator, should be considered suspect if they differ by more than 6.3 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of

two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 19.9 % relative.

11.2 *Bias*—Bias has not been determined for this test method.

12. Index Terms

12.1 This test method is indexed under the following terms: viscosity—paints/related coatings/materials; viscometer—ICI cone/plate.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Specification for Artists' Oil, Resin-Oil, and Alkyd Paints¹

This standard is issued under the fixed designation D 4302; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

- 1.1 This specification establishes requirements for composition, physical properties, performance, and labeling of artists' oil, resin-oil, and alkyd paints.
- 1.2 This specification covers pigments, vehicles, and additives. Requirements are included for pigment identification, lightfastness, consistency, and drying time.
- 1.3 Table 1 lists some pigments meeting the lightfastness requirements in this specification. In order to identify other pigments that meet these requirements, instructions are given for test specimen preparation. Test methods for determining relative lightfastness are referenced.

Referenced Documents

- 2.1 *ASTM Standards*:
 - D 79 Specification for Zinc Oxide Pigments²
 - D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
 - D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
 - D 476 Specification for Titanium Dioxide Pigments²
 - D 602 Specification for Barium Sulfate Pigments²
 - D 1133 Test Method for Kauri-Butanol Value of Hydrocarbon Solvents⁴
 - D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁵
 - D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁵
 - D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁶
 - D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁵
 - D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints⁴
 - D 2369 Test Method for Volatile Content of Coatings⁵
 - D 2689 Practices for Testing Alkyd Resins²
 - D 4236 Practice for Labeling Art Materials for Chronic Health Hazards⁵
 - D 4303 Test Methods for Lightfastness of Pigments Used in Artists' Paints⁵

¹ This specification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

Current edition approved May 25, 1990. Published November 1990. Originally published as D 4302 - 83. Last previous edition D 4302 - 88.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁴ Annual Book of ASTM Standards, Vol 06.03.

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ Annual Book of ASTM Standards, Vol 14.02.

D 4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints⁵

E 284 Terminology of Appearance of Materials⁶

3. Terminology

3.1 Definitions:

3.1.1 *Colour Index Name*—consists of the category (type of dye or pigment), general hue, and an assigned number given to a colorant in the Colour Index⁷ as an international identification system.

3.1.1.1 *Discussion*—For example, the Colour Index Name of one phthalocyanine blue pigment is Pigment Blue 15 (PB 15).

3.1.2 *Colour Index Number*—a five-digit number given in the Colour Index that describes the chemical constitution of a colorant.

3.1.2.1 *Discussion*—For example, the Colour Index Number of one phthalocyanine blue pigment is 74160.

3.1.3 Refer to Terminology E 284 for appearance terms used in this specification.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *alkali refined oil*—triglyceride oil of vegetable origin that has been treated with alkali to reduce the free acidity by formation of water-soluble salts, subsequently removed by washing.

3.2.1.1 *Discussion*—An appreciable degree of free acidity may cause a greater development of yellowing in a dried film of oil. Most artists' oil paints are ground in alkali refined oil.

3.2.2 *alkyd paint*—paint containing a resin produced by combining a polybasic acid, a polyhydric alcohol, and the fatty acid of a drying vegetable oil. For this specification, the resin produced must be soluble in mineral spirits or turpentine.

3.2.3 *drier (siccative)*—a substance, usually an organometallic compound, that accelerates the rate of drying of an oil paint or oil medium.

3.2.4 *oil paint*—paint containing an alkali refined triglyceride drying oil of vegetable origin.

3.2.5 *resin-oil paint*—paint containing 90 weight % minimum of vehicle solids, vegetable drying oil, and 10 weight % maximum of vehicle solids replaced by gum or resin.

4. Significance and Use

4.1 This specification establishes quality requirements and provides a basis for common understanding among producers, distributors, and users.

4.2 It is not intended that all paints meeting the requirements be identical nor of uniform excellence in all respects.

⁷ *Colour Index*, 3rd ed., 5 Vols and Revisions, The Society of Dyers and Colourists, London, 1971-75. Available from the American Association of Textile Chemists and Colorists, P. O. Box 12215, Research Triangle Park, NC 27709.

TABLE 1 Suitable Pigments List

NOTE—Underlined Information and the lightfastness rating in the table shall be included on every label.

Key:

Lightfastness Category:

Lightfastness I Excellent Lightfastness

Lightfastness II Very Good Lightfastness

Abbreviations Used in Colour Index Names:

NR Natural Red
PB Pigment Blue
PBk Pigment Black
PBr Pigment Brown
PG Pigment Green
PO Pigment Orange
PR Pigment Red
PV Pigment Violet
PW Pigment White
PY Pigment Yellow

Pigment Notations:

(BS) Blue shade
(CC) Concentrated cadmium pigments may contain up to 15 % barium sulfate for color control. Cadmium-barium pigments contain a much higher amount of barium sulfate.
(DL) May darken in strong light
(LF) Lightfast type
(NA) Colour index name or number not assigned
(NR) Not rated as lightfastness I or II
... Not tested
(RS) Red shade
(SM) Sensitive to moisture in direct sunlight
(SS) Sensitive to hydrogen sulfide

Colour Index Name	Lightfastness Category			Common Name and Chemical Class	Colour Index Number
	Oil	Alkyd	Resin-Oil		
YELLOWS					
<u>PY 1, PY1.1</u>	II	II	...	Arylide Yellow G, with option of adding the name Hansa Yellow Medium, arylide yellow	11580
<u>PY 3</u>	II	II	...	Arylide Yellow 10G, with option of adding the name Hansa Yellow Light, arylide yellow	11710
<u>PY 35</u>	I	...	...	Cadmium Yellow Light, concentrated cadmium zinc sulfide (CC) (SM)	77205
<u>PY 35.1</u>	I	...	...	Cadmium-Barium Yellow Light, cadmium zinc sulfide coprecipitated with barium sulfate (SM)	77205.1
<u>PY 37</u>	I	I	...	Cadmium Yellow Medium or Deep, concentrated cadmium sulfide (CC) (SM)	77199
<u>PY 37.1</u>	I	...	...	Cadmium-Barium Yellow Medium or Deep cadmium sulfide coprecipitated with barium sulfate (SM)	77199.1
<u>PY 40</u>	II	...	...	Aureolin, with option of adding the name Cobalt Yellow, potassium cobaltinitrite	77357
<u>PY 41</u>	I	...	...	Naples Yellow, lead antimonate (SS)	77589
<u>PY 42</u>	I	...	I	Mars Yellow, with option of adding the name Yellow Iron Oxide, synthetic hydrated iron oxide	77492
<u>PY 42</u>	I	...	I	Mars Orange, synthetic hydrated iron oxide	77492
<u>PY 43</u>	I	I	I	Yellow Ochre, natural hydrated iron oxide	77492
<u>PY 53</u>	I	...	I	Nickel Titanate Yellow, oxides of nickel, antimony and titanium	77788
<u>PY 65</u>	I	...	...	Arylide Yellow RN, with option of adding Hansa Yellow RN, arylide yellow	11740
<u>PY 73</u>	I	...	...	Arylide Yellow GX, with option of adding the name Hansa Yellow GX, arylide yellow	11738
<u>PY 74(LF)</u>	I	...	...	Arylide Yellow 5GX, with option of adding Hansa Yellow 5GX, arylide yellow	11741
<u>PY 83 HR 70</u>	I	...	I	Diarylide Yellow HR70, diarylide yellow	21108
<u>PY 97</u>	I	...	...	Arylide Yellow FGL, arylide yellow	11767
<u>PY 98</u>	II	...	...	Arylide Yellow 10GX, with option of adding the name Hansa Yellow 10GX, arylide yellow	11727
<u>PY 108</u>	I	...	I	Anthrapyrimidine Yellow, anthrapyrimidine	68420
<u>PY 109</u>	I	...	...	Isolindolinone Yellow G, tetrachloroisolindolinone	NA
<u>PY 110</u>	I	...	I	Isolindolinone Yellow R, tetrachloroisolindolinone	56280
<u>PY 112</u>	I	...	...	Flavanthrone Yellow, flavanthrone	70600
<u>PY 138</u>	I	...	I	Quinophthalone Yellow, quinophthalone	NA
<u>PY 139</u>	I	...	...	Isolindoline Yellow, isolindoline	NA
<u>PY 150</u>	I	...	...	Nickel Azo Yellow, nickel complex azo	NA
<u>PY 151</u>	I	...	I	Benzimidazolone Yellow H4G, benzimidazolone	13980
<u>PY 153</u>	I	...	I	Nickel Dioxine Yellow, dioxine yellow nickel complex	NA
<u>PY 154</u>	I	...	I	Benzimidazolone Yellow H3G, benzimidazolone	11781
<u>PY 175</u>	I	...	...	Benzimidazolone Yellow H6G, benzimidazolone	11784

TABLE 1 Continued

Colour Index Name	Lightfastness Category			Common Name and Chemical Class	Colour Index Number
	Oil	Alkyd	Resin-Oil		
ORANGES					
PO 5	II	...	...	Dinitraniline Orange, dinitraniline (SM)	12075
PO 20	I	I	I	Cadmium Orange, concentrated cadmium sulfo-selenide (CC)	77202
PO 20:1	I	...	...	Cadmium-Barium Orange, cadmium sulfoselenide coprecipitated with barium sulfate	77202:1
PO 23	I	...	...	Cadmium Vermilion Orange, concentrated cadmium mercury sulfide (CC)	77201
PO 23:1	I	...	...	Cadmium-Barium Vermilion Orange, cadmium mercury sulfide coprecipitated with barium sulfate	77201:1
PO 36	I	...	...	Benzimidazolone Orange HL, benzimidazolone	11780
PO 43	I	...	...	Perinone Orange, perinone	71105
PO 48	I	...	...	Quinacridone Gold, quinacridone	NA
PO 49	I	...	...	Quinacridone Deep Gold, quinacridone	NA
PO 60	I	...	...	Benzimidazolone Orange HGL, benzimidazolone	11782
PO 62	I	...	...	Benzimidazolone Orange H5G, monoacetolone	11775
REDS					
PR 5	II	...	...	Naphthol ITR, naphthol ITR	12490
PR 7	I	...	I	Naphthol AS-TR, naphthol AS-TR	12420
NR 9	II	...	...	Natural Rose Madder, natural madder	75330
PR 9	II	I	...	Naphthol AS-OL, naphthol AS-OL	12460
PR 14	II	...	...	Naphthol AS-D, naphthol AS-D	12380
PR 88 MRS ^A	I	...	I	Thioindigold Violet, thioindigold	73312
PR 101	I	...	...	Indian Red, synthetic red iron oxide (bluish hue)	77491
PR 101	I	...	...	Light or English Red Oxide, synthetic red iron oxide (yellowish hue)	77491
PR 101	I	...	...	Mars Red, with option of adding the name Red Iron Oxide, synthetic red iron oxide	77491
PR 101	I	...	I	Mars Violet, with option of adding the name Violet Iron Oxide, synthetic iron oxide (violet hue)	77015
PR 101	I	...	...	Venetian Red, synthetic iron oxide (yellowish hue)	77491
PR 102	I	...	...	Light Red, calcined yellow ochre	77492
PR 106	I	...	...	Vermilion, mercuric sulfide (DL)	77766
PR 108	I	I	I	Cadmium Red Light, Medium, or Deep, concentrated cadmium-seleno sulfide (CC)	77202:1
PR 108:1	I	...	...	Cadmium-Barium Red Light, Medium or Deep, cadmium seleno-sulfide coprecipitated with barium sulfate	77202:1
PR 112	II	...	...	Naphthol AS-D, naphthol AS-D	12370
PR 113	I	...	...	Cadmium Vermilion Red Light, Medium or Deep, concentrated cadmium mercury sulfide (CC)	77201
PR 113:1	I	...	...	Cadmium-Barium Vermilion Red Light, Medium or Deep, cadmium mercury sulfide coprecipitated with barium sulfate	77201:1
PR 119	I	...	...	Naphthol Red, naphthol	NA
PR 122	I	I	I	Quinacridone Magenta, γ quinacridone	73915
PR 123	I	...	...	Perylene Vermilion, perylene	71145
PR 149	I	...	...	Perylene Red, perylene	77137
PR 166	II	...	...	Brominated Anthranthrone, brominated anthranthrone	58300
PR 170 F3RK-70	II	...	...	Naphthol Red, naphthol carbamide	12475
PR 170 F5RK	II	...	...	Naphthol Crimson, naphthol carbamide	12475
PR 175	I	...	...	Benzimidazolone Maroon, benzimidazolone	71513
PR 177	...	...	I		
PR 178	...	...	I		
PR 179	I	...	...	Perylene Maroon, perylene	71130
PR 188	I	...	...	Naphthol AS, naphthol AS	12467
PR 190	I	...	...	Perylene Red, perylene	71140
PR 192	I	...	...	Quinacridone Red, γ quinacridone red	NA
PR 194	I	...	...	Perinone Red Deep, perinone	71100
PR 207	I	...	...	Quinacridone Scarlet, quinacridone red	NA
PR 242	...	...	I		
PV 19	I	...	...	Quinacridone Red, γ quinacridone red	73900
PURPLES					
PV 14	I	...	I	Cobalt Violet, cobalt phosphate, cobalt ammonium phosphate	77360
PV 15	I	...	I	Ultramarine Red, complex silicate of sodium and aluminum with sulfur	77007
PV 15	I	...	I	Ultramarine Violet, complex silicate of sodium and aluminum with sulfur	77007
PV 16	I	...	...	Manganese Violet, manganese ammonium pyrophosphate	77742
PV 19	I	I	I	Quinacridone Violet, quinacridone violet b	73900
PV 23BS(I)	II	...	...	Dioxazine Purple, carbazole dioxazine	51319
PV 23RS(I)	I	I	...	Dioxazine Purple, carbazole dioxazine	51319
PV 31	I	...	...	Isocyanthrone Violet, isocyanthrone	60010

TABLE 1 Continued

Colour Index Name	Lightfastness Category			Common Name and Chemical Class	Colour Index Number
	Oil	Alkyd	Resin-Oil		
BLUES					
PB 15			...	Phthalocyanine Blue, copper phthalocyanine	74160
PB 16		...		Phthalocyanine Blue, metal free phthalocyanine	74100
PB 22		...	...	Indanthrone Blue, indanthrone	69810
PB 27				Prussian Blue, with the option of adding the name Millori Blue, ferriammonium ferrocyanide	77510
PB 28				Cobalt Blue, oxides of cobalt and aluminum	77346
PP 29				Ultramarine Blue, complex silicate of sodium and aluminum with sulfur	77007
PB 33		...		Manganese Blue, barium manganate with barium sulfate	77112
PB 35		...		Cerulean Blue, oxides of cobalt and tin	77368
PB 36		...	...	Cerulean Blue, Chromium, oxides of cobalt and chromium	77343
PB 60		...		Indanthrone Blue, indanthrone	69800
GREENS					
PG 7				Phthalocyanine Green, chlorinated copper phthalocyanine	74260
PG 10		...	...	Green Gold, with option of adding the name Nickel Azo Yellow, nickel chelated azo	12775
PG 17		...		Chromium Oxide Green, anhydrous chromium sesquioxide	77288
PG 18			...	Viridian, hydrous chromium sesquioxide	77289
PG 19		...		Cobalt Green, oxides of cobalt and zinc	77335
PG 23				Green Earth or Terra Verde, natural green	77009
PG 36		...		Phthalocyanine Green, chlorinated and brominated phthalocyanine	74265
PG 50		...		Light Green Oxide, oxides of nickel, cobalt and titanium	77377
BROWNS					
PBr 6		...		Mars Brown, with option of adding the name Brown Iron Oxide, synthetic brown iron oxide or mixtures of synthetic iron oxides	77499
PBr 7				Burnt Sienna, calcined natural iron oxide	77492
PBr 7			...	Burnt Umber, calcined natural iron oxide containing manganese	77492
PBr 7			...	Raw Sienna, natural iron oxide	77492
PBr 7			...	Raw Umber, natural iron oxide containing manganese	77492
BLACKS					
PBk 6			...	Lamp Black, nearly pure amorphous carbon	77266
PBk 7		...	...	Carbon Black, nearly pure amorphous carbon	77266
PBk 9				Ivory Black, amorphous carbon produced by charring animal bones	77267
PBk 11		...		Mars Black, with option of adding the name Black Iron Oxide, synthetic black iron oxide	77499
WHITES					
PW 1			...	Flake White, basic lead carbonate (SS)	77597
PW 4		...	...	Zinc White, zinc oxide	77947
PW 6			...	Titanium White, titanium dioxide (rutile or anatase) with option of including some barium sulfate or zinc oxide	77891

^A Applies only to Permanent Red Violet MRS, product of American Hoechst Corp., Coventry, RI 02816. Pigments described as thioindigoids have varying degrees of lightfastness.

Variations in manufacture, not covered by this specification, may cause some artists to prefer one brand over another, either of which may be acceptable under this specification.

5. Labeling Requirements

5.1 Pigment(s) Identification:

5.1.1 Every label shall include for each pigment contained in the paint (1) the information underlined in Table 1 (which includes the Common Name, Colour Index Name, and any additional terms necessary to identify the form of the pigment) and (2) the appropriate Lightfastness Category.

5.1.2 The complete pigment identification given in Table 1, which also includes the Colour Index Number and a simple chemical description, shall be given in an appropriate producer publication. Manufacturers are encouraged to put this complete identification on the container label when label size permits.

5.1.3 The Common Name shall be placed on the front of the label and shall be the name of the paint except as described in 5.1.5 and 5.1.6. Other identification may be

placed elsewhere on the container.

5.1.4 The Colour Index name may be spelled out in full or abbreviated depending on the size of the label. Examples: Pigment Blue 15, or Pig. Blue 15 or PB 15.

5.1.5 *Substituted Pigment*—In the case of substituted pigments, the word "Hue" in equal size letters shall follow in the title, on the front of the tube, immediately after the name of the pigment that has been simulated. Directly below the title, the Common Name of the significant pigment used shall be given in letters no less than the next type size smaller than the title. For example:

COBALT BLUE HUE
(ULTRAMARINE BLUE).

5.1.6 Proprietary names or optional names may be used provided the Common Name(s) given in Table 1 appears on the front of the label directly under the proprietary or optional name in letters no less than the next type size smaller than the proprietary or optional name.

5.1.7 *Mixed Pigments*—Artists' paints containing more than one colorant comply with this specification if all

colored pigments used are on the suitable pigment list (Table 1) and provided the mixture itself has passed all other test requirements in this specification. The lightfastness category shall be that of the least lightfast pigment. This lightfastness category may be changed if these paints are tested for lightfastness in accordance with Test Methods D 4303 and results indicating a different category are submitted to ASTM Subcommittee D01.57 for evaluation.

5.2 Provide on the label:

5.2.1 *Artists' Oil Paints*—Vegetable origin of the oil and method of refinement.

NOTE 1—The type of oil can be identified in accordance with Test Method D 2245.

5.2.2 *Artists' Alkyd Paints*—Type of fatty acid used. If free vegetable oil is used in combination with an alkyd resin, declare this also on the label. State if compatible with artists' oil paints.

NOTE 2—The type of alkyd can be identified in accordance with Practices D 2689.

5.2.3 *Artists' Resin-Oil Paints*—Vegetable origin and method of refinement of the oil and type of resin or gum. If the colors in a resin-oil paint line contain more than one, or different, gums or resins, the identification on the individual labels may uniformly include all of the gums or resins used in the paint line. Example: "Damar or Mastic Gum." State if compatible with artists' oil paints.

NOTE 3—The type of oil can be identified in accordance with Test Method D 2245.

5.3 *Lightfastness*—The label shall contain the word "lightfastness" followed by the appropriate rating, I or II, as given for each pigment in Table 1.

5.3.1 Lightfastness I pigments, when made into paint specimens as described in Section 8 and exposed, tested, and rated in accordance with Test Methods D 4303, shall have a color difference (ΔE^*_{ab}) of 4 or less CIELAB units between the specimens measured before and after exposure.

5.3.2 Lightfastness II pigments, when made into paint specimens as described in Section 8 and exposed, tested, and rated in accordance with Test Methods D 4303, shall have a color difference (ΔE^*_{ab}) of more than 4.0 but not more than 10 CIELAB units between the specimens measured before and after exposure.

5.3.3 Pigments were placed in a lightfastness category on the basis of either known historical performance in art works or the ratings from four lightfastness tests conducted as described in Test Methods D 4303. Results from further tests on these, or other pigments, are solicited by Subcommittee D01.57.

5.3.3.1 The lightfastness category of a pigment shall be changed if results from several further tests conducted in accordance with Test Methods D 4303 and approved by ASTM Subcommittee D01.57, establish a different lightfastness category than the one given in Table 1.

5.3.3.2 Additional pigments shall be placed in Table 1 if they have been tested for lightfastness in accordance with Test Methods D 4303 and the test results submitted to ASTM Subcommittee D01.57 for evaluation, provided the results demonstrate that the pigments have the lightfastness ratings required for Lightfastness I or Lightfastness II, as described above.

5.3.4 For information and to establish nomenclature, pigments in Lightfastness III category are given in Table X1.1 in Appendix X1, but are not to be used in paint conforming to this specification. These pigments have a color difference before and after exposure of more than 8.0 but not more than 16.0 CIELAB units.

5.4 *Contents*—To be expressed in volumetric measure as required by law.

5.5 *Toxicity*—All products and labeling must conform to the Federal Hazardous Substances Act and to Practice D 4236.

5.6 *Statement of Conformance*—"Conforms to ASTM Specification D 4302," or "Conforms to ASTM D 4302," or "Conforms to the quality requirements of ASTM D 4302." This statement may be combined with other conformance statements, such as, "Conforms to the quality and health requirements of ASTM Specification D 4302 and Practice D 4236."

5.7 *Address*—Include on the label (1) the name and address of the manufacturer or importer and (2) the country of manufacture.

6. Quality Assurance for Artists' Oil Paints and Artists' Resin-Oil Paints

6.1 *Conditions Not Covered in This Specification that Affect Artists' Oil and Resin-Oil Paints:*

6.1.1 *Substrate*—Factors such as the texture, gloss, effective pH, porosity, chemical composition, and condition of the substrate will affect gloss, gloss uniformity, drying time, adhesion, and the flexibility of the dried film.

6.1.2 *Environmental Conditions*—Factors such as temperature, humidity, air flow, and light conditions affect application properties, film formation, drying time, and adhesion.

6.1.3 *Storage*—With aging and elevated temperatures, there may be a change in consistency and a separation of oil from the paste paint.

6.2 *Vehicles*—Only vegetable drying oils shall be used in artists' oil paints. Resin-oil paints shall contain 90 weight % minimum of vehicle solids, vegetable drying oil (see Note 1), and 10 weight % maximum of vehicle solids replaced by gum or resin.

6.3 *Pigments*—Pigments to be used in oil paints shall be limited to those listed in the column labeled "Oil" and pigments to be used in resin-oil paints shall be limited to those listed in the column labeled "Resin-Oil" in Table 1. Their lightfastness rating shall be the numeral given in the same row. Those pigments listed as "not tested" shall not be used.

6.4 *Driers*—Driers may be used in minimal amounts in paints that contain a pigment which has a retarding effect on the drying of oil. This is allowed so these paints can conform to the drying requirements of this specification.

6.5 *Inerts*—Minimal amounts of inert pigments may be used to produce desirable working qualities and consistency, to prevent separation, to develop chromatic properties, or to ensure the durability of the paint film.

6.6 *Preparation of Sample*—Empty the contents of a previously unopened container onto a glass slab and mix thoroughly with a spatula to a homogeneous sample.

6.7 *Nonvolatile*—The nonvolatile content shall not be less than 97 weight % for the oil paints and 90 weight % for the

resin-oil paints, as determined by Test Method D 2369.

6.8 *Coarse Particles*—Paints shall be free of oversize particles and shall form a uniform film. The maximum content of coarse particles shall be 1 weight % as determined by Test Methods D 185.

6.9 *Fineness of Dispersion*—On a glass plate, using a spatula, mix the paint with an equal volume of linseed oil until homogeneous. If the paint is very thick, it may be necessary to add a minimum amount of mineral spirits or turpentine to make the paint sufficiently fluid to read the gage accurately. Determine the fineness of dispersion by Test Method D 1210. The maximum allowable grind reading is 1.5 mils (40 μ m).

6.10 *Consistency*—The paste type of paint shall not flow or level when applied with a palette knife.

6.11 *Drying*—Using a film applicator with a 3-mil (75- μ m) clearance, make a uniform drawdown on a lacquer-sealed panel. At a relative humidity of 50 to 75 % and a temperature of 65 to 80°F (18 to 27°C), the dust-free drying time, as determined by Test Methods D 1640, shall not be more than ten days.

6.12 Tinting strength requirements will be included in this specification as appropriate tinting strength standards for individual pigments are established. Test Method D 387 may be used to determine the tinting strength of pigments or paints when all ingredients are known. Test Method D 4838 can be used to determine the relative tinting strength of chromatic paints containing a single pigment and the same vehicle but where other ingredients are unknown.

7. Quality Assurance for Artists' Alkyd Paints

7.1 *Conditions Not Covered in This Specification that Affect Artists' Alkyd Paints:*

7.1.1 *Substrate*—See 6.1.1.

7.1.2 *Environmental Conditions*—See 6.1.2.

7.1.3 *Storage*—With aging and elevated temperatures, there may be a change in consistency and possible solvent loss. Some separation of the medium is also possible.

7.2 *Vehicles*—Only alkyds that are soluble in mineral spirits or turpentine may be used in artists' alkyd paints. Free vegetable drying oils may be included in artists' alkyd paints up to 25 weight % of the vehicle solids.

7.2.1 The amount of yellowing in a vehicle should not exceed the yellowing of a linseed oil paint. To assess excessive yellowing of the alkyd vehicle or alkyd vehicle/drying vegetable oil blend used in alkyd artists' paints, put the vehicle including its usual drier and all additives in the following formula:

	Weight %
Alkyd vehicle under test including driers	22
Mineral spirits	11
Rutile titanium white* (conforming to Type II of Specification D 476)	30
China clay	14
Blanc fixe (conforming to Specification D 602)	22
Anti-skinning agent	1

NOTE 4—More or less mineral spirits may be used for ease of brushing.

* DuPont R900 Rutile titanium dioxide, manufactured by E. I. du Pont de Nemours & Co., 1007 Market St., Wilmington, DE 19898, has been found satisfactory for this purpose.

7.2.2 Prepare a white oil paint using the same titanium white dispersed in alkali refined linseed oil. Make specimens from the alkyd and oil paints as directed in Test Methods D 4303, and expose them to light filtered through glass using Test Method 1 and either Test Method 2 or Test Method 3 as described in the Procedure section of Test Methods D 4303.

7.2.3 Following exposure check that the alkyd specimens are the same or less yellow than the oil specimens exposed by the same test method. (Use Practice D 1729 or Test Method D 2244 to determine amount of yellowing.)

7.3 *Pigments*—Pigments shall be limited to those recommended for use in alkyd paints in the column labeled "Alkyd" in Table 1. Their lightfastness rating shall be the numeral given in the same row. Those pigments listed as "not tested" shall not be used.

7.4 *Driers*—Minimal amounts of driers may be used to allow paints to conform to the drying requirements of this specification.

7.5 *Inerts*—See 6.5.

7.6 *Sampling*—See 6.6.

7.7 *Coarse Particles or Foreign Matter*—See 6.8.

7.8 *Fineness of Dispersion*—On a glass plate, using a spatula, mix the paint with linseed oil in a one-to-one volumetric ratio until homogeneous. If the paint is very thick, it may be necessary to add a minimum amount of turpentine or mineral spirits to make the paint sufficiently fluid to read the gage accurately. Determine the fineness of dispersion in accordance with Test Method D 1210. The maximum allowable grind reading is 1.5 mils (40 μ m).

7.9 *Drying*—Using a film applicator with a 3-mil (75- μ m) clearance, make a uniform drawdown on a lacquer-sealed panel. At a relative humidity from 50 to 75 % and a temperature from 65 to 80°F (18 to 27°C), the dust-free drying time shall be not more than 2 days or less than 30 min.

8. Lightfastness Determination

8.1 If a pigment is not listed in Table 1, test specimens of a paint containing the pigment shall be prepared. These test specimens shall be tested in accordance with the requirements for exposure and evaluation given in Test Methods D 4303.

NOTE 5—A report of the results of these tests may be submitted to Subcommittee D01.57 for inclusion of the pigments in Table 1. The report shall include information on test conditions and instruments used and shall be accompanied by test specimens, which will be returned.

8.2 Materials:

8.2.1 *Aluminum Exposure Panels*⁹ 3 by 6 in. (75 by 150 mm).

8.2.2 *Posterboard*, lightweight, approximately 20 mils (0.5 mm) thick, having a glossy finish on one side.

8.2.3 *White Soya Alkyd Enamels* used to prepare white ground coats.

8.2.4 *White Artists' Paints*—Depending on the vehicle in which the pigments are to be tested:

8.2.4.1 Titanium dioxide yellowing-resistant oil paint,

⁹ The No. A-36 Aluminum Panel, manufactured by The Q-Panel Co., 26200 First St., Cleveland, OH 44145, has been found satisfactory for this purpose.

8.2.4.2 Titanium dioxide yellowing-resistant alkyd paint,

8.2.4.3 Titanium dioxide yellowing-resistant resin-oil paint.

8.2.5 *Spray Apparatus*, for applying ground coats.

8.2.6 *Paint Brush*, 2.5 in. (60 mm) wide for applying ground coats if spray apparatus is not available.

8.2.7 *Soft Hair Brush*, 1 in. (25 mm) wide for applying ground coats to aluminum panels. An oxhair artists' "stroke" brush is suitable.

8.3 *Summary of Method:*

8.3.1 Pigments to be tested are dispersed in the appropriate vehicle and diluted with a standard mixing white of the same type of vehicle until the spectrophotometric measurement of the dried film reads from 35 to 45 % reflectance at the wavelength of maximum absorption for that pigment. Prepared artists' paints of known composition may be tested.

8.4 *Specimen Preparation:*

8.4.1 *Ground Coats:*

8.4.1.1 For two ground coats to be used under oil, alkyd, and resin-oil paints, prepare the following enamel:

	Weight %
Medium oil length soya alkyd, 50 % nonvolatile ¹⁰	20
Rutile titanium chloride ⁸ (conforming to Type II of Specification D 476)	40
Blanc fixe (conforming to Specification D 602)	40
Driers 0.15 % zinc and 0.15 % zirconium as metal on the alkyd nonvolatile	
Sufficient mineral spirits for milling	

8.4.1.2 Mill to a Hegman fineness of 7 as measured by Test Method D 1210. Thin with mineral spirits to appropriate viscosity for spraying or for flow coating by brush.

NOTE 6—These soya alkyd enamels are used for the ground coats because of their color stability, nonabsorbency, adhesion of the specimen coats under humid conditions, and freedom from blistering that occur with an acrylic ground coat under high humidity.

8.4.2 *Application of Ground Coats:*

8.4.2.1 Degrease aluminum substrates before applying the ground coat.

8.4.2.2 Under oil, alkyd, and resin-oil paints, apply two coats of the enamel described in 8.4.1.1 to the aluminum or posterboard supports, either by spraying or by flow coating with a brush. To flow coat the aluminum panels for sun exposure use a 1-in. (25-mm) soft-hair brush. Coat the posterboard to be used for laboratory exposure on the less absorbent, glossy side.

NOTE 7—Coating the posterboard is most conveniently done on the sheet before cutting to size.

8.4.2.3 Allow a minimum of five days drying time following the first coat and two weeks or more before applying the specimen to be tested.

8.4.3 *Mixing Whites for Dilution of Colors:*

8.4.3.1 For oil paints use the following white:

	Weight %
Alkali refined safflower oil	22.5
Aluminum distearate	2.0
Rutile titanium dioxide ⁸ (conforming to Type II of Specification D 476)	30.0
Blanc fixe (conforming to Specification D 602)	39.5
Zinc oxide (conforming to Specification D 79)	6.0

8.4.3.2 Mill to a Hegman fineness of 7 as measured by Test Method D 1210.

8.4.3.3 Keep the viscosity of the mixing white to 250 P or slightly lower.

8.4.3.4 For diluting alkyd paints use the following white containing two alkyd resins:

	Weight %
Medium oil length soya alkyd, ¹⁰ 62 % oil length, 70 % nonvolatile, white spirit as volatile	10
Medium oil length soya alkyd, 62 % oil length, 51 % nonvolatile, mineral spirits as volatile	12
Mineral spirits	11
Rutile titanium dioxide ⁸ (conforming to Type II of Specification D 476)	30
Blanc fixe (conforming to Specification D 602)	16
China clay	14
Anti-skinning agent	1
Zinc oxide (conforming to Specification D 79)	6

8.4.3.5 Mill to a Hegman fineness of 7 as measured by Test Method D 1210.

8.4.3.6 For diluting resin-oil paints, use the following white:

	Weight %
Alkali refined safflower oil	20.7
Aluminum distearate	2
Damar resin dissolved in 3 % mineral spirits (Note 8)	1.8
Rutile titanium dioxide ⁸ (conforming to Type II of Specification D 476)	30
Blanc fixe (conforming to Specification D 602)	39.5
Zinc oxide (conforming to Specification D 79)	6

NOTE 8—The kauri-butanol value of the mineral spirits shall be ≥ 40 as determined by Test Method D 1133.

8.4.3.7 Mill to a Hegman fineness of 7 as measured by Test Method D 1210.

8.4.4 *Preparation of Test Paints:*

8.4.4.1 The pigment to be tested may be milled in a concentrated paste in a resin-oil vehicle. If a prepared resin-oil artists' paint of known composition is available, it may be used for this test.

8.4.4.2 Dilute the pigment paste or paint with the white given in 8.4.3.6 until the spectrophotometric measurement of the dried film shows 35 to 45 % relative reflectance at the wavelength of maximum absorption for that pigment. The wavelength of maximum absorption is located at the point of lowest reflectance on the spectral curve between 420 and 620 nm. If using a tristimulus filter colorimeter, the lowest of the three filter readings is the region of maximum absorption and the dilution should be adjusted so that a reading of 35 to 45 % reflectance is obtained with this filter. The diffuse white reference standard for all measurements should have an absolute reflectance between 97 and 100 %.

8.4.4.3 To obtain this reflectance, use the Kubelka-Munk Single Constant Method described in Appendix X2 or use a trial and error method.

8.4.4.4 Make instrumental readings with the specular reflectance included to minimize the effect on readings of

¹⁰ The following alkyd resins have been found satisfactory for this purpose: All No. 51-5196; McWhorter, DURAMAC 2409; Reichhold, No. 11-035; Amer-Kellog, AROPLAZ 1082 M50.

any change in gloss and to minimize the effect of brush strokes.

8.4.4.5 Use an applicator with a 6-mils (150- μ m) aperture to make a minimum size drawdown, $1\frac{1}{2}$ by $1\frac{1}{2}$ in. (40 by 40 mm), or other minimum size appropriate for the viewing area of the instrument used. Return all recoverable paint to the batch to allow for repeat mixes and measurements.

8.4.4.6 For oil, alkyd, or resin-oil paint, an initial amount of 40 g of white paint is sufficient if specimens are read wet and recoverable paint is returned to the batch. If the instrument provides a way to read wet paint behind glass, and specimens measured this way fall within the accepted range when dry, this method can be used.

NOTE 9—Another suggested method for reading oil paints while wet is to cut from 0.013-mm thick kitchen plastic wrap (such as Saran wrap) the minimum size piece that will protect the instrument and then die-cut a hole in the proper location $\frac{1}{4}$ in. (3.2 mm) smaller than the instrument aperture. Rubbing the plastic several times with a paper towel or tissue imparts sufficient static charge that the protective plastic adheres to the instrument enabling the operator to center the hole on the aperture. The wet oil paint drawdown can then be placed in position, taking care not to disturb the plastic shield.

8.4.4.7 For the initial weights of white stated in 8.4.3.1, 8.4.3.4 and 8.4.3.6 the weighing must be accurate to 0.05 g.

8.4.5 Application of Paints to Panels:

8.4.5.1 Prepare four specimen panels on appropriate substrates for each pigment under test. Two are used in the first lightfastness tests and two are retained in subdued light, one for visual comparisons with the exposed panels and one in case a third test is needed to supplement results from the first two tests, as described in Procedure section Test Methods D 4303.

8.4.5.2 Apply the test paints by brush to the aluminum or posterboard exposure panels described in 8.2, depending on

whether exposure is to be under glass to the sun or to laboratory apparatus.

8.4.5.3 Using the No. 12 artists' flat bristle brush, brush the panels lengthwise, then crosswise, and again lengthwise, this time with a light touch to produce a film as smooth as possible. The mixing whites must be fluid enough to facilitate leveling, but not have excess vehicle that can affect the test. Do not use thinners. Oil-paint brushouts retain minor striations. Apply two coats to all specimen panels to achieve complete opacity.

8.4.5.4 Allow specimens to dry between coats as described in the Procedure section (Dry-Through or Dry-to-Handle Time) of Test Methods D 1640. After recoating, allow specimens to dry hard, as described in the Procedure section (Dry-Hard Time) of Test Methods D 1640, before measuring them prior to exposure. General guidelines for drying times are one to two months to dry hard for oil and resin-oil paints; three days between coats for alkyd paints; and five days after recoating.

8.4.5.5 When testing alkyd paints, prepare and expose a specimen of the mixing white along with the colored specimens diluted with this white. Since the purpose of these lightfastness tests is to evaluate any color change in the pigments, if the white yellows appreciably during exposure, adjust any color change between unexposed and exposed colored specimens to eliminate the effect of this yellowing.

9. Exposure

9.1 Conduct exposure tests in conformance with Test Methods D 4303. Assign lightfastness categories as described in Section 8 of Test Methods D 4303.

10. Keywords

10.1 artists' alkyd paints; artists' oil paints; artists' resin-oil paints; lightfastness; quality requirements; test specimens

APPENDICES

(Nonmandatory Information)

X1. LIGHTFASTNESS III

X1.1 The pigments in Table X1.1 are not sufficiently lightfast to be used in paints that conform to this specification. These pigments are listed here solely to establish

common terminology. They may be satisfactory when used full strength (without dilution) or with extra protection from exposure to light.

TABLE X1.1 Lightfastness III

Colour Index Name	Lightfastness Category		Common Name and Chemical Class	Colour Index Number
	Oil	Alkyd		
PR 83	III	III	Alizarin Crimson; synthetic 1:2 dihydroxyanthraquinone on alumina base	58000
PR 83	III	...	Rose Madder, Alizarin, synthetic 1:2 dihydroxyanthraquinone on alumina base, more dilute than Alizarin Crimson	58000
PR 146	III	...	Naphthol Red, naphthoic arylide	12485
PR 181	III	...	Thioindigoid Magenta, thioindigoid	73360

X2. KUBELKA-MUNK SINGLE CONSTANT METHOD FOR PREPARING PAINT FILMS $40 \pm 5\%$ REFLECTANCE

X2.1 Prepare a mixture of white and colorant in proportions estimated to give a paint having a reflectance of 30 to 40 % at the wavelength of maximum absorption. Calculate the concentration of colorant in white in this batch.

$$C_c = W_c / W_w + W_c$$

where:

C_c = concentration of colorant, and
 W_w and W_c = weight of white and colorant, respectively, used in the batch.

Draw or brush a film of this paint over contrast paper at a thickness sufficient to give complete visual hiding.

X2.1.1 After allowing the film to dry, measure the reflectance of this film at the wavelength of maximum absorption with a spectrophotometer or, if using a colorimeter, the reflectance with the filter that gives the lowest reading (Note X2.1). Calculate the Kubelka-Munk Single Constant Value of this reflectance as follows:

$$(K/S)_m = [1 - (R_m - 0.04)]^2 / 2(R_m - 0.04)$$

where:

$(K/S)_m$ = Kubelka-Munk Value,
 R_m = reflectance measurement of the mixture, made with the specular component included expressed as a decimal value, and
 0.04 = decimal value correction for surface reflectance.

NOTE X2.1—To speed drying, accelerated methods may be used. It is recommended that water-based acrylic paints be warmed to 50 to 55°C or 2 h after 1 h of air drying before measuring the reflectance. Placing the drawdowns of oil paints near fluorescent lamps, for example in the fluorescent light exposure racks described in Annex A1 of Test Methods D 4303, will speed their drying time.

X2.1.2 Calculate the Kubelka-Munk absorption coefficient for unit concentration as follows:

$$K_c = (K/S)_m / C_c$$

where K_c = unit absorption coefficient.

X2.2 Based on the results of the trial mixture, calculate the concentration of colorant in white required to give the desired 35 to 45 % reflectance at the wavelength of maximum absorption as follows:

$$C_d = (K/S)_{40\%} / K_c = 0.4500 / K_c$$

where C_d = concentration of colorant in white predicted to give a paint with the desired 35 to 45 % reflectance value.

X2.3 Adjust the concentration of the trial batch so that the concentration of colorant equals C_d . Determine the changes in colorant concentration, ΔC , as follows:

$$\Delta C = C_d - C_c$$

These changes are positive if the trial batch has a reflectance greater than 40 % and negative if the reflectance is less than 40 %. If ΔC is positive, add more colorant to the trial batch to bring the concentration of colorant to C_d . If ΔC is negative, add more white to bring the concentration of colorant to C_d . Because it may be necessary to add large amounts of white to correct for concentrations of colorant giving reflectance values far below 40 %, it is usually best to prepare a trial batch expected to give a ΔC value that is moderately positive or only slightly negative.

X2.3.1 For the case where ΔC is positive, determine the weight of colorant needed to prepare a second batch as follows:

$$\Delta W_c = W_c - C_d(W_w + W_c) / C_d - 1 \times (L.F.)$$

where:

ΔW_c = weight of colorant to be added to the trial batch to obtain a corrected batch with a colorant concentration of C_d , and

L.F. = loss factor = weight of paint from trial batch used to prepare the corrected batch/weight of paint prepared as trial batch.

The loss factor, L.F., allows for paint removed to prepare drawdowns, loss by transfer to different containers, etc. This term may be neglected if losses are small compared to the batch size. The other terms are as defined in the previous sections of this appendix.

X2.3.2 For the case where ΔC is negative, the weight of white needed to prepare a corrected batch corresponds to,

$$\Delta W_w = W_c - C_d(W_w + W_c) / C_d \times (L.F.)$$

where ΔW_w = weight of white needed to adjust the colorant concentration in the batch to C_d . Other terms are as defined above or in previous sections of this appendix.

X2.4 Prepare a paint film from the adjusted batch. After drying, measure the reflectance at the wavelength of maximum absorption. If this value does not fall within 35 to 45 %, repeat X2.3 through X2.4, where C_d now becomes the C_c for the next batch correction. Normally only a single iteration is necessary and a second batch correction will not be needed.

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Standard Test Methods for Lightfastness of Pigments Used in Artists' Paints¹

This standard is issued under the fixed designation D 4303; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 Three test methods consisting of exposure to light (natural sunlight or simulated sunlight) filtered through glass are described as follows:

- 1.1.1 *Test Method A*—Exposure under glass to the sun,
- 1.1.2 *Test Method B*—Exposure to irradiance from artificial daylight fluorescent lamps, and
- 1.1.3 *Test Method C*—Exposure in xenon-arc lightfastness apparatus.

1.2 These test methods are used to approximate the color change that can be expected over time in pigments used in artists' paints in normal indoor exposure.

NOTE 1—The color changes that result from accelerated exposure may not duplicate the results of normal indoor exposure in a home or gallery. The relative resistance to change, however, can be established so pigments can be assigned to categories of relative lightfastness.

1.3 Lightfastness categories are established to which pigments are assigned based on the color difference between specimens before and after exposure.

1.4 Color difference units are calculated by the CIE 1976 $L^*a^*b^*$ color difference equation.

1.5 These test methods apply to artists' oil, resin-oil, acrylic emulsion, alkyd, and watercolor paints.

1.6 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²
- D 4302 Specification for Artists' Paints: Oil, Acrylic Emulsion, Alkyd, and Resin-Oil²
- D 5067 Specification for Artists' Watercolor Paints²
- D 5098 Specification for Artists' Acrylic Emulsion Paints²
- E 284 Terminology of Appearance³
- E 1347 Test Method for Color and Color Difference Measurement by Tristimulus (Filter) Colorimetry³

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.57 on Artists' Paints and Related Materials.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 4303 - 83. Last previous edition D 4303 - 90.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 14.02.

- E 1348 Test Method for Transmittance and Color by Spectrophotometry Using Hemispherical Geometry³
- E 1349 Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry³
- G 24 Practice for Conducting Natural Light Exposures Under Glass³
- G 26 Practice for Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials⁴

3. Terminology

3.1 *Definitions*—Appearance terms used in this standard are defined in Definitions E 284.

4. Summary of Test Methods

4.1 Color measurements are made on specimens that have been prepared as directed in Specification D 4302, D 5067, or D 5098 depending on the vehicle. The measurements are recorded for comparison with readings made after the specimens have been exposed.

4.2 Specimens are exposed to light filtered through glass using at least two of the three test methods described in Section 7.

4.3 The pigments are classified by color difference calculated in accordance with Test Method D 2244.

5. Significance and Use

5.1 The retention of chromatic properties by a pigment over a long period of years is essential in an artistic painting. Accelerated exposure simulates color changes that may reasonably be expected. The producer and the user of artists' paints, therefore, can be apprised of suitable pigments.

6. Apparatus

6.1 *Sun Exposure Facilities* as described in Practice G 24 using an exposure angle of 45°, facing the Equator.

6.2 *Xenon-Arc Lightfastness Apparatus* as described in Practice G 26:

6.2.1 If water-cooled xenon-arc devices are used (Types AH or BH of Practice G 26), use borosilicate glass inner and soda lime outer filters to simulate sunlight filtered through window glass.

6.2.2 If air-cooled xenon-arc devices are used (Types C, D or E of Practice G 26), use the combination of optical filters recommended by the manufacturer to simulate sunlight filtered through window glass.

6.3 *Fluorescent Lamp Lightfastness Apparatus*, using

⁴ Annual Book of ASTM Standards, Vols 06.01, 08.03, and 14.02.

Verilux F48T12VLX/HO lamps⁵ and constructed in accordance with instructions given in Annex A1.

6.4 *Spectrophotometer*, abridged spectrophotometer or colorimeter capable of including specular reflectance in its measurement.

Procedure

7.1 Specimens of pigments to be tested in oil, resin-oil, or alkyd panels shall be prepared as directed in Specification D 4302. Specimens of pigments to be tested in acrylic emulsion paints shall be prepared as directed in Specification D 5098. Specimens of pigments to be tested in watercolors shall be prepared as directed in Specification D 5067.

7.2 Immediately before exposure, measure all test specimens on a spectrophotometer (see Test Method E 1348 or E 1349) or colorimeter (see Test Method E 1347) with the specular reflection included in the measurement and record. These results shall be compared with subsequent readings of the exposed specimens to calculate color difference, or with readings of retained specimens made at the time of visual comparison or before beginning corroborative tests to assure that there has been no color change in the interval.

7.2.1 Read specimen panels always with the striations in the same direction and with panels in the same position. If the design of the instrument allows, three readings at different locations on the panel should be made and the mean calculated. If feasible, mark on the back of the specimen the spot(s) measured, and re-measure these same spots following exposure.

7.2.2 If it is necessary to store specimens in the dark for a period of time, prior to measurement and exposure, those that contain oil in the vehicle shall be placed in subdued light for seven days to eliminate any yellowing of the oil due to storage.

7.3 Expose specimen panels to light passing through glass, either to natural sunlight or to simulated daylight in laboratory apparatus. Oil, resin-oil, alkyd, and acrylic emulsion paint specimens shall be exposed by Test Method A⁶ and other Test Method B or Test Method C. Watercolors and other paints sensitive to moisture shall be exposed by Test Methods B and C.

7.3.1 *Test Method A—Exposure Under Glass to Natural Sunlight:*

7.3.1.1 Mount the specimens on an open type rack and expose at 45° angle to the vertical to a total radiation dose of 1260 MJ/m² in accordance with Practice G 24.

7.3.1.2 For tests in southern Florida expose panels during the months of October through May.

7.3.2 *Test Method B—Exposure to Simulated Daylight (Fluorescent Lamp Type):*

7.3.2.1 Mount the specimens with the test face 3 in. (75

mm) from the plane of the lamps.

7.3.2.2 Unless specified otherwise, expose the specimens to a repeating cycle of 8 h light followed by 4 h dark until the specimen has been exposed to a total radiant energy dose of 1260 MJ/m². Rotate the specimen panels two positions to the right after each 100 MJ/m² dose. This test method takes approximately nine months to complete.

7.3.2.3 Maintain the ambient room temperature at 24 ± 3°C and prevent the test chamber from exceeding room temperature by more than 6°C.

7.3.2.4 Monitor irradiance at the specimen location for total radiation for each rotation period. Measure radiation 1 h after the lamps are turned on and at the end of the period. The mean of these readings in joules per square centimetre (J/cm²) per hour multiplied by the number of hours of radiation gives the J/cm² for that period.

7.3.3 *Test Method C—Exposure to Simulated Daylight (Xenon-Arc Type):*

7.3.3.1 Mount specimens in unbacked holders avoiding positions that place specimens at the extreme top or bottom of the specimen rack.

7.3.3.2 Unless specified otherwise, expose specimens continuously to a total radiant dose of 1260 MJ/m² in accordance with Practice G 26, Test Method C. Maintain the relative humidity in the test chamber at 55 ± 5 % and a black panel temperature of 63 ± 2°C.

NOTE 2—When mutually agreed upon, an alternative dark cycle may be employed in accordance with Test Method D of Practice G 26, optical filter combination (c), and test conditions reported. It has been found that Alizarin Crimson, and possibly other pigments, are affected by a light and dark cycle, owing to the oxidation-reduction effect of titanium dioxide changing valence with the changes from light to dark and vice versa, characteristic of daylight and indoor light.

7.3.3.3 When continuously monitoring irradiance with a narrow bandpass radiometer, the dosage at the monitored wavelength may be calculated knowing the total and spectral irradiance from the lamp operating at a specified wattage, using the equation:

$$Q_{e\lambda} = 3.6 (10^3) t E_{e\lambda} \quad (1)$$

where:

λ = wavelength bandpass expressed in nanometres, nm,

Q_e = radiometric symbol for radiant exposure, J/m²,

$3.6 (10^3)$ = a constant, s/h, for converting hours to seconds,

t = time expressed in hours, h, and

E_e = radiometric symbol for irradiance, W/m².

NOTE 3—1 W = 1 J/s.

NOTE 4—An example is given in Appendix X1.

7.4 Measure test specimens immediately after exposure with specular reflection included in the measurement and record.

7.5 Calculate the color difference between the specimen before exposure and after exposure in accordance with the CIE 1976 L*a*b* color difference equation in Test Method D 2244. State the color change in total color difference units (ΔE^*_{ab}).

7.6 Visually check the exposed specimen against the unexposed specimen of the same paint kept in subdued light, in order to verify that the color difference stated in ΔE^*_{ab} units agrees with visual assessment. Make this check also

⁵ The Verilux lamps manufactured by Verilux, Inc., P.O. Box 1512, Greenwich, CT 06836, are temporarily not available. Other fluorescent lamps that closely simulate daylight may be suitable, and Subcommittee D01.57 seeks information that will allow these fluorescent lamps to be recommended. Lamps should have a correlated color temperature of 6000 ± 500 K and a color rendering index of at least 90 as calculated by Method of Measuring and Specifying Colour-Rendering Properties of Light Sources, International Commission on Illumination (CIE) Publication No. 13.2 (TC-3.2), 1974. Available from the U.S. National Committee for the CIE, c/o Director of Marketing, OEM Division, North American Philips Lighting Corp., Philips Square CN8800, Somerset, NJ 08873.

following any subsequent exposures.

7.7 Since all test methods can produce aberrant results in pigments that are sensitive to some aspect of a particular type of exposure, assign pigments to lightfastness categories based on results from a minimum of two of the methods described in 7.3.1 through 7.3.3. Four exposures are preferred but there may be fewer in many cases using the following procedure:

7.7.1 Unless specified otherwise, expose one specimen inland outdoors under glass to sunlight in southern Florida below 27° latitude following Test Method A and expose the second specimen indoors to simulated daylight following either Test Method B or Test Method C. Place pigment in the appropriate category if both tests indicate the same category and neither result is within $\pm 0.5 \Delta E^*_{ab}$ of the borderline between categories.

7.7.2 If the results from the first two tests place the pigment in different lightfastness categories, or if either of the test results fall within $\pm 0.5 \Delta E^*_{ab}$ of the dividing line between two categories, either assign the pigment to the poorer of the two categories involved or for a more accurate rating, proceed with the third exposure. The third exposure may use either the method not used in the two previous exposures, or may repeat the exposure which gave the poorer result, providing the two test results are within $4 \Delta E^*_{ab}$ of one another. To guard against accepting an aberrant test result, if there is more than $4 \Delta E^*_{ab}$ difference between the first two test results, use the method not employed in the first two tests for the third exposure.

7.7.3 Calculate the mean of the color differences from the three exposures to determine the appropriate lightfastness category unless one of them differs from the nearest test result by more than $4 \Delta E^*_{ab}$, or the mean is within $\pm 0.5 \Delta E^*_{ab}$ of the dividing line between categories. In these cases make a fourth exposure.

7.7.4 For the fourth exposure use the method not previously employed, or if all three methods have been used, for the fourth exposure repeat the method with poorest result, unless the results from that method differs from the nearest other test result by more than $4 \Delta E^*_{ab}$. In this case, discard this one test result. Calculate the mean of the test results from the four exposures, or three exposures if one has been discarded, to determine the lightfastness category.

8. Interpretation of Results

8.1 *Lightfastness I*—Assign pigments that exhibit a mean color change of 4 or less ΔE^*_{ab} to Lightfastness Category I.

8.2 *Lightfastness II*—Assign pigments that exhibit a mean color change of more than 4.0 but not more than 8.0 ΔE^*_{ab} to Lightfastness Category II.

8.3 *Lightfastness III*—Assign pigments that exhibit a

mean color change of more than 8.0 but not more than 16.0 to Lightfastness Category III.

8.4 Since some exposed specimens have lost all color at a color difference of 20 units, additional categories are not appropriate for artists' paints.

9. Report

9.1 *Materials Tested*—Report the pigment tested, the name of the manufacturer, and the vehicle used in the test.

9.2 *Exposure*—Report the methods of exposure and their duration.

9.2.1 For Test Method A, in addition to the requirements of Practice G 24, report the total radiation dose in MJ/m².

9.2.2 For Test Method B, identify the apparatus used, including types and numbers of lamps, specimen to lamp distance, panel rotation sequence, light-dark cycle, method of monitoring radiation, exposure dosage in MJ/m², and test chamber and room temperature.

9.2.3 When using Test Method C, report as directed in Practice G 26.

9.3 *Test Results*—Report ΔE^*_{ab} for each pigment and the lightfastness category to which each is assigned.

10. Precision

10.1 Variation in test results can result from differences in pigment manufacture from time to time within a company, different varieties of a pigment from company to company, specimen preparation, different instruments and instrumental readings, variations in the surface of the specimen and the conditions of exposure. Allowance for these variables is made by requiring more than one test and by establishing lightfastness categories that include a range of color differences.

10.2 To establish the relationship between test methods, sets of 172 paint specimens, 90 in oil and 82 in acrylic emulsion vehicle, were made at the same time by one person and exposed in four sets of lightfastness tests: southern Florida sun filtered through glass, Kansas sun filtered through glass, fluorescent apparatus, and Xenon-arc apparatus.

10.2.1 All four tests placed 73 % of the pigments in the same category. When 12 aberrant test results were dropped from consideration and the third and fourth exposures conducted as required in cases where test results are near the border line between lightfastness categories (see 8.2), all combinations of the test results placed 99 % of the pigments in the same category.

11. Keywords

11.1 fluorescent exposure test; lightfastness; pigments; sun exposure test; Type C, D, and E; xenon-arc exposure test

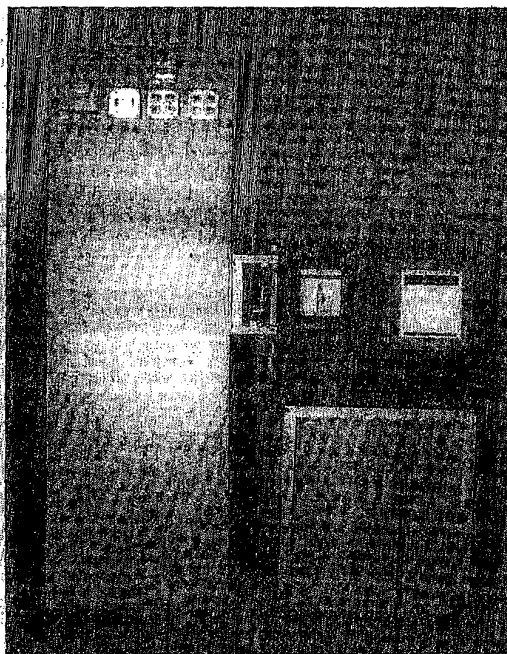
ANNEX

(Mandatory Information for Test Method B)

A1. FLUORESCENT DAYLIGHT LAMP LIGHTFASTNESS APPARATUS

A1.1 The apparatus for exposing paint specimens to fluorescent irradiation is shown in a large size version in Fig. A1.1 and in a smaller version in Fig. A1.2. Figure A1.3 provides the dimensions and arrangement for constructing the larger size apparatus out of materials that are easily available.

A1.2 *Cabinet Construction*—Make framework for the apparatus out of $1\frac{1}{2}$ by $1\frac{1}{2}$ by $\frac{1}{8}$ -in. (40 by 40 by 3.2-mm) aluminum angle available in hardware stores. The supports and the remainder of the enclosure are $\frac{1}{8}$ -in. tempered hardboard. The exposure panel supports, which are also the apparatus sides, are made removable by being fastened by reversed bolts with wing nuts. Coat them on the interior side with a non-yellowing white paint. Four reflectors, which each can hold four fluorescent lamps, are fastened together by the $1\frac{1}{2}$ -in. angles, back to back, to form a square in the center of the cabinet facing the specimens, which are attached to the hardboard panels support as shown in Fig. A1.3. Construct



NOTE—Pyranometer, recorder, and demand readout meter in place.
(a) Four-Bank Fluorescent Exposure Apparatus that Handles One hundred and eight 3 by 6-in. Panels

FIG. A1.1 Apparatus for Exposing Paint Specimens to Fluorescent Irradiation



NOTE—Notice hole for mounting Eppley pyranometer which is attached at right.
(b) Four-Bank Verilux Fluorescent Exposure Apparatus with One Panel Open

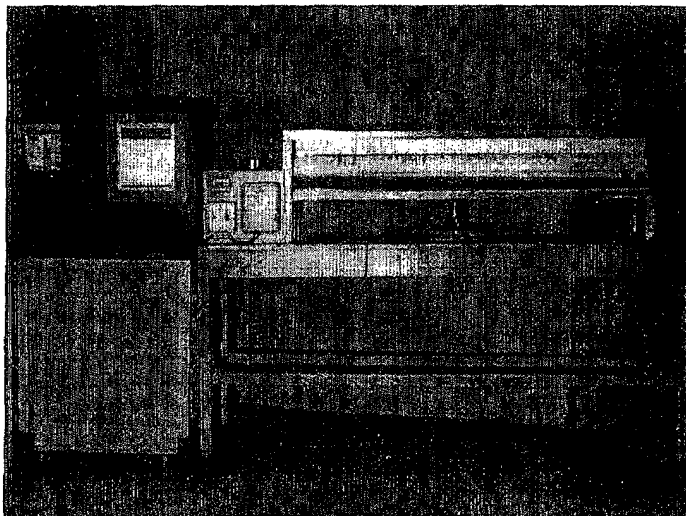
FIG. A1.1 Apparatus for Exposing Paint Specimens to Fluorescent Irradiation Continued

the framework so that the lamps are 3 in. (75 mm) from the test specimens. Cut holes in each side adjacent to the center and centered on a panel location to accommodate an Eppley pyranometer. With one panel space missing on each panel bank, the cabinet accommodates 108 test panels.

A1.3 *Irradiation*—The reflector fixtures hold sixteen 48 in. (1.2 m) long high powered Verilux⁶ lamps with a correlated color temperature of 6000 K and a spectral irradiance simulating sunlight.

A1.3.1 Irradiance can be monitored by a spectral radiometer or a filter radiometer which has been calibrated to provide information on the total spectral radiant exposure.

⁶ Verilux F48T12VLX/HO lamps have recessed double contact sockets and use a four gang fixture, Series 152 Industrial Fixture for Four High Output Lamps. Both are manufactured by Verilux, Inc., P.O. Box 1512, Greenwich, CT 06836. The lamps are temporarily not available.



NOTE—Pyranometer, recorder, and demand readout meter in place.

FIG. A1.2 Single-Bank Device of Four Verilux Daylight Fluorescent Lamps that Handles Twenty-seven 3 by 6-in. Panels

A1.3.2 Install a timer, shown as K in Fig. A1.3, to cycle exposure for 8 h of light followed by 4 h of dark.

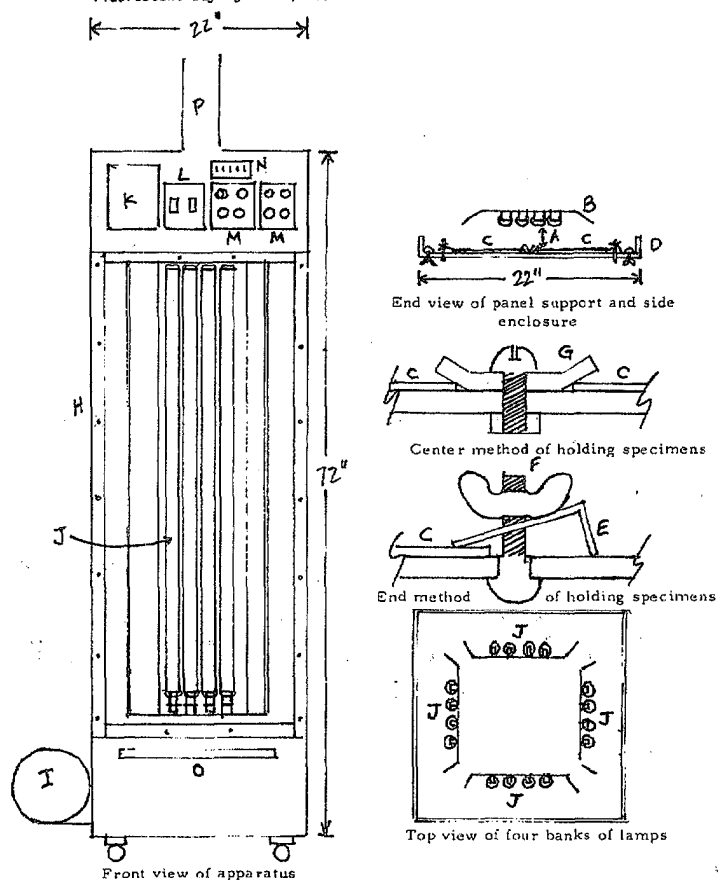
A1.3.3 Install an electric time meter in the circuit, shown as N in Fig. A1.3 to record the total amount of time the lamps are turned on.

A1.4 *Temperature and Humidity Control*—Install a centrifugal blower unit at the base of the cabinet, shown as I in Fig. A1.3. The air from the blower passes immediately through an air filter O and, after passing through the specimen exposure area, is vented out through the top of the cabinet. The blower furnishes the filtered air draft that keeps the temperature in the cabinet to within 6°C of the ambient

temperature of the room. The small 6°C temperature rise is sufficient to limit the humidity in the cabinet to about 60% relative humidity even when the general humidity is very high. Install a thermometer in the top of the cabinet near the vent and away from any direct illumination from the lights.

A1.5 If preferred, a smaller open apparatus holding a single bank of four Verilux daylight fluorescent lamps may be constructed as shown in Fig. A1.2. A small fan blowing through the unit from one end furnishes the air draft, but otherwise the apparatus must have the same distance between lamps and specimens and the same monitoring equipment described in A1.2 and A1.3.

Fluorescent Daylight Lamp Apparatus Showing One of Four Sides



Drawing: 1 in. = 25 mm.

- A 3-in. distance from lamp to exposure specimen
- B Four-gang fixture for fluorescent lamps
- C Exposure specimens
- D 1½ by 1½ by ½-in. aluminum angle frame
- E 1 by ½-in. metal trim strip
- F ¾-in. carriage bolt, ¾-in. long, spaced 6 in. apart, with wing nut
- G Metal corner moulding reversed
- H Aluminum angle frame throughout

- I Centrifugal blower
- J Fluorescent lamps in 4-gang fixture
- K Timer to control cycles
- L Two 2-way switches for Off-On and Interval-On
- M Individual switches for each bank
- N Time meter
- O Air Filter, inside
- P Vent

NOTE—Frame is 1½ by 1½ by ½-in. aluminum angle. Enclosure and removeable panel support sides are tempered hardboard.

FIG. A1.3 Dimensions for Constructing Apparatus

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE OF DOSAGE CALCULATION

X1.1 The calculation is as follows:

Given: Total dose, $Q_T = 1260 \text{ MJ/m}^2$

Assume: Total irradiance, $E_t = 1000 \text{ W/m}^2$

Spectral irradiance, $E_\lambda = 0.5 \text{ W/m}^2$

where λ = wavelength of measured spectral irradiance.

$$Q_T = 3.6 (10^3) t E_T$$

$$1260 (10^6) = 3.6 (10^3) t (1000)$$

$$\text{so } t = 1260 (10^6) / 3.6 (10^6) = 350 \text{ h}$$

Thus, if the total irradiance from an energy source remains constant at 1 kW/m^2 , 350 h are required to provide a total dose of 1260 MJ/m^2 .

X1.2 If the spectral irradiance at wavelength, λ , remains

constant, then the spectral irradiation, Q_λ , or the control dosage, is calculated by the equation, as follows:

$$Q_\lambda = 3.6 (10^3) t E_\lambda$$

$$Q_\lambda = 3.6 (10^3) 350 (0.5),$$

$$Q_\lambda = 630 (10^3) \text{ J/m}^2, \text{ or}$$

$$Q_\lambda = 630 \text{ kJ/m}^2.$$

(X1.2)

X1.3 The time required to obtain this dosage may be varied by varying the spectral irradiance. For $E_\lambda = 0.25$, the exposure time will double to 700 h.

X1.4 If the fluorescent lamp system requires nine months to accumulate 1260 MJ/m^2 when the lamps operate two thirds of the time, about 4380 h, then the mean irradiance from the lamps would be:

$$E_T = 1260 (10^6) / 3.6 (10^3) 4.38 (10^3) = 75.1 \text{ W/m}^2 \text{ (X1.3)}$$

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Determining Whether a Material Is a Liquid or a Solid¹

This standard is issued under the fixed designation D 4359; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

Scope

- 1.1 This test method covers the determination of whether a viscous material is a liquid or a solid for regulatory purposes.
- 1.2 The values stated in inch-pound units are to be regarded as the standard.
- 1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

- D 1 ASTM Standards:
 - D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²
 - E 300 Practice for Sampling Industrial Chemicals³

Summary of Test Method

- 2.1 The material under test is held at 100°F (38°C) in a tightly closed can. The lid is removed and the can inverted. The flow of the material from the can is observed to determine whether it is a solid or a liquid.

Significance and Use

- 3.1 Compliance with regulations often requires the determination of whether a material is a liquid or a solid. A similar test is used by the U. S. Department of Transportation for this purpose.

Apparatus

- 4.1 Can, quart (1 L), friction top with lid, diameter approximately 4 1/4 in. (108 mm), height 4 3/4 in. (120 mm).
- 4.2 Oven, maintained at a temperature of 100°F (38°C).
- 4.3 Tripod or Ring Stand.
- 4.4 Watchglass, tared.
- 4.5 Stopwatch.
- 4.6 Ruler.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.22 on Health and Safety.

² Current edition approved May 25, 1990. Published July 1990. Originally published as D 4359 - 84. Last previous edition D 4359 - 84.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.03 and 15.05.

6. Sampling

- 6.1 Using standard sampling techniques, secure a representative sample from the lot or batch being tested, in accordance with Practices D 3925 or E 300.

7. Test Specimen

- 7.1 If the sample is supplied in a friction top quart can that is at least 85 % full, use the can and material as supplied.
- 7.2 If not, place the material to be tested into the quart can, filling it to at least 85 % and allowing enough ullage for expansion of the material without displacement of the cover. Tightly close the can so that no vapor escapes.

NOTE—The can is considered 85 % filled when the material level is no more than 3/4 in. (20 mm) below the lip of the can.

8. Procedure

- 8.1 Place the can with the material in an oven maintained at 100 ± 5°F (38 ± 3°C). Allow the contents of the can to come to temperature equilibrium (18 to 24 h).
- 8.2 Place the tripod or ring stand on a level surface.
- 8.3 After the material has come to temperature equilibrium take the can from the oven and immediately remove the lid. With the ruler, measure the distance from the surface of the material to the top of the lip of the can and record.
- 8.4 Invert the can onto the tripod or ring stand in a vertical position over the tared watchglass. If a skin has formed on the surface of the material, break the skin prior to inverting the can. Determine the flow of the specimen out of the can at the end of 3 min, by measuring the vertical distance the material has flowed from the lip of the can to the tip of the specimen.
- 8.5 Add the distance from the surface of the material to the top of the lip of the can (8.3) to the distance the material has flowed below the lip of the can (8.4).
- 8.6 If there is an appreciable flow from the can before the 3 min are up, that is, more than 2 in. (50 mm), immediately stop the test and place the can in an upright position.
- 8.7 Collect any free-flowing material onto the tared watchglass and weigh.

9. Interpretation of Results

- 9.1 A material that flows a total of 2 in. (50 mm) or less within 3 min is considered a solid. Otherwise, it is considered a liquid.
- 9.2 If 1 g or more of liquid is collected on the watchglass, the viscous specimen is considered a liquid regardless of its flow characteristics.

10. Precision and Bias

10.1 *Precision*—It is not practical to specify the precision of this procedure as the results of the test are reported only as the state of the material under test.

10.2 *Bias*—Since there is no accepted reference material

suitable for determining the bias, no statement is made.

11. Keywords

11.1 regulations; sample definition; viscous material characterization

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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Standard Test Method for Apparent Tack of Printing Inks and Vehicles by the Inkometer¹

This standard is issued under the fixed designation D 4361; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method covers the procedure for determining the apparent tack of printing inks using a mechanical or electronic model of the Inkometer.

1.2 This test method is applicable to paste-type printing inks and vehicles that are essentially nonvolatile under ordinary room conditions.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Terminology

2.1 Descriptions of Terms Specific to This Standard:

2.1.1 *tack*—a function of the force required to split a thin film of a printing ink or vehicle between two rapidly separating surfaces; it is a rheological parameter indicative of internal cohesion of the fluid.

2.1.1.1 *Discussion*—Tack of a printing ink or vehicle is not a fixed number but varies with operating conditions, primarily separation velocity, splitting area, and film thickness. Tack also varies with changes in the rheological properties of the ink or vehicle due to time, temperature, and interactions with the separating surfaces. In practice, one or more of these surfaces usually consist of rubber-like rollers that differ in composition and geometry and whose properties tend to change with age, nature of previously run fluids, type of wash-up solvent, and mechanical flaws. On laboratory instruments, tack readings are also sensitive to the calibration and zero accuracy of the tackmeter employed.

2.1.2 *apparent tack*—a tack reading obtained at a specific set of conditions.

2.1.3 *flying*—the tendency of a printing ink or vehicle to be ejected, as large globules from a roller distribution system.

2.1.3.1 *Discussion*—Flying is generally most severe during rapid roller acceleration such as occurs when switching immediately from zero or a slow speed to a high operating speed.

2.1.4 *misting*—the tendency of a printing ink or vehicle to be ejected as a fine aerosol from a roller distribution system.

2.1.4.1 *Discussion*—Misting is generally most severe at

high operating speeds and with fluids that produce long filaments.

3. Summary of Test Method

3.1 A thin film of the test printing ink or vehicle is applied to the three-roller distribution system of the Inkometer, which operates at speeds comparable to those on production printing presses. Measurement of the frictional torque induced by drag forces in the splitting film provides an arbitrary value for apparent tack. On mechanical models, the torque is determined with a manually balanced lever arm, a direct-reading attachment, or a recorder; on electronic models, with a digital readout, recorder, or printer. Readings are in units of gram-meters (g-m).

3.2 The procedure in this test method is designed to give a single value for apparent tack at a specific set of Inkometer conditions. Typical conditions are as follows: a cooling water temperature of 90°F (32.2°C); a volume of 1.32 mL (film thickness 12.3 μ m) of the test printing ink or vehicle applied to the rollers; an operating speed of 400 r/min for vehicles, 800 r/min for sheet-fed offset inks, and 1200 r/min for web-fed inks; and a reading after 1 min of operation. Alternative conditions may be used by agreement between the supplier and the customer.

3.3 Instructions are also given for calibration of the Inkometer and for minimizing effects of interactions among the rollers, test fluids, and wash-up solvents.

4. Significance and Use

4.1 Tack of printing inks controls their high-speed transfer properties, as manifested by throughput in roll milling, picking of paper during printing, and wet trapping in multicolor printing. Although an apparent tack measurement does not completely predict the transfer performance of an ink or a vehicle, it provides a meaningful parameter for quality control, development, and research.

4.2 A given Inkometer will produce repeatable results on a day-to-day basis only if proper attention is paid to calibration and maintenance procedures and to control of experimental variables referred to in 2.1.1.1.

4.3 Two or more Inkometers may not produce identical apparent tack readings, but if each gives repeatable results, they may be mathematically correlated.

NOTE 1—Besides the Inkometer, a number of three-roller tackmeters are available which differ in design features such as roller weight, geometry, and composition. It cannot be presumed that test results from these other types of tackmeters will either agree or correlate with those from the Inkometer.

This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.56 on Printing Inks. Subcommittee D01.37 on Ink Vehicles assisted in the development of the vehicle portion of this test method.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 4361 - 84. Last previous edition D 4361 - 84.

5. Interferences

5.1 *Inkometer Squeal*—A high pitched whine or squeal may be noted when running high tack fluids or at high rotating speeds, or both. Inkometer squeal may result in instability of the balance beam or direct reading attachment of the mechanical Inkometer or fluctuation of the digital readout of the electronic Inkometer, making definite readings difficult.

6. Apparatus

6.1 *Inkometer*²—one of five models differing in available speeds and type of readout as follows:

6.1.1 *Mechanical Models B-45³, C-45³, or MBC*, operating at fixed speeds of 400, 800 and 1200 r/min on the B-45; 400, 1200, and 2000 r/min on the C-45; and 400, 800, 1200 and 2000 r/min on the MBC. A direct reading attachment² or a recorder² is recommended to supplement the manually operated balance beam.

6.1.2 *Electronic Models 101-A¹ or 101-B*, operating at variable speeds ranging from 100 to 2000 r/min on the 101-A and from 100 to 3000 r/min on the 101-B. A recorder² or printer², or both, are recommended to supplement the digital readout.

NOTE 2—To convert to units of linear speed, multiply Inkometer revolutions per minute by 0.785 to obtain feet per minute or by 0.004 to obtain metres per second.

6.2 *Inkometer Rollers*,² of suitable composition, preferably one set for each major system to be evaluated (see 10.3.1.) A set consists of a top (measuring) roller 3½ in. (79 mm) in diameter and 6½ in. (155 mm) in length, and a vibrator 2.0 in. (51 mm) in diameter and 7¼ in. (184 mm) in length. Together with the fixed brass roller, the total surface area of the distribution system is 166 in.² (0.107 m²). The measuring roller weighs 9.2 lb (4.2 kg) on mechanical models and 9.6 lb (4.4 kg) on electronic models.

6.3 *Ink Pipet*, consisting of a metal cylinder and a metal or TFE-fluorocarbon plunger. Suitable pipets include the fixed-volume Inkometer pipe,² 1.32-mL capacity; and variable volume micropipets,⁴ 2-mL capacity, accurate to 0.01 mL.

6.4 *Stopwatch or Timer*, accurate to 1 s.

6.5 *Ink Knife*, small, free from nicks and rough edges.

6.6 *Manufacturer's Calibration Apparatus*,² for the specific model Inkometer.

7. Reagents and Materials

7.1 *Wash-Up Solvent*, compatible with the test system, fast evaporating, and having minimal effect on the rollers; it should be acceptable environmentally. Hydrocarbon solvents with an initial boiling range of 250 to 350°F (120 to 177°C), a final boiling range of 300 to 400°F (150 to 205°C), a Kauri-Butanol value of 30 to 40 and less than 1 % benzene content are appropriate for many sheet-fed and heat-set

systems. Specific solvents may be required for unique systems.

7.2 *Rags or Wipers*, clean, soft, absorbent, lint-free.

7.3 *Manufacturer's Current Manual*,² for the specific model Inkometer.

8. Hazards

8.1 Never let an ink or a vehicle dry completely on the rollers of the Inkometer.

8.2 **Caution**—Never turn the ZERO button except during the calibration process (see 12.2.1).

8.3 Take care not to damage the rollers during the cleaning process or by leaving them in contact when the instrument is not in use.

8.4 Do not disengage the balance beam of the mechanical Inkometer except when taking a reading.

9. Sampling and Test Specimen

9.1 Carefully select a sample that is free of skin and other contamination and representative of the lot being evaluated. A minimum of 3 to 4 mL is sufficient for two specimens. Transfer to a clean container, protect with skin paper, close and seal.

9.2 When ready to make a run (see 12.3), fill the ink pipet as follows: Transfer 1.5 to 2 mL of sample to a clean glass plate; close and reseal the container. Gently work up with an ink knife but do not aerate. Fill the ink pipet with 1.32 mL of the worked sample (or with a smaller volume (0.5 to 1.0 mL) if a thinner film thickness is desired). Use the ink knife to force the specimen into the cylinder while slowly pulling back the ram. Wipe excess material off the top of the pipet.

NOTE 3—A specimen volume of 1.32 mL, divided by the roller surface area of 0.107 m², gives an initial film thickness of 12.3 µm when distributed uniformly on the roller system of the Inkometer. However, the occurrence of appreciable flying or misting will result in loss of specimen from the rollers. Hence, operating film thickness is unknown.

10. Preparation and Conditioning of the Inkometer

10.1 Locate the Inkometer on a sturdy bench in a draft-free temperature-controlled environment, preferably 73.5 ± 3.5°F (23 ± 2°C). Humidity control is necessary for test samples that are moisture-sensitive or prone to misting.

10.2 Set the Inkometer water bath at 90.0 ± 0.2°F (32.2 ± 0.1°C). All tests are to be run at this temperature. (See also Annex A1.3.)

10.3 Prior to use, ascertain the nature of the test sample for the following reasons:

10.3.1 *Roller conditioning*—Use only an Inkometer having rollers well broken in for the type of test system. The break-in procedure is given in Annex A1.2. A separate set of broken-in rollers is mandatory for radiation curing systems. The necessity for separate sets of broken-in rollers, or for extensive reconditioning when switching among different types of conventional test systems shall be determined in each laboratory.

10.3.2 *Operating speed*—Vehicles are most commonly run at 400 r/min, alternatively at 800 r/min; sheet-fed inks at 800 r/min, alternatively at 400 or 1200 r/min; and web-fed inks at 1200 r/min, alternatively at 800 or 2000 r/min. (The conversion to linear speed is given in Note 2.)

² Available from Thwing-Albert Instrument Company, 10960 Dutton Rd., Philadelphia, PA 19154.

³ No longer manufactured.

⁴ Available from I.G.T. Reptest B.V., 1004 Berlin, Cherry Hill, NJ 08034, Pantone, Inc., 55 Knickerbocker Rd., Moonachie, NJ 07074, or Eastern Scientific, 1459 Lower Ferry Rd., Trenton, NJ 08618.

10.4 Prior to the first use of the day, equilibrate the Inkometer as follows:

10.4.1 Warm up the instrument by activating the water washing system. Engage the two composition rollers and run at the lowest available speed for about 30 min.

10.4.2 Make a conditioning run with a specimen representative of the system to be evaluated. Apply 1 to 1.5 mL of ink or vehicle, and run for 5 to 10 min at the specified speed (see 10.3.2). Clean up as directed in Section 13.

11. Calibration of the Inkometer

11.1 Calibrate the Inkometer before initial use and periodically as needed. First, conduct the necessary steps in 10.3 and 10.4.

11.2 Using the manufacturer's calibration apparatus, follow the directions in the instrument manual.

11.2.1 *Mechanical Models*—Zero and calibrate the balance beam (and direct reading attachment or recorder, if they are to be used) at the test speed specified in 10.3.2.

11.2.2 *Electronic Models*—Zero and calibrate the digital readout (and recorder, if it is to be used) at 1000 r/min. When calibration is completed, check the dry reading at the specified test speed (see 10.3.2).

NOTE 4—The Inkometer can be calibrated at only one speed.

11.3 After each calibration or at regular periods, make a test run with a standard ink or vehicle. (See Annex A1.5.)

12. Procedure for Tack Evaluation

12.1 If necessary, make preparations as in 10.3 and 10.4, and calibrate the Inkometer as in Section 11. If using an electronic model, make sure the motor is preset to the test speed specified in 10.3.2 and the drive is in the LOW mode.

12.2 Engage the rollers and run at the specified test speed. If the dry reading differs from zero by more than ± 0.5 g-m, clean the rollers in accordance with 13.1 or recalibrate in accordance with Section 11.

12.2.1 The dry reading on a properly calibrated instrument is directly related to the condition of the top (measuring) roller; therefore, large deviations from zero are suspect. Usual causes are inadequate cleaning, residual sample or wash-up solvent, or mechanical damage. *Do not push the ZERO button, as doing so will shift the scale. Do not attempt to compensate by subtracting the dry reading from the test reading. Always reclean or recalibrate.* Should large deviations from zero persist, contact the manufacturer about the possibility of serious mechanical damage.

12.3 Turn the Inkometer off, disengage the rollers, and fill the pipet as in 9.2. Transfer its contents to the vibrator in a series of thin ribbons around the middle 5 in. (125 mm) of the roller. Wipe any specimen remaining in the pipet onto a clean place on the same roller. Reengage the rollers.

12.4 Distribute the specimen on the rollers and start the run as follows:

12.4.1 *Mechanical Models C-46 and B-45:*

12.4.1.1 Manually turn the motor coupling about ten revolutions, or until the specimen appears evenly distributed among the three rollers.

12.4.1.2 Set the gears at 400 r/min, start the motor and the stopwatch simultaneously, and let the ink distribute for 15 s. Stop the motor but not the stopwatch.

12.4.1.3 Quickly switch the gears to the test speed (specified in 10.3.2) and immediately restart the motor, noting the time on the stopwatch.

12.4.2 *Mechanical Model MBC:*

12.4.2.1 Place the fingertips against the sides of the brass roller and manually turn about ten revolutions, or until the specimen appears evenly distributed among the three rollers. Do not touch the surface of the rollers.

12.4.2.2 Place the speed control switch at the 150 r/min position. Simultaneously depress the power switch on the Inkometer and start the stopwatch. Let the ink distribute for 15 s.

12.4.2.3 Quickly reposition the speed control switch to the test speed, noting the time on the stopwatch.

12.4.3 *Electronic Models:*

12.4.3.1 Place the fingertips against the sides of the brass roller and manually turn about ten revolutions, or until the specimen appears evenly distributed among the three rollers. Do not touch the surface of the rollers.

12.4.3.2 Depress the DRIVE button and simultaneously activate the stopwatch. Let the ink distribute for 15 s at the automatic LOW speed of 150 r/min.

12.4.3.3 Quickly switch to the test speed (preset in 12.1) by depressing the HIGH/LOW button again, noting the time on the stopwatch.

12.5 After 60 s of running at the test speed, record the apparent tack of the test specimen from the balance beam (see Annex A1.4); direct-reading attachment, or the recorder of a mechanical Inkometer or the digital readout, recorder, or printer of an electronic Inkometer.

12.6 *Optional*—Rather than restrict the test to a single apparent tack determination, valuable information may, in some cases, be gained by continuing the run, taking readings at uniform time intervals (facilitated by the use of a recorder) until the apparent tack begins to decrease. Alternatively, the Inkometer speed may be varied stepwise and a tack reading taken after a specified time at each speed.

12.7 After the run, stop the Inkometer and clean up, as directed in Section 13.

12.8 Make a replicate test with another specimen of the same sample by repeating 12.2 through 12.6. The two tests should agree within the repeatability given in Table 1.

13. Wash-up Procedure

13.1 With the Inkometer running at the lowest speed, apply a small amount of wash-up solvent to the rollers. Remove most of the specimen from the system by placing pads of the clean, soft, absorbent lint-free rags or wipers firmly against the bottom of the brass roller. Repeat this procedure with additional solvent and pads until the rollers are free from ink or vehicle. If any material remains on the edges of the composition rollers, remove very gently with a solvent-moistened rag.

NOTE 5: *Caution*—Remove material directly from the measuring or vibrator rollers with extreme care. Undue pressure will cause uneven wear of the rollers and may place significant strain on the torsion bar of the electronic Inkometer. Use extreme care to ensure that the cleaning pad does not go through the roller nip; otherwise, serious mechanical problems may result and recalibration will be essential.

13.2 Dry the rollers thoroughly by running them in

TABLE 1 Standard Deviation and Precision of Apparent Tack Readings

Samples	Inkometer Type	Speed of Rollers, r/min	Standard Deviation				Repeatability		Reproducibility	
			Within-laboratory		Between-laboratory		1 min	5 min	1 min	5 min
			1 min	5 min	1 min	5 min				
Ink	mechanical	800	0.49	0.47	0.52	0.97	2.0	1.8	2.0	3.8
	electronic	800	0.31	0.46	0.67	1.26	1.2	1.8	2.6	5.0
Vehicles	electronic	400	0.38	0.37	1.30	1.60	1.1	1.0	3.8	4.7
	electronic	800	0.26	...	1.20	...	0.7	...	3.4	...

contact at high speed for a minimum of 5 min or until all of the solvent has evaporated.

13.3 Check the zero reading as in 12.2. Continue cleaning and drying until the dry reading reaches 0 ± 0.5 g-m.

13.4 When the rollers are satisfactorily clean, stop the Inkometer and disengage the measuring and vibrator rollers.

13.5 Clean the pipet, the ink knife, and the glass plate with a solvent-wet rag.

14. Report

14.1 Report the following information:

14.1.1 Complete identification of the sample,

14.1.2 Inkometer model used,

14.1.3 Test speed,

14.1.4 Ambient temperature,

14.1.5 Any modifications to this test method,

14.1.6 Whether significant flying or misting was observed,

14.1.7 Whether Inkometer squeal was noted during the test,

14.1.8 Average apparent tack reading of two determinations, and

14.1.9 Any additional apparent tack readings determined at constant speed-constant time intervals or varying speeds-constant time intervals.

15. Precision and Bias

15.1 Precision:

15.1.1 In an interlaboratory study⁵ of this test method six

inks with a broad range in tack were measured for apparent tack ten times at 1 min and 5 min on mechanical Inkometers in six laboratories and on electronic Inkometers in eight laboratories.

15.1.2 In a separate interlaboratory study⁶ of this test method, four quick-set vehicles and four heat-set vehicles with a broad range in tack were measured in duplicate on two different days at 1 min and 5 min at 400 r/min on electronic Inkometers in six laboratories. The four quick-set vehicles were also measured at 1 min at 800 r/min.

15.1.3 The within-laboratory/ and between-laboratory standard deviations are shown in Table 1. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

15.1.3.1 *Repeatability*—Two individual results obtained by the same operator should be considered suspect if they differ by more than the values given in Table 1.

15.1.3.2 *Reproducibility*—Two results, each the mean of two readings, obtained by operators in different laboratories should be considered suspect if they differ by more than the values given in Table 1.

15.2 *Bias*—Bias cannot be determined as there are no known methods for measuring actual splitting forces. There are indications that apparent tack measurements from the Inkometer correlate best with transfer performance when a series of test samples is based on the same vehicle chemistry.

16. Keywords

16.1 apparent tack; Inkometer; printing inks; splitting forces; tack; tackmeters; vehicles

⁵ Supporting data are available from ASTM Headquarters. Request RR:D01-1039.

⁶ Supporting data are available from ASTM Headquarters. Request RR:D01-1062.

ANNEX

(Mandatory Information)

A1. INFORMATION CONCERNING INKOMETERS

A1.1 Routine Maintenance of the Inkometer

A1.1.1 Routine maintenance of the Inkometer is extremely important to the mechanical integrity of the instrument; see the manufacturer's current instruction manual for the specific model.

A1.1.2 The measuring and vibrator rollers may acquire a glazed or shiny appearance with use, depending on the test system and the wash-up solvent. This glaze may result in a significant change in the apparent tack reading of an ink or vehicle. The glaze may be removed by extra cleaning with a strong solvent such as acetone. If not, the composition rollers should be replaced.

A1.2 Breaking-in the Inkometer Rollers

A1.2.1 New Inkometer measuring and vibrator rollers may selectively absorb certain components of some test systems, up to a saturation point, at which point they may be said to be broken in. Until this selective absorption is complete, tack determinations made with these rollers may not be repeatable. Break in new rollers using the following procedure:

A1.2.1.1 Place the rollers on the Inkometer. Choose as break-in samples those representative of the system that will be evaluated on the rollers. Run approximately 1.0 to 1.5 mL of the break-in sample for extended periods of time, wash-up with the solvent to be used, reapply the sample, run, wash-up, etc.

NOTE A1.1—The wash-up is a significant part of the break-in process.

A1.2.1.2 Break-in time may vary from several hours to several days. Reproducible apparent tack readings on standard samples (see A1.5.1), over a period of several days, indicate that the rolls are broken in; they may then be put into routine use.

A1.2.2 A major change in ink systems may adversely affect the Inkometer rollers. When a set of rollers has been used for one system, and it is to be used for another, use this same break-in procedure. The rollers may then no longer be suitable for the original system.

A1.3 Temperature Control of the Inkometer Water Bath

A1.3.1 Extremely precise temperature control of the Inkometer water bath is essential for repeatable apparent tack readings.

A1.3.2 The thermometer furnished with the mechanical Inkometer, and the temperature gage of the electronic Inkometer are accurate to $\pm 0.5^\circ\text{F}$ ($\pm 0.3^\circ\text{C}$).

A1.3.2.1 It may be advantageous to use a Bomb Calorimeter Thermometer ASTM 56F, 66.0 to 95.00°F, with divisions of 0.05°F, or ASTM 56C, 19.00 to 35.00°C, with divisions of 0.02°C, in the Inkometer water bath. Position the thermometer in such a manner that the bottom of the mercury bulb is in line with the return inlet from the brass roller.

A1.3.3 The temperature-control system of the Inkometer is capable of controlling the temperature inside the brass roller within $\pm 0.5^\circ\text{F}$ ($\pm 0.3^\circ\text{C}$).

A1.3.3.1 It may be advantageous, particularly, if high-tack samples are run for extended periods of time, or if the Inkometer is in constant use, to augment the temperature control system with a cold-water cooling coil. A coiled length of 1/4-in. (6.3-mm) outside-diameter copper tubing may be placed in the water-bath reservoir, in the flow area away from the thermometer. The coil is connected to a cold-water tap with a pressure regulator and emptied into a sink or drain.

A1.4 Reading the Mechanical Inkometer Balance-Beam

A1.4.1 In order to take a reading from the balance beam of a mechanical Inkometer, disengage the beam and move the sliding weight until the beam is continuously in balance. Read the scale at the left of the sliding weight, using the scale alignment cutout to facilitate reading.

A1.4.2 Minimization of parallax is necessary for repeatable apparent tack readings. It may be useful to mount a small reflective surface on the beam stop behind the zero indicator and the balance beam. The zero indicator and the zero line on the balance beam are aligned in the reflective surface when an apparent tack reading is being taken.

A1.4.3 Reengage the balance beam immediately after taking the reading.

A1.5 Standard Test Samples

A1.5.1 It may be useful to designate one or more inks or vehicles as standards. Samples that are stable and have a good shelf life without a change in apparent tack reading (for example, tack rated or tack graded) are appropriate. Daily apparent tack readings on these samples ensures that the Inkometer is in calibration and serves as a check on repeatability.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Methods for Hardness of Organic Coatings by Pendulum Damping Tests¹

This standard is issued under the fixed designation D 4366; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 These test methods cover the use of pendulum damping testers in the determination of hardness of organic coatings that have been applied to acceptably plane rigid surfaces, such as a metal or glass panel.

1.2 Two test methods based on different pendulum types are covered as follows:

1.2.1 Test Method A—König Pendulum Hardness Test

1.2.2 Test Method B—Persoz Pendulum Hardness Test

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²

D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *König hardness*—time in seconds for the swing amplitude of the König pendulum to decrease from 6 to 3°.

3.1.2 *Persoz hardness*—time in seconds for the swing amplitude of the Persoz pendulum to decrease from 12 to 4°.

4. Summary of Test Methods

4.1 A pendulum resting on a coating surface is set into oscillation (rocking) and the time for the oscillation amplitude to decrease by a specified amount measured. The shorter the damping time, the lower the hardness.

5. Significance and Use

5.1 The pendulum damping test has been found to have

good sensitivity in detecting differences in coating hardness, where hardness is defined as resistance to deformation.

5.2 The two procedures given in these test methods embody the principle that the amplitude of oscillation of a pendulum touching a surface decreases more rapidly the softer the surface. However, these test methods differ in respect to pendulum dimensions, and period and amplitude of oscillation.

5.3 In general, the damping time of the König pendulum is approximately half that of the Persoz pendulum.

5.4 The Persoz pendulum has a greater degree of discrimination than the König for measuring the hardness of soft coatings, but it may not be as suitable for testing hard, slippery films because of its tendency to skid on surfaces with a low coefficient of friction.

5.5 The interaction between the pendulum and the paint film is complex, it depends on both elastic and viscoelastic properties, and it may not be possible to establish a precise relationship between the two types of pendulum tests.

TEST METHOD A—KÖNIG PENDULUM HARDNESS TEST

6. Apparatus

6.1 *König Pendulum Tester*,³ consisting of a stand that supports a pendulum, a test panel, and a pendulum displacement scale. The stand has a stirrup to support the pendulum above the table and a mechanism for shock-free lowering of the pendulum onto the test panel. A typical apparatus is shown in Fig 1.

NOTE 1—Optional features may be the inclusion of an electronic device for automatically timing the oscillation damping and the inclusion of an oscillation counter.

6.2 *König Pendulum*, consisting of an open framework connected by a cross-bar, to the underface of which are two balls, 5 ± 0.005 mm in diameter of hardness 63 ± 3.3 HRC, inset to serve as the fulcrum. The lower end of the framework is formed into a pointer. A weight sliding on a vertical rod attached to the cross-bar is used to counterpoise the pendulum. The total weight of the pendulum shall be 200 ± 0.2 g.

6.3 *Stop Watch*, or other timing device for timing the oscillation damping of the pendulum.

6.4 *Polished Plate (Float) Glass Panel*, for calibrating the pendulum.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 4366 - 84. Last previous edition D 4366 - 87.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Pendulum hardness testers meeting the apparatus requirements of this test method may be obtained from BYK-Gardner, Inc., 2435 Linden Lane, Silver Spring, MD 20910, Paul N. Gardner Co., Inc., 316 N.E. First Street, P.O. Box 10688, Pompano Beach, FL 33061-6688 and Zorelco Limited, P.O. Box 25500, Cleveland, OH 44125.

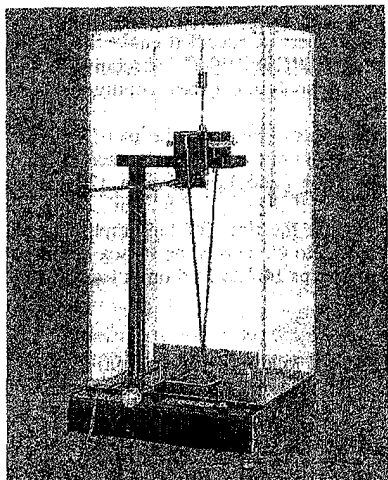


FIG. 1 Apparatus

Calibration

- 7.1 Check the alignment of the pendulum and panel table as follows:
 - 7.1.1 Place the polished glass panel on the panel table and gently bring the pendulum to rest on the surface of the glass. Ensure the pendulum oscillates freely.
 - 7.1.2 Place a spirit level on the glass panel surface. Level the glass panel by means of the adjusting screws at the base of the instrument.
 - 7.1.3 Clean the glass panel by wiping with a soft, lintless cloth wetted with the solvent mixture specified in Practice D 891.
 - 7.1.4 Clean the fulcrum balls by wiping with a soft tissue wetted with solvent. Leave the pendulum in ambient conditions and then bring it to rest on the glass panel.
 - 7.1.5 Check the position of the scale relative to the pendulum pointer. With the pendulum at rest, its pointer should indicate zero on the scale. If the pointer does not indicate zero, move the scale to obtain the correct zero setting.
- 7.2 Check the duration of the pendulum swing on the glass panel.
 - 7.2.1 Deflect the pendulum through 6° , release it and simultaneously start a stopwatch or other timing device.
 - 7.2.2 Determine whether the time for 100 swings of the pendulum falls within 140 ± 2 s.
 - 7.2.3 If the measured time is less than specified, move the weight on the pendulum rod upward. If the measured time is more than specified, move the weight downward. Continue adjustments until the specified time is obtained. If the time cannot be obtained, the instrument should be judged faulty and be repaired.
- 7.3 Check the duration of damping of the pendulum on the glass panel as follows:
 - 7.3.1 Deflect the pendulum through 6° , release it and simultaneously start the stopwatch or other timing device.
 - 7.3.2 Determine whether the time for the amplitude of

swing to decrease from 6 to 3° falls within 250 ± 10 s (corresponding with 172 to 185 pendulum swings).

8. Test Panel Preparation and Conditioning

8.1 Apply uniform coatings of the material to be tested to plane, rigid surfaces, such as metal or glass panels, by one of the procedures given in Test Methods D 823.

8.2 Cure the coated panels under the conditions of humidity and temperature, as agreed upon between the purchaser and seller.

8.3 Measure the thickness of the dry coating in accordance with Test Methods D 1005, D 1186, or D 1400.

NOTE 2—Coating thickness must be controlled closely because the pendulum test results can be affected by thickness variations. A minimum thickness of $25 \mu\text{m}$ is required to minimize substrate effects.

9. Procedure

9.1 Unless otherwise specified, make the hardness determination at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, after holding the test panels under these conditions for at least 16 h.

9.2 Place the test panel on the panel table and gently bring the pendulum onto the panel surface.

9.3 Deflect the pendulum through 6° , release it and simultaneously start the stopwatch or other timing device.

9.4 Determine the time for the amplitude of swing to decrease from 6 to 3° . This is the König hardness.

9.5 Repeat steps 9.2 through 9.4 on at least two other areas of the test panel.

10. Report

10.1 Report the following information:

- 10.1.1 Mean and range of the König hardness values in sections obtained for the test panel,
- 10.1.2 Manufacturer and model of the hardness tester used,
- 10.1.3 Mean and range of the thickness values obtained for the coating on the test panel, and the method used for determining the thickness,
- 10.1.4 Temperature and relative humidity during the test, and
- 10.1.5 Test panel preparation and conditioning techniques used.

11. Precision⁴

11.1 *Method A—König Pendulum Test*—On the basis of an interlaboratory test of this test method in which operators in four laboratories made three hardness measurements on each of six coated panels covering a wide range of hardness on two days, the within-laboratory coefficient of variation was found to be 3 % with 24 df and the between-laboratories coefficient of variation 8 % with 18 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

11.1.1 *Repeatability*—Two results, each the mean of three determinations on a specimen, obtained by the same oper-

⁴ Supporting data are available from ASTM Headquarters. Request RR: D01-1050.

ator should be considered suspect if they differ by more than 8 % of their mean value.

11.1.2 *Reproducibility*—Two results, each the mean of three determinations on a specimen, obtained by operators in different laboratories should be considered suspect if they differ by more than 23 % of their mean value.

TEST METHOD B—PERSOZ PENDULUM HARDNESS TEST

12. Apparatus

12.1 *Persoz Hardness Tester*,⁴ consisting of a stand that supports a pendulum, a test panel, and a pendulum displacement scale. The stand has a stirrup to support the pendulum above the table and a mechanism for shock-free lowering of the pendulum onto the test panel. A typical apparatus is similar to that shown in Fig. 1.

12.2 *Persoz Pendulum*, consisting of an open framework connected by a cross-bar, to the underface of which are two stainless steel balls, 8 ± 0.005 mm in diameter of hardness 59 HRC, inset to form a fulcrum.

12.2.1 *Pointer*, attached to the lower end of the framework, the tip of which shall be 400 ± 0.2 mm below the plane of the fulcrum. The total weight of the pendulum shall be 500 ± 0.1 g.

13. Calibration

13.1 Check the alignment of the pendulum and panel table.

13.1.1 Follow the procedures outlined in 7.1.1 to 7.1.4.

13.2 Check the duration of the pendulum swing on the glass panel.

13.2.1 Deflect the pendulum through 12° , release it and simultaneously start the stopwatch or other timing device.

13.2.2 Determine whether the time for 100 swings of the pendulum falls within 100 ± 0.1 s.

13.2.3 If this value cannot be achieved, reclean the glass panel and the bearing balls of the pendulum, recheck the level of the glass panel, and retest. It is not permissible to adjust the instrument scale at this point.

13.3 Check the duration of damping of the pendulum on the glass panel as follows:

13.3.1 Deflect the pendulum through 12° , release it and simultaneously start the stopwatch or other timing device.

13.3.2 Determine whether the time for the amplitude of swing to decrease from 12° to 4° falls within 400 ± 10 s.

13.3.3 If this value cannot be achieved, repeat the glass panel and instrument checks described in 13.2.3.

14. Procedure

14.1 Unless otherwise specified, make the hardness determination at $23 \pm 2^\circ\text{C}$ and 50 ± 5 % relative humidity, after holding the test panels under these conditions for at least 16 h.

14.2 Place the test panel on the panel table and gently bring the pendulum onto the panel surface.

14.3 Deflect the pendulum through 12° , release it, and simultaneously start the stopwatch or other timing device.

14.4 Determine the time for the amplitude of swing to decrease from 12° to 4° . This is the Persoz hardness.

14.5 Repeat steps 14.2 to 14.4 on at least two other areas of the test panel.

15. Report

15.1 Report the following information:

15.1.1 Mean and range of the Persoz hardness values in seconds obtained for the test panel,

15.1.2 Manufacturer and model of hardness tester used,

15.1.3 Mean and range of the thickness values obtained for the coating on the test panel, and the method used for determining the thickness,

15.1.4 Temperature and relative humidity during the test, and

15.1.5 Test panel preparation and conditioning techniques used.

16. Precision⁴

16.1 *Method B—Persoz Pendulum Test*—On the basis of an interlaboratory test of this test method in which operators in five laboratories made two or three hardness measurements on each of six coated panels (covering a wide range of hardness), the within-laboratory coefficient of variation was found to be 1 % with 24 df and the between-laboratories coefficient of variation 3 % with 12 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

16.1.1 *Repeatability*—Two results, each the mean of three determinations on a specimen, obtained by the same operator should be considered suspect if they differ by more than 3 % of their mean value.

16.1.2 *Reproducibility*—Two results, each the mean of three determinations on a specimen, obtained by operators in different laboratories should be considered suspect if they differ by more than 8 % of their mean value.

17. Keywords

17.1 damping hardness tests; König pendulum hardness tester; organic coatings; pendulum hardness tests; Persoz pendulum hardness tester

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Standard Test Methods for Acid and Base Milliequivalent Content of Electrocoat Bath¹

This standard is issued under the fixed designation D 4370; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Editorial changes were made throughout in May 1990.

1. Scope

1.1 These test methods cover the determination of acid and base milliequivalent contents of anodic and cathodic electrocoat baths and their ultrafiltrates.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*
D 1193 Specification For Reagent Water²

3. Summary of Test Methods

3.1 Specimens are titrated with standard acid and alkali solutions respectively. Alternative procedures are given for determining acid and base concentrations potentiometrically or using a pH meter.

4. Significance and Use

4.1 The acid and base concentrations are a measurement of the titratable acidic and alkaline components in the electrocoat baths. These measurements are used for research, production or electrocoat bath process control.

5. Apparatus

5.1 *Automatic Potentiometric Titrator with Stirrer and Recorder*, any model.

5.2 *Analytical Balance*, with sensitivity of 0.1 mg.

5.3 *pH Meter*, any model.

5.4 *Glass and Saturated Calomel Electrodes*.

5.5 *Syringes*, 5-mL disposable.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other

grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—References to water shall be understood to mean water conforming to Type II of Specification D 1193.

6.3 *Potassium Hydroxide Solution in Methanol*, 0.1 N—Prepare by dissolving 5.6 g of potassium hydroxide (KOH) pellets in 1 L of methanol. Standardize against NIST standard reference material of acid potassium phthalate No. 84 using an automatic potentiometric titrator⁴ to a given end point or, alternatively, to a phenolphthalein end point.

6.4 *Hydrochloric Acid Solution*, 0.1 N—Prepare by mixing about 8.50 mL of concentrated hydrochloric acid (HCl) (1.19 sp gr) into a mixture of 600 mL water and 400 mL methanol. Standardize against 0.1 N potassium hydroxide solution (see 6.3).

6.5 *1,3-Propanediol* (Propylene Glycol) (PG).

6.6 *Tetrahydrofuran* (THF).

6.7 *Reference pH Standard Solutions*—Commercial standards of pH 4.0, 7.0, and 10.0.

7. Sampling and Sample Preparation

7.1 The sample should be obtained while the electrocoat bath is under proper circulation such that a uniform material is obtained. In case of an ultrafiltrate, the material should be thoroughly mixed or stirred prior to sampling to assure uniformity.

7.2 After sampling and prior to removing a test specimen, it is mandatory that the samples be shaken or stirred until they are homogeneous and free of any settled material. This is particularly important if there is a delay between sampling the bath and performing the test. The absence of settled material can be ascertained visually (in a transparent container) or by inserting a spatula, scraping the bottom of the container, and making sure that there is no settled matter. The shaking or stirring of the samples should be carried out up to the moment of taking a specimen; *this Point is Very Important*.

8. Base Concentration Content

8.1 Stir the sample very thoroughly to disperse materials that might have settled to the bottom of the container. With the aid of a syringe, withdraw approximately 5 mL of the

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

Current edition approved June 28, 1984. Published November 1984.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by

Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

⁴ Svehla, G., *Automatic Potentiometric Titration*, Pergamon Press, 1978, p. 187.

sample quickly, weigh the full syringe to 0.1 mg, and record this weight as W_1 . Transfer the entire contents of the syringe into a 100-mL beaker. Reweigh the empty syringe to 0.1 mg and record as W_2 . Duplicate the procedure using a second specimen.

8.2 Add approximately 40 mL of THF/PG 80/20 mixture to the specimens in the beakers and cover, preferably with aluminum foil, to minimize evaporation of the solvent mixture.

NOTE 1—In cases where the recommended THF/PG 80/20 mixture causes precipitation of the material, substitute the same amount of an appropriate solvent or solvent mixture to a new specimen. The formation of a precipitate during the titration might foul the electrode system and mask potentiometric changes. Other solvents suitable for dilution are acetone, dimethylformamide, toluene, or methanol. Any other solvent or solvent mixture that does not cause the formation of a precipitate throughout the titration is suitable. In certain instances it may be necessary to run a titration blank and to make the appropriate corrections for the solvent.

8.3 Titrate both specimens with 0.1 N HCl solution using the automatic potentiometric titrator until an end point "break" is reached.⁴ Add approximately 5 mL of additional titrant to complete the curve. A typical titration curve is shown in Fig. 1. Record as V_1 the volume of titrant needed for the end point.

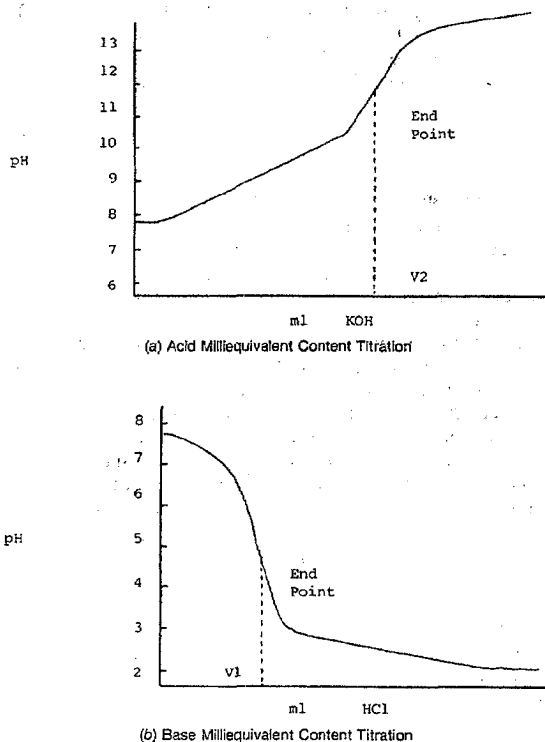


FIG. 1 Potentiometric Titration Curves for the Acid and Base Milliequivalent Content

NOTE 2—If the titration curve does not show an inflection point (end point), back titration with 0.1 N KOH will give an acceptable result.

9. Base Content Calculation

9.1 Calculate the base content as follows:

$$A = \frac{(V_1 \times N_1) \times 100}{(W_1 - W_2)M}$$

where:

A = milliequivalents of base per gram of nonvolatile matter,

V_1 = volume of HCl titrant used, mL,

N_1 = normality of HCl titrant,

W_1 = mass of the syringe filled with sample, mg,

W_2 = mass of the empty syringe after delivery of the specimen, mg, and

M = nonvolatile matter content, %.

10. Acid Concentration Content

10.1 Using a fresh portion of the sample, follow 8.1 and 8.2.

10.2 Titrate both specimens with 0.1 N KOH solution using the automatic potentiometric titrator until an end point break is reached. Add about 5 mL more of titrant to complete the curve. From the curve, determine and record, as V_2 , the volume of titrant needed for the end point.⁴

11. Acid Content Calculation

11.1 Calculate the acid content as follows:

$$A = \frac{(V_2 \times N_2) \times 100}{(W_3 - W_4)M}$$

where:

A = milliequivalents of acid per gram of nonvolatile matter,

V_2 = volume of KOH titrant used, mL,

N_2 = normality of KOH titrant,

W_3 = mass of the syringe filled with sample, mg,

W_4 = mass of the empty syringe after delivery of the specimen, mg, and

M = nonvolatile matter content, %.

12. Base Concentration Content Using a pH Meter

12.1 Proceed in accordance with 8.1 and 8.2.

12.2 Standardize the pH meter at 4.0 and 7.0.

12.3 Titrate both specimens with 0.1 N HCl until a pH of 4.0 is obtained. Make certain that the solutions are well agitated during titration (a magnetic stirrer is recommended). Record volume of titrant, as V_1 , used for each titration. Because electrocoating paints vary greatly, it might be advisable to titrate to a pH value agreed upon between the producer and the user.

13. Base Content Calculation

13.1 Calculate the base content as described in Section 9.

14. Acid Concentration Content Using a pH Meter

14.1 Proceed in accordance with 8.1 and 8.2.

14.2 Standardize the pH meter at 7.0 and 10.0.

14.3 Titrate both specimens with 0.1 N KOH until a pH of 10.0 is obtained. Make certain that solutions are well agitated during titration (a magnetic stirrer is recommended). Record volume of titrant, as V_2 used for each.

5. Acid Content Calculation

5.1 Calculate the acid content as described in Section 11.

6. Precision

6.1 In an interlaboratory study of the test methods, in which six laboratories measured in duplicate on two days four electrocoat samples with acid and base milliequivalents ranging from 0.2 to 0.8, the within-laboratory coefficient of variation, after discarding one result, was found to be 1.4 % relative at 23 df and the between-laboratory coefficient of variation 5.7 % relative at 19 df. Based on these coefficients,

the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

16.1.1 *Repeatability*—Two results, each the mean of duplicate determination, obtained by the same operator on different days should be considered suspect if they differ by more than 4.1 % relative.

16.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 17 % relative.

17. Keywords

17.1 acid content; base content; electrocoat baths

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Measuring Electrical Conductivity of Electrocoat Baths¹

This standard is issued under the fixed designation D 4399; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of the electrical conductivity of electrocoat baths or ultrafiltrate samples using commercially available equipment.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water²

D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 A specimen is placed in a conductivity cell, or conversely a conductivity cell is placed in an electrocoat material, and the cell is connected to a conductivity bridge. The electrical conductivity is read directly off the meter of the bridge as the instantaneous peak reading.

4. Significance and Use

4.1 The conductivity of electrocoat baths results from the presence of ionic species in the bath, which come from the vehicle and from the presence of impurities present as ionizable acids, bases, salts, or combinations of these. The presence of excessive amounts of ionic impurities is detrimental to the application and performance properties of electrocoating paints. The test is suitable for use in research, production, quality control and electrocoat bath process control.

4.2 Other related methods for determining the electrical conductivity of water are described in Test Methods D 1125.

5. Apparatus

5.1 *Conductivity Bridge*—Battery, or AC/DC line-operated, capable of providing a conductivity reading almost instantaneously.

5.2 *Conductivity Cell*—Dip or fill type, cell constant of 1.0.

5.3 *Thermometer*—Any type capable of 0.5°C accuracy with a -2 to 32°C range.

5.4 *Measuring Vessel*—Any suitable cylindrical container capable of holding sufficient electrocoat sample to cover the electrodes of the conductivity cell, and allowing at least 25 mm between the conductivity cell and the sides of the vessel.

6. Reagents and Materials

6.1 *Purity of Water*—References to water shall be understood to mean water conforming to Type II of Specification D 1193.

6.2 *Cleaning Solvent*—An appropriate solvent for the electrocoat material under measurement.

7. Sampling and Sample Preparation

7.1 The sample should be obtained while the electrocoat bath is under proper circulation so that a uniform sample is obtained. In the case of an ultrafiltrate, the material should be thoroughly mixed or stirred prior to sampling to ensure uniformity.

7.2 After sampling and prior to removing a test specimen, it is mandatory that the samples be shaken or stirred until they are homogeneous and free of any settled material. This is particularly important if there is a delay between sampling the bath and performing the test on the bath materials. The absence of settled material can be ascertained visually (in a transparent container) or by inserting a spatula, scraping the bottom of the container and making sure that there is no settled matter. Shake or stir the sample until specimens are taken for measurement; **THIS POINT IS VERY IMPORTANT.**

8. Procedure

8.1 Calibrate the conductivity cell prior to use following the manufacturer's instructions.

8.2 Rinse the measuring container several times with portions of the electrocoat material under test.

8.3 Take a representative portion of the electrocoat bath or permeate sample and place it in the measuring container. Stir the specimen thoroughly to keep it from settling.

8.4 Adjust the temperature of the specimen to $25 \pm 0.5^\circ\text{C}$. This is very important, since temperature differences cause disagreements in results and are a major source of error in these measurements.

8.5 Immerse the cell in the specimen, and move the cell up and down several times to displace any air bubbles from inside the cell. Hold the cell in a slightly inclined position in the center of the container with the vents at the top of the cell housing beneath the surface of the liquid. If a fill-type cell is used, pour the specimen into the conductivity cell.

8.6 Read and record the conductivity as described in the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

Current edition approved May 25, 1990. Published July 1990. Originally published as D 4399 - 84. Last previous edition D 4399 - 85a.

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

manufacturer's instructions regarding the use of the instrument. Avoid measuring times longer than 15 s to prevent electrocoat sample deposition on the cell electrodes. Perform a second measurement on another specimen, repeating steps 8.3 and 8.6. Calculate the mean of both measurements.

8.7 Take extreme care to prevent contamination of the sample and equipment. Rinse the cell with deionized water and an appropriate solvent immediately after use. Keep cell clean and do not allow the dry electrocoat sample to accumulate on the interior of the cell.

9. Report

9.1 Report the conductivity in terms of microsiemens (or microohms per centimetre) to the nearest 1 % of the determined conductivity.

10. Precision and Bias

10.1 *Precision*—In an interlaboratory study of this test method, with five laboratories measuring five electrocoat

bath samples with conductivities ranging from 720 to 1750 $\mu\text{S}/\text{cm}$ (microohms/cm), the intralaboratory coefficient of variation was found to be 2.6 % relative at 25 df, and the interlaboratory coefficient of variation 4.2 % relative at 20 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

10.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 7.6 % relative.

10.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 16.5 % relative.

10.2 *Bias*—No bias has been determined for this test method.

11. Keywords

11.1 electroconductivity; electrocoat bath

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Standard Test Method for Sag Resistance of Paints Using a Multinotch Applicator¹

This standard is issued under the fixed designation D 4400; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

NOTE—Paragraph 9.3.1.1 was editorially corrected and the year date was changed Oct. 6, 1989.

INTRODUCTION

The multinotch applicator used in this test method is a drawdown blade with a series of notches of successively higher clearance, referred to as the Anti-Sag Meter. See Figs. 1 and 2 for a representative diagram and photograph. The numerical value for sag resistance obtained with this instrument is referred to as the Anti-Sag Index.

Anti-Sag Meters are made with several clearance ranges for different types of coatings (see 5.1). In developing this test method the task group used an instrument with a range from 4 to 24 mils, but this test method is applicable to any clearance range, and results using instruments with overlapping ranges correlate and have equal validity.

This test method covers two procedures. Procedure A was developed in 1962^{2,3} and is referenced in U.S. Federal specifications TT-E-508, TT-E-506, and TT-P-1511. Procedure B is a newer method that obviates concern about the effect of substrate wetting as a possible variable. Numerical values obtained with the two procedures are not necessarily equal, but their rank orders are essentially the same.

A preshear program is essential for a drawdown sag test to duplicate the breakdown in structure that occurs when thixotropic paints are applied by brushout or other practical application methods. The procedures therefore include the preshearing of paints just prior to making test applications.

1. Scope

1.1 This test method covers the laboratory determination of the sag resistance of aqueous and nonaqueous liquid coatings at any level of sag resistance.

1.2 Procedure A is applicable to coatings of any type or color.

1.3 Procedure B is applicable to any type of coating but does not work well with dark colors.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 6, 1989. Published December 1989. Originally published as D 4400 - 84. Last previous edition D 4400 - 89.

² "Design of an Improved Sag Tester," *Official Digest*, Vol 34, No. 453, October 1962.

³ "Thixotropy . . . Trade Sales Paints," *Official Digest*, Vol 36, No. 468, January 1964.

D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁴

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials⁴

2.2 U.S. Federal Standards:⁵

Federal Test Method Standard 141, Method No. 494, Sag Test (Multinotch Blade)

2.3 U.S. Federal Specifications:⁵

Fed. Spec. TT-E-508 Alkyd semi-gloss enamel

Fed. Spec. TT-E-506 Alkyd gloss enamel

Fed. Spec. TT-P-1511 Interior latex gloss and semi-gloss finishes

3. Summary of Test Method

3.1 *Procedure A, Horizontal Stripes*—After preshearing the coating is applied to a test chart with a multinotch applicator. The charts are immediately hung vertically with the drawdown stripes horizontal, similar to rungs of a ladder with the thinnest stripe at the top. After drying in this position, the drawdown is examined and rated for sagging. A typical sag pattern obtained by this procedure is shown in Fig. 3.

3.2 *Procedure B, Vertical Stripes*—Two straight lines are

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

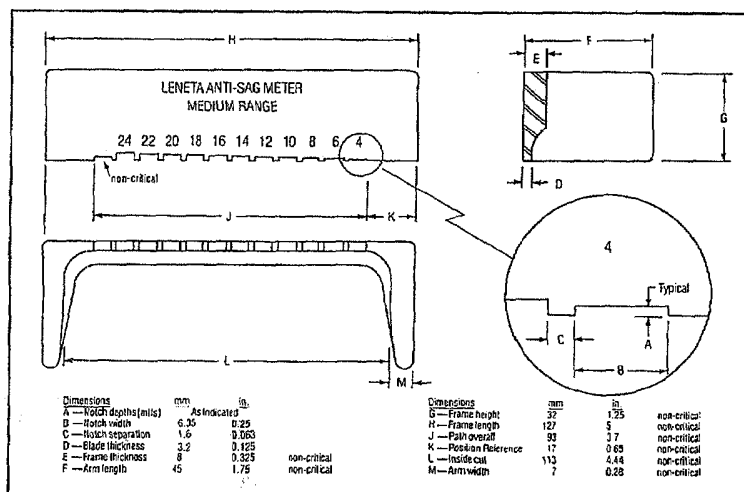


FIG. 1 Diagram of the Medium Range Anti-Sag Meter



FIG. 2 Medium Range Anti-Sag Meter

drawn across a test chart using a suitable marker pen. After preshearing, the coating is applied with a multinotch applicator so that the stripes are perpendicular to the lines. After waiting briefly for the marker ink to migrate through the wet film, the charts are hung vertically with the stripes vertical, similar to the slats of a picket fence, with the thinnest stripe on the left. After drying in this position, the ink lines are examined and rated for sagging. A typical sag pattern obtained by this procedure is shown in Fig. 4.

Significance and Use

4.1 Evaluation of sag resistance is essential in quality control for both producers and purchasers of coatings. Practical application tests are poor in reproducibility while isometric methods, for example Test Methods D 2196, are time-consuming and lack the convincing aspect of actual sagging. This method provides simple and rapid tests, whereby sag resistance is demonstrated by a visible sag pattern, and is rated objectively in terms of numerical values that correlate with brushout test observations.

Apparatus

5.1 *Multinotch Applicator*, Anti-Sag Meter⁶, a drawdown

⁶ Available from The Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ 07423. An equivalent may be used.

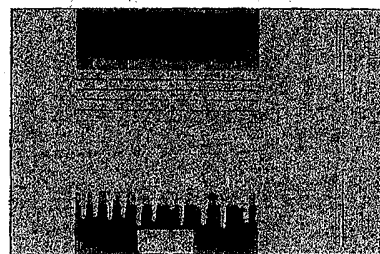


FIG. 3 Typical Sag Pattern—Procedure A

blade with a series of notches of successively higher clearance. Select a clearance range suitable for the type of coating under test in accordance with Table 1.

5.2 *Test Surfaces*, sealed, smooth-surfaced paper test charts, with sizes and designs as follows:

5.2.1 *Black and White Charts*,⁷ about 7 $\frac{3}{8}$ by 11 $\frac{1}{4}$ in. (193 by 288 mm), the black area comprising about 5 $\frac{1}{2}$ in. (140 mm) centered on the drawdown path. A chart of this design is shown in Figs. 3 and 5.

5.2.2 *Plain White Charts*,⁷ about 7 $\frac{3}{8}$ by 11 $\frac{1}{4}$ in. (193 by 285 mm).

5.2.3 *Plain White Charts*,⁷ about 5 $\frac{1}{2}$ by 11 $\frac{1}{4}$ in. (140 by 285 mm). The use of a chart of this description is shown in Fig. 4.

5.3 *Glass Drawdown Plate*, plus straightedge guide for attachment thereto.

⁷ The following are available from The Leneta Co.:
Black and White Charts, Form 7B.
Plain White Charts, 7 $\frac{3}{8}$ by 11 $\frac{1}{4}$ in., Form WB.
Plain White Charts, 5 $\frac{1}{2}$ by 11 $\frac{1}{4}$, Form WM.
Catch-Papers, Form CP-2.

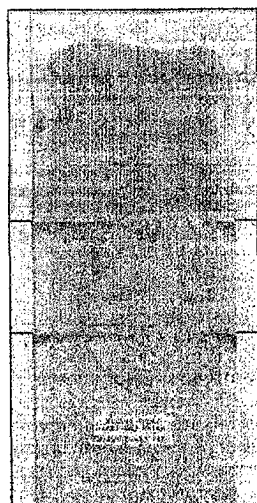


FIG. 4 Typical Sag Pattern—Procedure B

5.4 *Catch-papers*,⁷ thin sheets of sealed paper, for catching surplus paint at the completion of a drawdown.

5.5 *Fine-Line Marker Pens*—The ink shall be a solvent-based type that wets readily and dries quickly on smooth nonporous surfaces, specifically:

5.5.1 A pen⁸ that applies a hydrophilic ink line for use with water-dispersed coatings.

5.5.2 A pen⁹ that applies an oleophilic ink line for use with solvent-dispersed coatings.

5.6 *Equipment for the Preshearing of Aqueous Coatings:*

5.6.1 *Syringe*, 10-mL, Becton-Dickinson, Luer-Lok disposable plastic type.¹⁰

5.6.2 *Syringe Needle*, 15 g by 1½ in. (40 mm) to fit syringe.

5.6.3 *Syringe Extension Tubing*, clear vinyl, inside diameter ⅛ in. (3.2 mm), outside diameter ⅜ in. (5 mm).

5.7 *Equipment for the Preshearing of Nonaqueous Coatings:*

5.7.1 *Rotary Mechanical Stirrer*, variable speed.

5.7.2 *Circular Mixing Paddle*,¹¹ diameter approximately 1⅞ in. (48 mm).

5.7.3 *Mixing Container*, cylindrical jar or can with capacity of up to 1 pt (500 mL).

6. Procedure A—Horizontal Stripes

6.1 Preparation of Sample:

⁸ "Vis-A-Vis" pens, manufactured by Sanford Pen Company, Bellwood, IL 60104, have been found satisfactory. These are sold at commercial stationers.

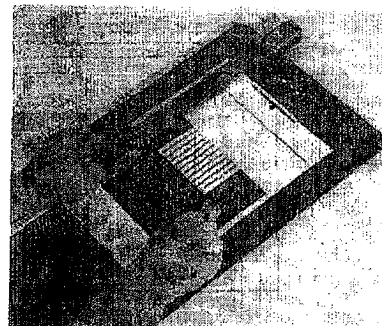
⁹ A "Flow Master" refillable pen with a fine line adapter and red Flow Master ink have been found satisfactory. The pen and the ink are sold separately. Both are manufactured by Faber-Castell Pen Corp. Newark, NJ 07107, and are available at commercial stationers.

¹⁰ Luer-Lok is a trademark of Becton-Dickinson, Beaver Brook Rd., Lincoln Park, NJ 07035. Available from distributors of general laboratory supplies.

¹¹ A mixing paddle (Catalog Number PM-70) with a diameter of 1⅞ in. (48 mm) made by Shur-Line Mfg. Co., 2200 Commerce Rd., Lancaster, NY 14086 has been found satisfactory for this purpose. This or equivalent paddles may be purchased at most hardware stores.

TABLE 1 Anti-Sag Meter Types

Type of Coating Under Test	Clearance Range, mils	Range Reference	Number of Clearances
Architectural	3–12	Standard	10
Industrial—OEM.	1–6	Low	11
High build	14–60	High	11
Architectural	4–24	Medium	11



NOTE—Note use of straightedge guide.

FIG. 5 Drawing Down with the Anti-Sag Meter

6.1.1 Stir thoroughly with a spatula in the original container.

6.1.2 Strain if necessary to remove large particles or skins.

6.1.3 Adjust the temperature of the coating to 73.5 ± 3.5°F (23 ± 2°C).

6.2 *Preshearing with Syringe and Needle (Aqueous Coatings):*

6.2.1 Prepare the paint as described in 6.1.

6.2.2 Cut a 2½-in. (60-mm) length of syringe extension tubing and attach it to the syringe.

6.2.3 Press the syringe barrel firmly to expel air, dip the end of the syringe into the coating, pump slightly to expel remaining air, then withdraw 8 mL of coating.

6.2.4 Remove and discard the extension tubing and then attach a syringe needle.

6.2.5 Eject the contents of the syringe in front of the applicator as rapidly as possible, with firm, steady pressure.

6.3 *Preshearing with a Rotary Mechanical Mixer (Nonaqueous Coatings):*

6.3.1 Prepare the paint as described in 6.1 and fill the mixing container slightly more than half. Set the container under the stirrer so that the paddle is about ¼ in. (5 mm) from the bottom.

6.3.2 Mix vigorously for 1 min at a speed sufficient to form a moderate vortex, with the entire contents of the can in rapid circular motion. Rotor speeds of 1300 to 3600 r/min have been found satisfactory, the optimum speed depending on the relative diameters of the mixing paddle and container. For referee tests the operators should agree upon the specific container, paddle, and mixing speed.

6.3.3 Immediately after mixing place about 8 mL of paint in front of the applicator and draw down in accordance with 6.4 (Procedure A) or 7.4 (Procedure B).

6.4 *Application of the Test Coating:*

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6.4.1 Affix a suitable test chart onto the drawdown plate. Use black and white charts in accordance with 5.2.1 for light or moderately dark colored coatings and white charts in accordance with 5.2.2 for very dark coatings.

6.4.2 Fasten the straightedge onto the drawdown plate in suitable position.

6.4.3 Place the Anti-Sag Meter at the far end of the chart, the open side toward the operator and shoulder against the straightedge guide.

6.4.4 If desired, position a catch-paper just underneath the lower edge of the chart.

6.4.5 Preshear in accordance with 6.2 or 6.3 and immediately draw down the coating at a uniform speed of about 6 in. (150 mm)/s, with the applicator pressed against the straightedge to maintain a straight path. See Fig. 5 for illustration of this step.

6.4.6 Immediately hang the chart with the drawdown stripes in a horizontal position like rungs in a standing ladder, the thinnest stripe at the top, and allow to dry in that position. A typical test pattern derived using this procedure is shown in Fig. 3.

6.5 Rating the Drawdown:

6.5.1 When the film is dry, note the notch numbers marked on the Anti-Sag Meter and identify the corresponding stripes accordingly.

6.5.2 Observe the sag pattern, ignoring the bottom stripe, which serves only as a position reference for the stripe above it, and the leading and trailing edges of the drawdown, considering only the central 5½ in. (140 mm) of the blade path. This corresponds to the black area of the black and white chart described in 5.2.1. (See Fig. 3 for a typical sag pattern of this type.)

6.5.3 Select the lowest (thickest) stripe that has resisted crossing the gap to touch the next lower stripe. This is referred to as the index stripe.

6.5.4 Estimate the degree to which the next lower stripe (the post-index stripe) has merged with the one below it, and determine the corresponding addendum fraction, as specified in Table 2.

6.5.5 Multiply the fraction from 6.5.4 by the difference between the index and post-index stripe number to obtain the index addendum.

6.5.6 Add the index addendum to the index stripe number to obtain the Horizontal Anti-Sag Index and record the same.

Procedure B—Vertical Stripes

7.1 Preparation of Sample—Follow procedure in 6.1.

7.2 Preshearing with Syringe and Needle (Aqueous Coatings)—Follow procedure in 6.2.

7.3 Preshearing with a Rotary Mechanical Mixer (Non-aqueous Coatings)—Follow procedure in 6.3.

TABLE 2 Intermediate Ratings

Degree of Merger of Post-Index Stripe	Addendum Fraction
Complete	0.0
Almost complete	0.2
Somewhat more than half	0.4
Half	0.5
Somewhat less than half	0.6
Slight (just touching)	0.8

7.4 Application of Test Coating:

7.4.1 On a plain white test chart as described in 5.2.3, using the appropriate pen described in 5.5, rule two parallel lines about 1½ in. (40 mm) apart, across the intended path of the drawdown blade (see Fig. 4). If desired the operator can prepare a supply of such charts for future use, since the ink line is unaffected by aging.¹²

7.4.2 Affix the ruled test chart onto the drawdown plate and fasten the straightedge in suitable position.

7.4.3 Place the Anti-Sag Meter at the top of the chart, the open side toward the operator and shoulder against the straightedge guide.

7.4.4 If desired, position a catch-paper just underneath the lower edge of the chart.

7.4.5 Preshear in accordance with 6.2 or 6.3 and immediately draw down the coating at a uniform speed of about 6 in. (150 mm)/s, with one arm of the applicator pressed gently against the straightedge to maintain a straight path. This procedure is the same as that referred to in 6.4 and illustrated in Fig. 5.

7.4.6 Fifteen seconds after completing the drawdown, hang the chart with the stripes vertical (like fence pickets), thinnest stripe to the left, and allow to dry in that position. A typical test pattern obtained using this procedure is shown in Fig. 4.

7.5 Rating the Drawdown:

7.5.1 When the film is dry, note the notch numbers marked on the Anti-Sag Meter and identify the corresponding stripes and sag loops accordingly.

7.5.2 Examine Fig. 6 and note the "reference loop," which was selected because it indicates a limited but unambiguous sag movement. By actual measurement this loop represents ¼ in. (1.5 mm) of sagging, which amount can be considered as defining the reference loop.

7.5.3 For each of the two ink lines on the test drawdown, note the loop that shows the same or almost as much sag as the reference loop. This is referred to as the "index" loop. The loop to its immediate right is the "post-index" loop and the corresponding stripes are the "index" and "post-index" stripes.

7.5.4 Estimate the visual difference between the index and reference loops as a fraction (to the nearest fifth or half) of the difference between the index and post-index loops. This is the addendum fraction, permissible values being 0, 0.2, 0.4, 0.5, 0.6, or 0.8.

7.5.5 Multiply the addendum fraction from 7.5.4 by the difference between the index and post-index stripes to obtain the index addendum.

7.5.6 For each ink line, add the index addendum to the index stripe number to obtain the Vertical Anti-Sag Index and record the mean value.

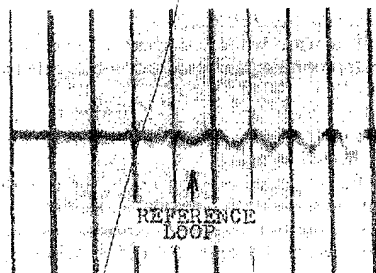
8. Report

8.1 Report the following information:

8.1.1 Procedure A—The Horizontal Anti-Sag Index (HASI) of the coating as recorded in 6.5.6.

8.1.2 Procedure B:

¹² Charts with suitable ink lines are available from The Leneta Co. Refer to Form WM-BL (Blue Line) and Form WM-RL (Red Line) for hydrophilic and oleophilic ink lines respectively.



NOTE—The "reference loop" is 1.5 mm (1/16 in.) deep.

FIG. 6 Sag Loops Obtained with Procedure B

8.1.2.1 The Vertical Anti-Sag Index (VASI) of the coating as recorded in 7.5.6.

9. Precision¹³

9.1 *Correlation*—In an interlaboratory study in which operators in seven laboratories tested six water-reducible paints covering a wide range of sag resistance and in five laboratories tested four solvent-reducible paints covering a wide range of sag resistance, the Spearman Rank Correlation Coefficient was 0.92 for Procedure A and 0.96 for Procedure B versus brushouts (a coefficient of 1.0 indicates perfect agreement in ranking).

9.2 *Sensitivity*—In the interlaboratory study described in 9.1, the sensitivity criterion values have been computed to be 4 for brushouts, 11 for Procedure A, and 13 for Procedure B.

¹³ Supporting data are available from ASTM Headquarters. Request RR: D01-1040.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Thus, Procedures A and B are approximately three times more sensitive to differences in sag resistance than brushouts.

9.3 Precision:

9.3.1 Two interlaboratory tests were conducted to establish the precision of this test method. The first test was that described in 9.1. The second test consisted of operators in five laboratories performing three tests on each of three paints. On the basis of the second interlaboratory test, the within-laboratory pooled coefficients of variations for both water- and solvent-reducible paints were 4.4 % for Procedure A and 5.0 % for Procedure B. On the basis of the first interlaboratory test, the between-laboratory pooled coefficient of variation for Procedure A was found to be 12.4 % for water-reducible paints and 8.8 % for solvent-reducible paints. The between-laboratory pooled coefficient of variation for Procedure B was found to be 8.0 % for water-reducible paints and 6.6 % for solvent-reducible paints. Based on these coefficients of variation, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.3.1.1 *Repeatability*—Two results obtained by the same operator for either water-reducible or solvent-reducible paints should be considered suspect if they differ by more than 10.7 % using Procedure A and by more than 13.6 % using Procedure B.

9.3.1.2 *Reproducibility*—Two results obtained by operators in different laboratories using Procedure A should be suspect if they differ by more than 34.4 % for water-reducible paints and 23.4 % for solvent-reducible paints. Two results obtained by operators in different laboratories using Procedure B should be suspect if they differ by more than 23.1 % for water-reducible paints and 19.8 % for solvent-reducible paints.

10. Keywords

10.1 Anti-Sag Index; rheological properties; sag-resistance



Standard Practice for Measurement of Wet Film Thickness by Notch Gages¹

This standard is issued under the fixed designation D 4414; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Sections 5 through 10 were editorially renumbered in May 1990.

1. Scope

1.1 This practice describes the use of thin rigid metal notched gages, also called step or comb gages, in the measurement of wet film thickness of organic coatings, such as paint, varnish, and lacquer.

1.2 Notched-gage measurements are neither accurate nor sensitive, but they are useful in determining approximate wet film thickness of coatings on articles where size(s) and shape(s) prohibit the use of the more precise methods given in Methods D 1212.

1.3 This practice is divided into the following two procedures:

1.3.1 *Procedure A*—A square or rectangular rigid metal gage with notched sides is used to measure wet film thicknesses ranging from 0.5 to 80 mils (13 to 2000 μm). Such a gage is applicable to coatings on flat substrates and to coatings on articles of various sizes and complex shapes where it is possible to get the end tabs of the gage to rest in the same plane on the substrate.

1.3.2 *Procedure B*—A circular thin rigid metal notched gage is used to measure wet film thicknesses ranging from 1 to 100 mils (25 to 2500 μm). Such a gage is applicable to coatings on flat substrates and to coatings on objects of various sizes and complex shapes.

1.4 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standard:

D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings²

3. Summary of Practice

3.1 The material is applied to the articles to be coated and the wet film thickness measured with a notched gage.

3.2 *Procedure A*—A square or rectangular thin rigid metal gage with notched sides, having tabs of varying lengths, is pushed perpendicularly into the film. After removal from the film, the gage is examined and the film thickness is deter-

mined to lie between the clearance of the shortest tab wet by the film and the clearance of the next shorter tab not wetted by the film.

3.3 *Procedure B*—A circular thin rigid metal gage having spaced notches of varying depths around its periphery is rolled perpendicularly across the film. After removal from the film, the gage is examined and the film thickness is determined as being between the clearance of the deepest face wetted and the clearance of the next deepest notch face not wetted by the film.

4. Significance and Use

4.1 Wet film thickness measurements of coatings applied on articles can be very helpful in controlling the thickness of the final dry coating, although in some specifications the wet film thickness is specified. Most protective and high performance coatings are applied to meet a requirement or specification for dry film thickness for each coat or for the completed coating system, or for both.

4.2 There is a direct relationship between dry film thickness and wet film thickness. The wet film/dry film ratio is determined by the volume of volatiles in the coating as applied, including permitted thinning. With some flat coatings the dry film thickness is higher than that calculated from the wet film thickness. Consequently, the results from the notch gage are not to be used to verify the nonvolatile content of a coating.

4.3 Measurement of wet film thickness at the time of application is most appropriate as it permits correction and adjustment of the film by the applicator at the time of application. Correction of the film after it has dried or chemically cured requires costly extra labor time, may lead to contamination of the film, and may introduce problems of adhesion and integrity of the coating system.

4.4 The procedures using notched gages do not provide as accurate or sensitive measurements of wet film thickness as do the Interchemical and Pfund gages described in Methods D 1212. Notch gages may, however, be used on nonuniform surfaces, like concrete block, that are too rough to use the Interchemical and Pfund gages. Also notched gages can be very useful in the shop and field for determining the approximate thickness of wet films over commercial articles where size(s) and shape(s) are not suitable for measurements by other types of gages. Examples of such items are ellipses, thin edges, and corners.

4.5 An operator experienced in the use of a notched gage can monitor the coating application well enough to ensure the minimum required film thickness will be obtained.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee B01.23 on Physical Properties of Applied Paint Films.

Current edition approved Oct. 26, 1984. Published January 1985.

² Annual Book of ASTM Standards, Vol 06.01.

4.6 Application losses, such as overspray, loss on transfer, and coating residue in application equipment, are a significant unmeasurable part of the coating used on a job and are not accounted for by measurement of wet film thickness.

5. Report

5.1 Report the following information:

5.1.1 The mean and range of the readings taken and the number of readings.

5.1.2 The smallest graduation of the gage used.

6. Precision and Bias

6.1 The precision and bias of Procedure A or B for measuring wet film thickness with notch gages are very dependent on methods of film application, time that the measurement is taken after film application, mechanical condition of the notch gages, and the step range of the gages.

6.2 Generally, the agreement between notch gages is good because they are insensitive to small differences in film thickness, that is the step intervals of the gages are relatively large.

PROCEDURE A

7. Apparatus

7.1 *Notched Gage*, square or rectangular, thin rigid metal plate, with notched sides (see Fig. 1), made from steel or aluminum³ (Note 1). Nonmetallic gages shall not be used.

NOTE 1—Aluminum or aluminum alloy gages are more easily distorted and may exhibit greater wear than steel gages. Gages made of plastic or deformable metal are not suitable.

7.1.1 Each notched side shall consist of a series of tabs (between notches) varying in length and located in a line between two end tabs equal in length and longest in the row.

7.1.2 As an example, the tabs on one row of a gage may differ in length as follows:

By 0.5 mil (13 μm) between 0 and 6 mils (0 to 150 μm),

By 1 mil (25 μm) between 6 and 10 mils (150 to 250 μm),

By 2 mils (50 μm) between 10 and 30 mils (250 to 750 μm), and

By 5 mils (125 μm) over 30 mils (750 μm).

8. Procedure

8.1 Apply the coating material to a rigid substrate and test with the gage immediately. The gage must be used immediately following application of the coating. Some coatings lose solvents quickly and spray application increases the speed. The resulting rapid reduction in wet film thickness can cause misleading readings.

8.2 Locate an area sufficiently large to permit both end tabs of the gage to rest on the substrate in the same plane.

8.3 Push the gage perpendicularly into the wet film so that the two end tabs rest firmly on the substrate at the same time.

8.4 Or, set one end tab firmly on the substrate and lower the gage until the other end tab is firmly in contact with the substrate.

8.5 Remove the gage from the film and examine the tabs.

³ These gages are commercially available from various coating equipment and instrument suppliers.

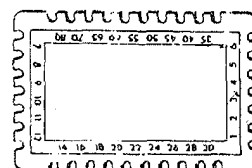


FIG. 1 Rectangular Notched Gage

The film thickness is determined as being between the clearance of the shortest tab wetted and the clearance of the next shorter tab not wetted by the film.

8.6 Clean the gage immediately after each reading by wiping it on a dry or solvent-dampened cloth so that subsequent readings are not affected. Do not clean with metal scrapers.

8.7 Repeat the procedure in 8.2 through 8.5 for at least three locations on the film. The number of readings required to obtain a good estimate of the film thickness varies with the shape and size of the article being coated, with the operator's experience, and whether one or more of the following problems are encountered:

8.7.1 Some coatings may not wet (leave residue on) some metal gages. However, the film itself may show where contact was made. When reading the gage, look at both the gage and the film itself for verification of the reading.

8.7.2 The gage may slip on the surface. Ignore such readings.

8.7.3 The surface may be coarse and false readings produced. The spot where the gage is used must be as uniform as possible and questionable readings ignored.

8.8 Determine the mean and range of the readings.

PROCEDURE B

9. Apparatus

9.1 *Circular Notched Gage*, thin aluminum disk, with notches of various depths spaced around its periphery (see Fig. 2). Each notch has a recessed flat face. A $\frac{3}{8}$ -in. (10-mm) hole is in the center of the disk.

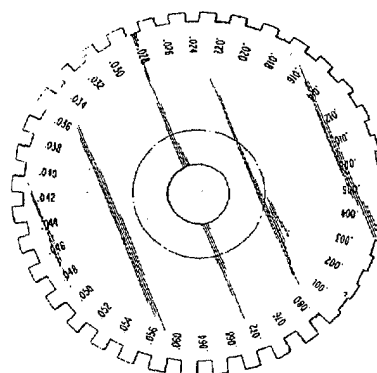


FIG. 2 Circular Notched Gage



Standard Test Methods for Field Measurement of Surface Profile of Blast Cleaned Steel¹

1. Scope

1.1 These test methods cover the description of techniques for measuring the profile of abrasive blast cleaned surfaces in the laboratory, field, or in the fabricating shop. There are additional techniques suitable for laboratory use not covered by these test methods.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Method

2.1 The methods are:

2.1.1 *Method A*—The blasted surface is visually compared to standards prepared with various surface profile depths and the mode determined.

2.1.2 *Method B*—The depth of profile is measured using a fine pointed probe at a number of locations and the arithmetic mean determined.

2.1.3 *Method C*—A composite plastic tape is impressed into the blast cleaned surface forming a reverse image of the profile, and the peak to valley height on the tape measured with a micrometer.

3. Significance and Use

3.1 The height of surface profile has been shown to be a factor in the performance of various coatings applied to steel. For this reason, surface profile should be measured prior to coating application to ensure that it meets the specification. The instruments described are readily portable and sufficiently sturdy for use in the field.

NOTE—Optical microscope methods serve as a referee method for surface profile measurement. Profile depth designations are based on the concept of average maximum profile ($\bar{h}$ max); this value is determined by averaging a given number (usually 20) of the highest peak to lowest valley measurements made in the field of view of a standard measuring microscope. This is done because of evidence that coatings performance in any one small area is primarily influenced by the highest surface features in that area and not by the average roughness.²

4. Apparatus

4.1 *Method A*—A profile comparator³ consisting of a number of areas (each approximately one square inch in size), usually side by side, with a different profile or anchor

pattern depth. Each area is marked giving the nominal profile depth in mils or micrometres. Typical comparator surfaces are prepared with steel shot, steel grit, or sand or other nonmetallic abrasive, since the appearance of the profile created by these abrasives may differ. The comparator areas are used with or without magnification of 5 to 10 $\times$.

4.2 *Method B*—A dial gage⁴ depth micrometer fitted with a pointed probe. The probe is machined at a 60° angle with a nominal radius of 50 μ m. The base of the instrument rests on the tops of the peaks of the surface profile while the spring loaded tip projects into the valleys.

4.3 *Method C*—A special tape containing a compressible foam attached to a noncompressible uniform plastic film. A burnishing tool is used to impress the foam face of the tape into the surface to create a reverse replica of the profile that is measured using a spring-loaded micrometer.⁵

5. Test Specimens

5.1 Use any metal surface that, after blast cleaning, is free of loose surface interference material, dirt, dust, and abrasive residue.

6. Procedure

6.1 *Method A:*

6.1.1 Select the comparator standard appropriate for the abrasive used for blast cleaning.

6.1.2 Place the comparator standard directly on the surface to be measured and compare the roughness of the prepared surface with the roughness on the comparator segments. This can be done with the unaided eye, under 5 to 10 $\times$ magnification, or by touch. When using magnification, the magnifier should be brought into intimate contact with the standard, and the depth of focus must be sufficient so that the standard and surface are in focus simultaneously.

6.1.3 Select the comparator segment that most closely approximates the roughness of the surface being evaluated or, if necessary, the two segments to which it is intermediate.

6.1.4 Evaluate the roughness at a sufficient number of locations to characterize the surface as specified or agreed upon between the interested parties. At each location make three evaluations against the comparator and determine the mode. The mode of the three evaluations represents the profile at the specific location, with the mode of all locations representing the profile of the entire surface.

6.2 *Method B:*

6.2.1 Prior to use set the gage to zero by placing it on a piece of plate glass. Hold the gage by its base and press firmly against the glass. Adjust the instrument to zero.

6.2.2 To take readings, hold the gage firmly against the prepared substrate. Do not drag the instrument across the

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.46 on Industrial Protective Painting.

Current edition approved Nov. 14, 1984. Published January 1985.

² John D. Keane, Joseph A. Bruno, Jr., Raymond E. F. Weaver, "Surface Profile for Anti-Corrosion Paints," Oct. 25, 1976. Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

³ Suitable comparators include Keane-Tator Surface Profile Comparator and Clemtex coupons.

⁴ Suitable depth micrometers include the Elcometer Model 123 Surface Profile Gage.

⁵ Suitable replica tape and micrometers include Testex "Press-O-Film" tape and Mitutoyo Model 7326 Spring Micrometer.

surface between readings, otherwise the spring-loaded tip may become rounded.

6.2.3 Measure the profile at a sufficient number of locations to characterize the surface, as specified or agreed upon between the interested parties. At each location make ten readings and determine the mean. Then determine the mean for all the locations and report it as the profile of the surface.

6.3 Method C:

6.3.1 Select the correct tape range for the profile to be measured: coarse, 0 to 2 mils (0 to 50 μm) and extra coarse, 2.5 to 4.5 mils (40 to 115 μm).

6.3.2 Remove the wax paper backing and place the tape on the prepared surface with the foam side down, that is, put the dull side down.

6.3.3 Hold the tape firmly on the surface and rub the circular cut-out portion (approximately $\frac{3}{8}$ in. (6.5 mm) diameter) with the burnishing tool until a uniform gray color appears.

6.3.4 Remove the tape and place it between the anvils of a spring-loaded micrometer. Measure the thickness of the tape

(compressed foam and non-compressible plastic film combined). Subtract the thickness of the noncompressible plastic film to obtain the surface profile.

6.3.5 Measure the profile at a sufficient number of locations to characterize the surface, as specified or agreed upon between the interested parties. At each location make three readings and determine the mean. Then determine the mean for all the locations and report it as the profile of the surface.

7. Report

7.1 Report the range and the appropriate average (mean or mode) of the determinations, the number of locations measured, and the approximate total area covered.

8. Precision

8.1 Precision is under active study.⁶

⁶ Other organizations, such as National Association of Corrosion Engineers (NACE), are currently involved in such studies.

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Standard Test Method for Visual Evaluation of Gloss Differences Between Surfaces of Similar Appearance¹

This standard is issued under the fixed designation D 4449; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers the visual evaluation of gloss differences of coating surfaces, using special types of lamps for illumination. It identifies six aspects or types of gloss that one may look for when using the lamp to assess gloss differences between surfaces. It describes the conditions for using the lamps to best identify small differences in each of the six types of gloss. Four levels of visual gloss differences are distinguished.

1.2 While this technique is useful for both weathered and unweathered specimens, it has not been applied to metallics.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 3964 Practice for Selection of Coating Specimens for Appearance Measurements²

E 284 Definitions of Terms Relating to Appearance of Materials³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method, see Definitions E 284.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *directionality of surface*—perceived change of glossy appearance of a surface with rotation of the surface in its own plane (see Fig. 1).

3.2.2 *gloss, distinctness-of-image*—perceived sharpness of images reflected by an object surface (see Fig. 2).

3.2.3 *gloss, of a surface*—perceived directionally selective reflecting properties responsible for the degree to which reflected highlights or images of objects may be seen as superimposed on the surface.

3.2.4 *reflection haze*—cloudy or milky appearance of a

surface adjacent to directions of specular reflection (see Fig. 3).

3.2.5 *sheen*—perceived shininess at a near-grazing angle of incidence for an otherwise matte specimen (difficult to photograph).

3.2.6 *specular gloss*—the relative luminous reflectance factor of a specimen in the specular direction. The luminous reflectance factor is the ratio of the luminous flux reflected from, to that incident on, a specimen for specified solid angles (see Fig. 4).

3.2.7 *texture*—perceived structure, pattern, or topography or combination thereof, of a surface (see Fig. 5).

4. Summary of Test Method

4.1 Test specimens are illuminated by special light sources that provide reflected images suitable for rating the gloss of coating surfaces. Two types of lamps may be used to produce reflected images. Lamp A consists of a modified fluorescent desk lamp covered with screen mesh. Lamp B consists of an incandescent bare filament bulb installed in an adjustable angle fixture.

4.2 Light from the selected lamp illuminates the test specimens. The reflected images reveal specular gloss reflection; the sharpness of the images of the screen or lamp filament reveals the detail and quality of the surface reflection (distinctness of image); and spreading of the reflected light into dark areas, such as the space between fluorescent tubes or near the filament image, reveals the presence of near-specular haze.

5. Significance and Use

5.1 Gloss⁴ is associated with the capacity of a surface to reflect more light in some directions than in others. The directions associated with mirror (or specular) reflection normally have the highest reflectances. Gloss is best seen and analyzed when the surfaces studied are illuminated by a light source that provides strong contrasting patterns of light and dark. Such a light source is described in this test method.

5.2 The simplest concept of gloss is that it corresponds to the mirror-like reflectances of surfaces. However, the distributions and intensities of this surface-reflected light are (for real materials) highly variable and affected by a variety of factors: surface smoothness and contour, refractive index, absorptance, angle of incidence, and (to a generally small extent) wavelength. From the great variety of surfaces

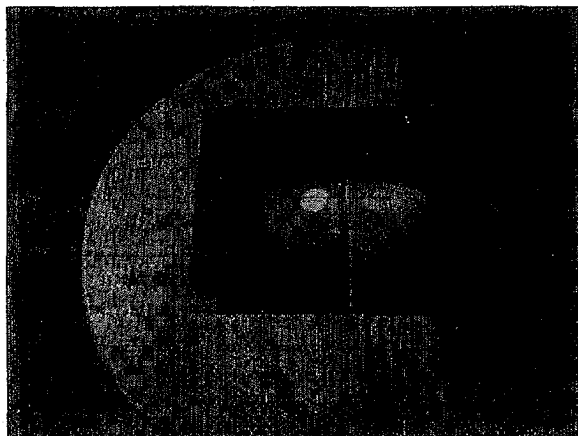
¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.26 on Optical Properties.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as D 4449 - 85. Last previous edition D 4449 - 85 ϵ .

² Annual Book of ASTM Standards, Vol 06.01.

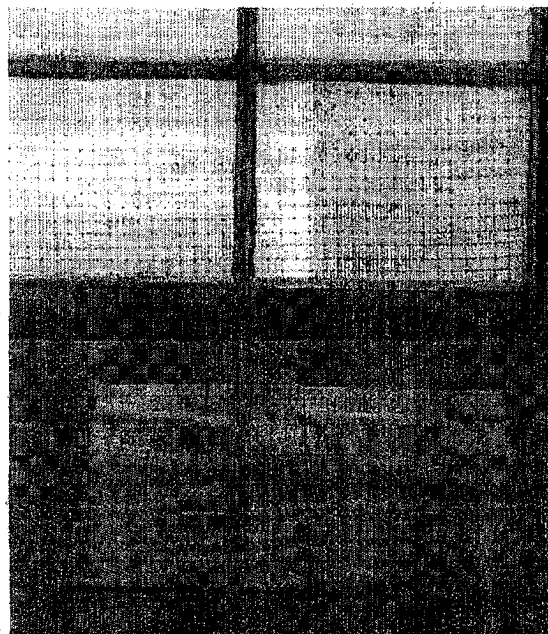
³ Annual Book of ASTM Standards, Vol 14.02.

⁴ For a more detailed account of gloss, its measurement, and relationship to appearance generally, see R. S. Hunter, *The Measurement of Appearance*, Wiley-Interscience New York, NY 1975.



NOTE—The left panel is free of directionality. The right panel is highly directional because of buffing marks.

FIG. 1 Reflection of Bright Incandescent Lamp in Two Panels



NOTE—The panels exhibit a difference in distinctness-of-reflected image.

FIG. 2 Two White Porcelain Enamel Panels

Reflection patterns met in materials of commerce, it has been possible to identify seven surface-reflection criteria or "types of gloss" regularly used by skilled technologists for intercomparing and rating their products for gloss. Six of the seven criteria, or "types of gloss," are identified in the section on definitions. The seventh, luster or contrast gloss, is seldom of

concern to the coatings industry.

6. Apparatus

6.1 *Lamp A*—The recommended apparatus is constructed by modifying a conventional fluorescent desk lamp that has two 15-W, 18-in. (450-mm) tubes. Figure 6 is a photograph;

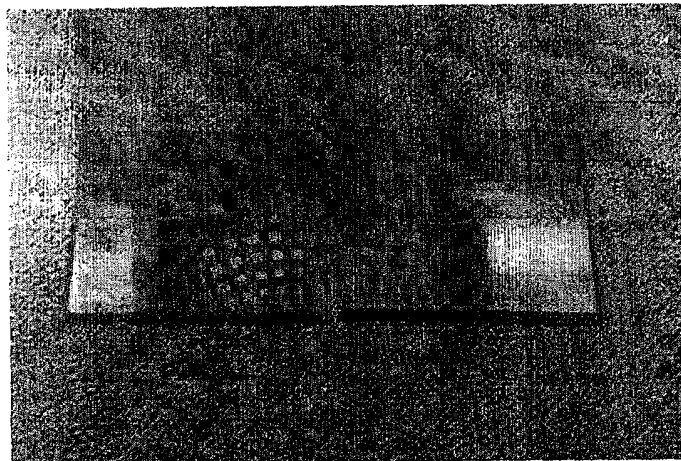


FIG. 3 Two Blue Plastic Wall Tiles Exhibiting a Difference in Reflection Haze

and care of the surface, while preparing and examining it for gloss, are critically important.

7.2 Methods for preparing paint surfaces for examination

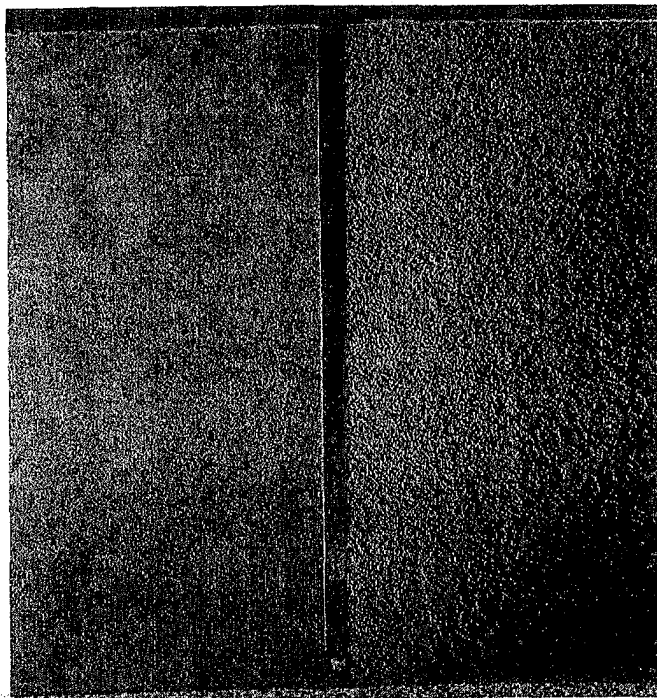
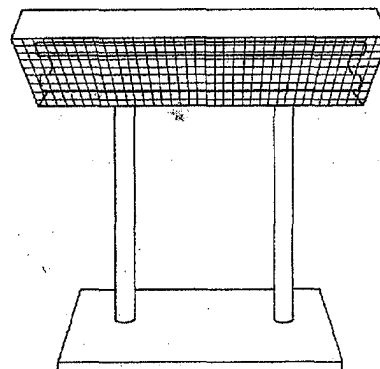


FIG. 5 Two Painted Panels Differing in Texture



NOTE—The lamp is used to demonstrate gloss or geometric differences in the appearances of surfaces.

FIG. 6 Modified Desk Lamp



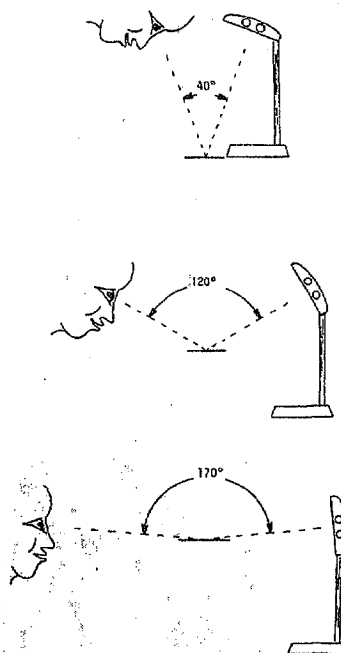
NOTE—The lamp is modified by the addition of a black reflecting surface behind tubes and a wire screen in front.

FIG. 7 Desk Lamp

Identify differences between each pair of surfaces compared as either:

None (or indistinguishable)	= 0
Very slight (barely distinguishable)	= 1
Moderate (easily seen)	= 2
Considerable	= 3

9.1.4 Which of the pair appears glossier (except in first case).



(a) For high specular gloss, distinctness-of-image gloss, and reflection haze
(b) For intermediate and low specular gloss and directionality of plane surfaces
(c) For sheen

FIG. 8 Recommended Positions of Gloss Inspection Lamp, Observer, and Surfaces of Different Levels of Gloss Being Intercompared

10. Precision

NOTE—The precision study has not been completed.

10.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than two in ratings.

10.2 *Reproducibility*—Two results, each the mean of two

runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 2.2 in ratings.

11. Keywords

11.1 distinctness of image; gloss; haze; visual gloss

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Standard Test Method for Pigment Content of Paints by Low-Temperature Ashing¹

This standard is issued under the fixed designation D 4451; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Keywords were added editorially in July 1991.

Scope

1.1 This test method covers the pigment content of paints and several traffic marking materials (thermoplastic and preformed tape) by low-temperature furnace ashing. Some organic pigments may be lost by this method and some water moisture contained in pigments will be lost.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 ASTM Standards:

D 3723 Test Method for Pigment Content of Water-Emulsion Paints by Low-Temperature Ashing²

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

3. Summary of Test Method

3.1 The specimen is transferred to a tared porcelain dish, dried (if necessary) at 105°C, and heated on a burner. The dish and specimen are transferred to a muffle furnace and heated at 450°C. The dish and specimen are reweighed and the pigment (ash) content calculated.

4. Apparatus

- 4.1 Muffle Furnace, maintained at 450°C $\pm$ 25°C.
- 4.2 Circulating Oven, maintained at 105°C $\pm$ 2°C.
- 4.3 Porcelain Dishes, 90-mm diameter.
- 4.4 Plastic Disposable Syringe, 10-mL capacity.
- 4.5 Burner, meker type.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chem-

ical Society, where such specifications are available.⁴

5.2 Toluene.

6. Procedure

6.1 Liquid Paint:

6.1.1 Mix the sample until homogeneous, preferably on a mechanical shaker. If air bubbles become entrapped in the paint, stir it by hand.

6.1.2 Draw slightly more than 10 g of the paint under test into a 10-mL syringe and weigh to 0.1 mg. Transfer about 5 mL toluene to a porcelain dish tared with a paper clip for use as a stirrer. Add 10 g of the material into the toluene. Reweigh the syringe to 0.1 mg and calculate the specimen weight. Mix well on a magnetic stirrer. Place the dish in the oven at 105°C for 30 min.

6.1.3 Remove and heat at the lowest temperature possible over a meker burner in a fume hood. Do not leave the dish on the burner after the flame has subsided. Transfer to the muffle furnace and proceed as in 6.4.

6.2 *Preformed Traffic Marking Tape*—Cut about a 10-g square of the product. Remove the adhesive by pulling it off or by using an appropriate solvent. Save the beads that are knocked off by this process and weigh with the tape. Dry the specimen for 30 min at 105°C to remove the solvent. Cool in a desiccator. Weigh the specimen along with the loose beads to 0.1 mg into a tared porcelain dish. Heat in a fume hood at the lowest temperature of a meker burner in a furnace hood until the material catches fire. Do not leave the dish on the burner after the flame has subsided. Transfer to the muffle furnace and proceed as in 6.4.

6.3 *Thermoplastic Traffic Marking Material*—This material may be delivered in block or powdered form. Transfer about 400 g of the sample to a quart container and heat in a forced draft oven at 400°F until completely melted (may take as long as 4 h). Stir vigorously until well mixed. Pour about 10 g of the sample into an aluminum dish and let it cool. Remove the 10 g wafer from the aluminum dish and weigh to 0.1 mg. Transfer to a tared porcelain dish. Heat the dish at the lowest temperature of a meker burner in a fume hood until the specimen catches fire. Do not leave the dish on the burner after the flame has subsided. Transfer to a muffle furnace and proceed as in 6.4.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Jan. 25, 1985. Published December 1985.

² Annual Book of ASTM Standards, Vol. 06.01.

³ Annual Book of ASTM Standards, Vol. 15.05.

⁴ "Reagent Chemicals. American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

6.4 Place the porcelain dish in a muffle furnace in a fume hood at 450°C (see Test Method D 3723). Heat overnight or until no further carbonaceous material is noted. Cool in a desiccator and reweigh the dish plus the residue to 0.1 mg. Calculate the residue as percent pigment or, in the case of thermoplastic material and preformed tape, as pigment and beads.

7. Calculation

7.1 Calculate the percent pigment, P , as follows:

$$P = C - A/S \times 100$$

where:

C = weight of dish and specimen after ignition in furnace, g,

A = weight of dish alone, g, and

S = specimen weight used, g.

8. Precision

8.1 The precision estimates are based on an interlaboratory study in which one operator in 7 different laboratories

analyzed in duplicate on two days six samples of commercial whole paint or thermoplastic material containing 50 to 80 % pigment. The results were analyzed statistically in accordance with Practice E 180 and the within-laboratory coefficient of variation was found to be 0.10 % relative at 36 df and the between-laboratories coefficient of variation 0.25 % relative at 30 df. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.28 % relative.

8.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.72 % relative.

9. Keywords

9.1 ignition, pigment; low temperature ashing; pigment content of paints

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph¹

This standard is issued under the fixed designation D 4457; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Keywords were added editorially in July 1991.

Scope

1.1 This test method covers the determination of total amount of dichloromethane or 1,1,1-trichloroethane, or both, in paints and coatings. It has been evaluated for cellulose nitrate, alkyd, vinyl, and styrene-butadiene systems. It has not yet been evaluated for other formulations, but is believed to be applicable. The established working range of this test method is from 31 to 65 % for 1,1,1-trichloroethane and 32 to 78 % for dichloromethane. There is no reason to believe it will not work outside of these ranges. The presence of 1-propanol in paints and coatings requires the use of a different internal standard. (See also Practice E 260.)

1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7.

Referenced Documents

2.1 ASTM Standards:

E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals²

E 260 Practice for Packed Column Gas Chromatography³

Summary of Test Method

3.1 Anhydrous 1-propanol (see 10.5) is added as an internal standard to suitable aliquot of the whole paint. The aliquot is then diluted with dimethylformamide and injected onto a gas chromatographic column containing a porous polymer packing that separates dichloromethane and 1,1,1-trichloroethane from other volatile compounds.

Significance and Use

4.1 Use of 1,1,1-trichloroethane and dichloromethane, which do not measurably contribute to the atmospheric oxidant level, is a way for industry to meet government or

other regulations on volatile organic compounds. This test method is designed to determine the content of these halohydrocarbon solvents in paints and coatings. That content can subsequently be used in calculating the volatile organic compound content of a coating.

5. Apparatus

5.1 *Chromatograph*, any gas-liquid chromatographic instrument equipped with a thermal conductivity detector and capable of being temperature programmed (see Table 1). Optionally, a flame ionization detector may be used if the sample is diluted so that no more than 1000 ppm each of dichloromethane and 1,1,1-trichloroethane is present in the injected specimen.

5.2 *Recorder*, a recording potentiometer with a full-scale deflection of 10 mV, a full-scale response time of 2 s or less, and a maximum noise of ± 0.03 % of full scale.

5.3 *Pre-Column*, 40 in. (100 mm) long by $\frac{1}{8}$ in. (3.2 mm) outside diameter stainless steel, packed with glass wool, fitted on the entrance end of the column to retain any nonvolatile materials and minimize sludge buildup in the column.

5.4 *Column*, 4 ft (1.22 m) long by $\frac{1}{8}$ in. (3.2 mm) outside diameter stainless steel, packed with 80/100 mesh (150 to 180 μ m) porous polymer packing material,⁴ or other suitable material.

5.5 *Liquid Charging Devices*, such as microsyringes of 5- μ L or 10- μ L capacity, cleaned with acetone or other suitable solvent. Visually inspect for plugs or cracks before and after each injection.

5.6 *Vials*, 25-mL to minimize head space, capable of being septum sealed.⁵

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests, unless otherwise specified (as in 6.7). Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paint and Paint Materials.

Current edition approved July 16, 1985. Published October 1985.

² Annual Book of ASTM Standards, Vol 15.05.

³ Annual Book of ASTM Standards, Vol 14.01.

⁴ Porapak R[®], available from Waters Associates, Inc., Milford, MA, has been found satisfactory for this purpose.

⁵ Mininert valves, available from The Pierce Chemical Co., Box 117, Rockford, IL 61105, have been found satisfactory for this purpose.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the

provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

6.2 *Carrier Gas*, helium of 99.995 % or higher purity. High purity nitrogen may also be used.

6.3 *Dimethylformamide (DMF)*, reagent grade.

6.4 *1-Propanol*, gas chromatography spectrophotometric quality (see 10.5).

6.5 *1,1,1-Trichloroethane* (see 6.7).

6.6 *Dichloromethane* (see 6.7).

6.7 *Halogenated Hydrocarbon Stabilizers*—All commercial grades of these halogenated hydrocarbons contain stabilizers. Either obtain the same solvent used in the coating for use as the standard, or find the type and quantity of stabilizer specified for use in the solvent of interest and add the appropriate quantity to the pure solvent.

7. Hazards

7.1 Dimethylformamide is harmful if inhaled or absorbed through skin. Use only with adequate ventilation. Avoid contact with skin, eyes, and clothing.

8. Preparation of Apparatus

8.1 *Column Conditioning*—The packed column is installed in the gas chromatographic unit leaving the exit end disconnected from the detector. This will prevent any contamination of the detector with the column bleed. Set the helium flow rate at 30 mL/min if a 1/8 in. (3.2 mm) outside diameter column is used. Purge the column 5 to 10 min before heating. Heat the column from room temperature to 200°C at 5°C/min and hold this temperature for at least 12 h (overnight). At the end of this period of time, heat the column to 240°C at a 5°C/min rate and hold this temperature for several hours. The maximum temperature for this packing is 250°C. Cool the column to 100°C and reheat to 240°C at 5°C/min to observe the column bleed. Optimum conditioning of this column may take several cycles of the heating program before a good recorder baseline is achieved. Conditioning of any column other than that suggested (5.4) should be in accordance with the manufacturer's recommendations.

8.2 Install the column in the chromatograph and use the information in Table 1 as a guide to establish the conditions required to give the desired separation. Allow sufficient time for the instrument to reach equilibrium as indicated by a stable recorder baseline. Adjust the carrier-gas flow to a constant rate. Before each calibration and series of determinations (or daily), condition the column at 200°C for 1 h with carrier-gas flow.

9. Calibration

9.1 *Preparation of Standards*—All standards, as well as samples and blanks, should be at a constant temperature. The given order of ingredient addition should be observed to minimize loss of volatile ingredients.

9.1.1 Weighing to 1.0 mg, add 16.0 g of dimethylformamide to a vial capable of being septum sealed. Add 2.0

TABLE 1 Typical Instrument Conditions

Detector	thermal conductivity
Column	4 ft (1.22 m) by 1/8 in. (3.2 mm) outside diameter packed with 80-100 mesh porous polymer packing
Temperature, °C	
Injection port	200
Detector block	250
Column	
Initial	100
Final	230 (for 8 min)
°C/min	8
Carrier gas	helium
Flow rate, mL/min	30
Specimen size, µL	1

g of 1,1,1-trichloroethane, 2.0 g of 1-propanol (see 10.5) and 2.0 g of dichloromethane. Seal the vial with a crimp-on or septum seal.

9.2 Determine the retention time of each component by injecting small amounts either separately or in known mixtures. The components should elute close to the typical retention times given in Table 1 and the chromatograms should closely approximate those shown in Fig. 1.

9.3 The area under each peak of the chromatogram is considered a quantitative measure of the corresponding compound. The relative area is proportional to concentration if the detector responds equally to all the sample components. The response to different components is generally significantly different for both flame ionization and thermal conductivity detectors and especially for flame ionization detectors. This difference in detector response may be corrected by use of relative response factors obtained by injecting and measuring the response of known blends. For precise and accurate determination of the halogenated

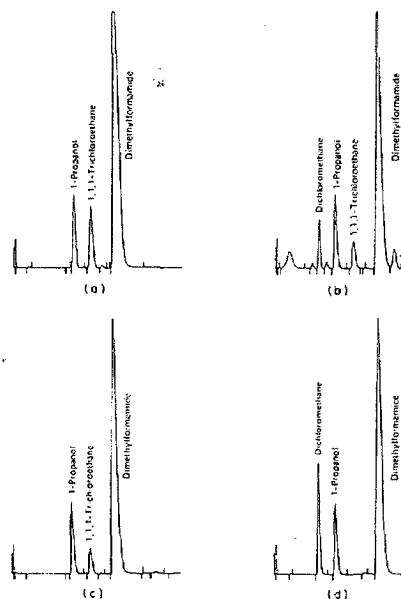


FIG. 1 Typical Chromatograms of Paints

American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

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hydrocarbons inject a 1 μ L specimen of the standard in accordance with the preparation in 9.1. Calculate the response factors relative to unity for the halogenated hydrocarbons.

10. Procedure

10.1 Keep all samples, blanks, and standards at a constant temperature. Observe the given order of ingredient addition to minimize loss of volatile ingredients. Shake paints, then sample from the middle of the container.

10.2 Weighing to 1.0 mg, add 16.0 g of dimethylformamide and 5.0 g of the paint to a vial capable of being septum sealed. Add 2.0 g of 1-propanol (see 10.5). Seal the vial with a crimp-on or septum seal.

10.3 Shake the vial. Then to facilitate settling, centrifuge using a low speed centrifuge at 1000 rpm for 5 min.

10.4 Inject a 1- μ L specimen of the supernatant from the prepared solution onto the chromatographic column, in accordance with the conditions established in 8.2. Record the peaks of all components.

10.5 If the composition of the paint is unknown, test for the presence of 1-propanol. Prepare a blank, omitting the 2.0 g of 1-propanol in 10.2, and inject a 1- μ L specimen. To this blank add 2.0 g of 1-propanol and inject a 1- μ L specimen. Then compare peak response to that from the test solution. If 1-propanol is present in the paint, substitute a different internal standard. Other possible internal standards include alcohols, esters, and hydrocarbons.

10.6 If the composition of the paint is unknown, establish whether peaks interfering with 1-propanol or the halogenated hydrocarbons are present by using both the column specified in 5.4 and a second column that yields different retention times.⁷

11. Calculation

11.1 Measure the area of all peaks (Note) and multiply each area by the appropriate attenuation factor to express the peak areas on a common basis.

NOTE—Peak areas may be determined by any method that meets the precision requirements of Section 12. Electronic integration is recommended for best results.

11.2 Calculate the percent halogenated hydrocarbon in the paint as follows:

$$RF_i = \frac{W_i}{A_i}$$

where:

RF_i = corrected peak response for i^{th} component, area units,

W_i = weight of i^{th} component in the standard solution, g, and

A_i = chromatographic peak area for the i^{th} component in the standard solution, area units,

then:

$$CH \text{ wt } \% = \frac{RF_{CH} \times A_{CH} \times IS_{amt}}{RF_{IS} \times A_{IS} \times W} \times 100$$

⁷ Packings from the Porapak series and the Chromosorb Century series may be satisfactory for this purpose. However, it is the responsibility of each analyst to check for interferences from paints or the internal standard chosen, or both, and to choose a column that gives symmetrical peaks.

where:

CH wt % = chlorinated hydrocarbon, weight %,

RF_{CH} = response factor for the chlorinated hydrocarbon in the standard solution, area units,

A_{CH} = area of the chlorinated hydrocarbon peak in the test solution, area units,

IS_{amt} = weight of internal standard added to the paint, g,

RF_{IS} = response factor for the internal standard in the standard solution, area units,

A_{IS} = area of the internal standard peak in the test solution, area units, and

W = specimen weight, g.

12. Precision and Bias⁸ (see also Practice E 180)

12.1 Precision:

12.1.1 *1,1,1-Trichloroethane*—On the basis of an inter-laboratory test of this test method in which one operator in each of eight laboratories tested three coatings containing from 31 to 65 % 1,1,1-trichloroethane (theoretical), the within-laboratory coefficient of variation was found to be 1.01 % relative at 20 degrees of freedom and the between-laboratories coefficient of variation was found to be 2.72 % relative at 17 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1.1 *Repeatability*—Two results, each the mean of duplicate runs, obtained by the same operator should be considered suspect if they differ by more than 3.0 % relative.

12.1.1.2 *Reproducibility*—Two results, each the mean of duplicate runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 8.1 % relative.

12.1.2 *Dichloromethane*—On the basis of an inter-laboratory test of this test method in which one operator in each of eight laboratories tested two coatings containing from 32 to 78 % dichloromethane (theoretical), the within-laboratory coefficient of variation was found to be 0.98 % relative at 14 degrees of freedom and the between-laboratories coefficient of variation was found to be 5.16 % relative at 12 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.2.1 *Repeatability*—Two results, each the mean of duplicate runs, obtained by same operator should be considered suspect if they differ by more than 3.0 % relative.

12.1.2.2 *Reproducibility*—Two results, each the mean of duplicate runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 17.92 % relative.

12.2 *Bias*—Determination of a bias statement for this test method is not practical at this time.

13. Keywords

13.1 chlorinated hydrocarbons in paints by gas chromatograph; dichloromethane, in paints; exempted solvent in paints; gas chromatograph, halohydrocarbon; 1,1,1-trichloroethane, in paints

⁸ Supporting data are available from ASTM Headquarters. Request RR:D01 - 1045.

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Standard Test Methods for Measuring Static Friction of Coating Surfaces¹

This standard is issued under the fixed designation D 4518; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of the resistance to sliding on coating surfaces by measuring the static friction.

1.2 Two test methods are described as follows:

	Sections
Method A—Inclined Plane Test.....	8 to 13
Method B—Horizontal Pull Test.....	14 to 19

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²

3. Terminology

3.1 *Definition:*

3.1.1 *static friction*—the force required to start the test sled moving, divided by the mass of the sled.

4. Summary of Test Methods

4.1 With the inclined plane test (Test Method A), a weighted sled is placed on a test specimen and the specimen is gradually inclined from the horizontal until the sled begins to slide. The tangent of this angle of inclination is reported as the static friction.

4.2 With the horizontal pull test (Test Method B), a weighted sled is placed on a horizontal test specimen and is pulled across the specimen. The static friction is reported as the force required to start the sled moving, divided by the mass of the sled.

5. Significance and Use

5.1 The friction characteristics of coating surfaces can be important to the use of the coatings. For example, low friction of exterior can coatings is beneficial to the flow of the cans on production lines. Also low friction of interior pipeline coatings is beneficial to the flow of materials

through pipes. On the other hand, low friction of floor coatings can be hazardous to foot traffic.

5.2 Under some conditions, measurement of the static friction can be used to evaluate the slip resistance of coatings under use conditions. However, results can be extremely dependent on the type of coating surface and the type of sliding unit used.

5.3 The tendency for footwear to slip may be influenced by foreign materials or lubricants on the shoe materials or on the walking surfaces. Also, these test methods do not incorporate all the directional forces involved in the walking process. Consequently, levels of slip resistance as determined by these test methods may not predict a person's resistance to slipping when walking on various surfaces.

5.4 The best precision and sensitivity are obtained when stainless steel is used as the facing of the sliding unit. In some tests where a leather facing is used, poor precision is obtained because of the inability to control the uniformity of its surface during the test. The use of a hard synthetic rubber facing provides somewhat better precision.

5.5 These test methods provide for static friction measurements when the sled facing and the coating surfaces are wet with water. Results from such tests must be treated with caution because frequently the static friction values obtained for wet, smooth coatings are higher than those obtained for the same surfaces dry. This is because, when stationary at the beginning of the test, the sled contact can produce a "suction cup" effect on a wet surface. Measurements performed on wet coatings with rough surfaces have been more satisfactory.

5.6 A test procedure is offered that eliminates the "suction cup" effect of wet surfaces. The wet sled is dropped onto the wet coating surface at the start of the test.

5.7 Results obtained by these test methods may be extremely sensitive to the age of the test coating because the blooming action of additives or plasticizers is often time-dependent. It may be meaningless to compare slip and frictional properties of test coatings applied at different times unless this effect is being studied.

5.8 The measurement of static friction may be influenced by the length of time that the sled rests on the test specimen before motion is initiated.

5.9 Static friction measurements have been useful in evaluating (1) the suitability of coatings for the exterior of cans, (2) the slipperiness of floor polishes, and (3) the slip resistance characteristics of footwear on floor tiles and floor coatings. Also, static friction measurements have been useful in determining the effect of coating additives (for example waxes, silicones) on the slipperiness of coating surfaces.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 4518 - 85. Last previous edition D 4518 - 90.

² *Annual Book of ASTM Standards*, Vol 06.01.

6. Test Specimens

6.1 Apply test coatings in accordance with Test Methods D 823 to substrates of at least 4 in. (100 mm) in width and length. The substrates may be of glass, steel, aluminum, or other appropriate material that remains smooth and plane after the test coating has been applied and cured at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity for 7 days, unless otherwise specified. Prepare at least two test panels for each coating.

6.2 Take care during application of the test coating to minimize entrainment of dust and particulate matter in the surface of the coating. Extreme care is required when handling the panels, even after sufficient cure. The surface should be kept free of all dust, lint, fingerprints, or any foreign matter that may change the characteristics of the surface.

7. Conditions for Testing

7.1 Test the coated test specimens under one or more of the following conditions as agreed upon by the purchaser and the seller:

7.1.1 Both sled facing and coating surfaces dry at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$).

7.1.2 Both the sled face and the coating surface wet with water containing a slight amount of wetting agent at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$). Sea water containing a slight amount of wetting agent may be used when it is appropriate. A wetting solution consisting of 1 mL of surfactant³ added to 200 mL of water has been found to be satisfactory.

7.1.3 One or both of the above at $35.5 \pm 3.5^\circ\text{F}$ ($2 \pm 2^\circ\text{C}$).

TEST METHOD A—INCLINED PLANE TEST

8. Apparatus

8.1 *Inclined Plane*, having a smooth, incompressible surface, at least 1 in. (25 mm) wider than the sliding unit and of sufficient length to allow the test sled to move by gravity at least 0.5 in. (12 mm), provided with clamps for the test specimen, and an inclinometer to indicate the angular displacement of the plane to within 0.5° .

NOTE 1—A suitable apparatus is shown in Fig. 1. It may be assembled from items obtained from laboratory instrument supply houses.

8.2 *Test Sleds*—Alternative sleds that may be used are:

8.2.1 *Stainless Rounded Edge Steel Block*, with a highly polished plane lower surface 3 by 3 in. (75 by 75 mm) in area and a mass of 1.8 lb (0.82 kg) to provide a pressure of 0.2 psi (1.4 kPa) when horizontal.

8.2.2 *Stainless Steel Block*, with a plane lower surface of 3 by 3 in. (75 by 75 mm) and a mass of 1.8 lb (0.82 kg). Adhered to the lower surface is a $\frac{1}{4}$ -in. (6-mm) thick vulcanized neoprene rubber having a Shore "A" hardness of 65 ± 5 . A screw eye or other means to attach the sled to the force-measuring device is provided.

8.3 *Inclinometer*—Means to smoothly increase the inclination of the plane from the horizontal through an arc of at

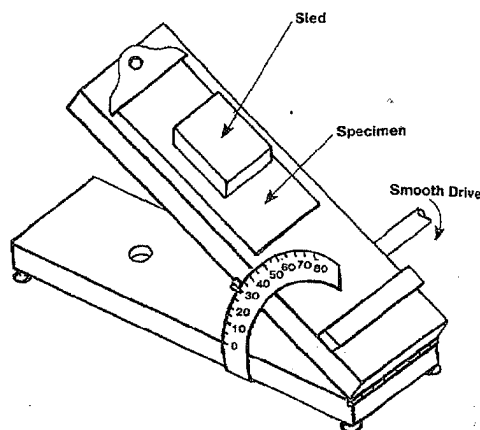


FIG. 1 Schematic of Inclined Plane Apparatus

least 45° at a rate of $1.5 \pm 0.5^\circ/\text{s}$.

NOTE 2—Procedures found suitable for smoothly increasing the inclination of the plane are (1) pulling the top of the plane upward with a motor driven dip coater and (2) pushing the top of the plane upward with a laboratory jack equipped with a hand crank.

9. Preparation of Apparatus

9.1 Preparation of Sled Facing:

9.1.1 If a synthetic rubber facing is used on the test sled, it must be preconditioned by light sanding before each determination.

9.1.2 Place a sheet of 400A wet or dry silicon carbide abrasive paper on a flat surface. Sand the synthetic rubber facing by rubbing it gently back and forth over the paper four times. Repeat at 90° to the first direction. Wipe the surface of the synthetic rubber facing with a clean, dry cloth to remove dust or loose material from the surface.

10. Procedure

10.1 Level the plane so it is horizontal when the inclinometer reads zero.

10.2 Clamp the test specimen to the plane. Recheck the levelness of the panel in two directions (along the length of the panel and across the panel at 90° to the first measurement).

10.3 Dry Surfaces:

10.3.1 Center the sled on the test coating. Immediately commence inclining at a rate of $1.5 \pm 0.5^\circ/\text{s}$. When the sled starts to move, stop the inclinometer immediately and read the angle of displacement at the moment the sled starts to move.

10.3.2 Return the plane to a level position and make two additional tests on the test specimen as described in 8.3.

10.4 For Wet Surfaces:

10.4.1 Check levelness of plane and test panel according to procedures given in 10.1 and 10.2.

10.4.2 Wet the test coating surface and the sled facing with the prescribed solution. Apply the solution with a soft brush. Make certain that all surfaces are completely wet.

10.4.3 Set the plane of the angle of displacement deter-

³ A surfactant such as Aerosol OT manufactured by American Cyanamid Co., Chemical Group, One Cyanamid Plaza, Wayne, NJ 07470, has been found suitable for this purpose.

TABLE 1 Precision Values Obtained in 1987 Round-Robin Test of Test Method D 4518

Inclined Plane Test				Dry Surfaces		
Type of Sled Surface	Standard Deviation		Coefficient of Variation	Degree of Freedom	Maximum Allowable Difference	
	Angle of Inclination	Static Friction			Angle of Inclination	Static Friction
Polished Steel	3.6	0.08	23 %	14	10.9	0.24
Abraded Steel	3.7	0.08	10 %	13	11.3	0.24

Interlaboratory						
Type of Sled Surface	Standard Deviation		Coefficient of Variation	Degree of Freedom	Maximum Allowable Difference	
	Angle of Inclination	Static Friction			Angle of Inclination	Static Friction
Polished Steel	4.2	0.09	28 %	32	12.1	0.26
Abraded Steel	4.0	0.09	20 %	32	11.5	0.26
Neoprene	4.2	0.21	17 %	19	12.4	0.62

measured for the dry coating surface in 10.3.1.

10.4.4 From a height of 1/2 in. (12 mm), drop the sled onto the coating surface. Determine if the sled begins to slide.

10.4.5 Change the angle of the slide, and after rewetting coating and sled surfaces, repeat dropping the sled. Continue in this manner until the plane angle at which the sled starts to slide is determined.

10.4.6 Return the plane to the initial position of 10.4.3 and repeat 10.4.4 and 10.4.5.

11. Calculation

11.1 Calculate the mean of the three measurements on the test specimen. Determine the trigonometric tangent of the mean angle, which is the static friction value.

12. Report

12.1 Report the following information for the coated panels tested:

12.1.1 Temperature and humidity during curing and at the time of testing,

12.1.2 Type of facing used on the test sled,

12.1.3 Whether facing and coating surface were wet or dry,

12.1.4 Inclination angle determined for each measurement,

12.1.5 Static friction value for each measurement, and

12.1.6 Mean static friction value for the replicate panels.

13. Precision

13.1 An interlaboratory study of Test Method A (Inclined Plane Test) in which one operator in each of four laboratories, using sled surfaces of neoprene, polished steel and mildly abraded steel, tested the dry surfaces of eight coated panels with a wide range of slip resistance. The intralaboratory and interlaboratory standard deviations were found to be as shown in Table 1. Based on these standard deviations, the following criteria should be used for judging, at the 95 % confidence level, the acceptability of results:

13.1.1 *Repeatability*—Two results obtained by the same operator should be suspect if they differ by more than the maximum allowable difference given in Table 1.

13.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than the maximum allowable difference given in Table 1.

13.2 Comparison of inclined plane results are shown in Table 2.

TABLE 2 Comparison of Inclined Plane Results Obtained with Stationary and Dropped Sleds on Panel Surfaces

Panel	Sled	Surface	Angle of Slide, °		Static Friction	
			Sled Stationary	Sled Dropped	Sled Stationary	Sled Dropped
A	Steel	Dry	12	12	0.21	0.21
	Neoprene		27	31	0.50	0.61
	Steel	Wet	...	14	...	0.25
	Neoprene		...	32	...	0.61
B	Steel	Dry	20	25	0.35	0.46
	Neoprene		47	34	1.07	0.68
	Steel	Wet	...	19	...	0.34
	Neoprene		...	26	...	0.49
C	Steel	Dry	17	26	0.31	0.49
	Neoprene		36	41	0.73	0.86
	Steel	Wet	...	19	...	0.23
	Neoprene		...	26	...	0.56
D	Steel	Dry	20	25	0.35	0.46
	Neoprene		47	34	1.07	0.68
	Steel	Wet	...	15	...	0.27
	Neoprene		...	29	...	0.56
G	Steel	Dry	20	20	0.35	0.35
	Neoprene		38	32	0.80	0.61
	Steel	Wet	...	19	...	0.34
	Neoprene		...	26	...	0.49
H	Steel	Dry	25	22	0.46	0.39
	Neoprene		14	36	0.96	0.74
	Steel	Wet	...	13	...	0.23
	Neoprene		...	18	...	0.32

13.3 Bias cannot be determined since there is no standard for slipperiness or slip resistance.

TEST METHOD B—HORIZONTAL PULL TEST

14. Apparatus⁴

14.1 *Test Base*, a horizontal plane surface of a smooth, incompressible material (metal, wood, plate glass, or plastic) having a width at least 1 in. (25 mm) wider than the test sled (14.2) with means of leveling it in two directions. This base serves as a support for the rest of the mechanism and the test specimen. When placed on a solid support, it must be free of vibration.

14.2 *Test Sleds*—Alternative sleds that may be used are:

14.2.1 *Stainless Steel Block*, with a highly polished lower plane surface of 3 by 3 in. (75 by 75 mm) and a mass of 1.8 lb (0.82 kg) to provide a pressure of 0.2 psi (1.4 MPa) on the horizontal surface. A screw eye or other means to attach the sled to the force-measuring device is provided.

14.2.2 *Steel Block*, with a smooth lower surface of 3 by 3 in. (75 by 75 mm) in area and a mass of 1.8 lb (0.82 kg). Adhered to the lower surface is a 1/4-in. (6-mm) thick vulcanized neoprene rubber having a Shore "A" hardness of 65 ± 5 . A screw eye or other means to attach the sled to the force-measuring device is provided.

14.2.3 *Steel Block*, having a smooth lower surface 4 by 5 in. (100 by 125 mm) in area and a thickness of 1 to 1.5 in. (25 to 40 mm). Adhered to the lower surface is vulcanized neoprene rubber having a Shore "A" hardness of 65 ± 5 and a thickness of 1/8 in. (3 mm). The total mass of the sled with the rubber facing shall be 6.0 ± 0.5 lb (2.7 ± 0.2 kg).

14.3 *Mechanical Power Unit*—Means for moving the test sled horizontally over the test specimen or the test specimen under a fixed test sled under the following conditions:

14.3.1 For the sleds described in 14.2.1 and 14.2.2, a speed of 6.0 ± 1.0 in./min (150 ± 25 mm/min) shall be provided and a sled (or panel) travel of 4 in. (100 mm).

14.3.2 For the sled described in 14.2.3, a speed of 12 ± 1 in. (300 ± 25 mm)/min shall be provided and the sled (or panel) allowed to travel 4 in. (100 mm).

14.3.3 *Force Measuring Device*—Means for measuring the force to 0.01 lb (0.04 N) required to move or restrain the test sled.

NOTE 3—A force gage⁵ that can be preset to display the highest force encountered is desirable.

NOTE 4—Figure 2 illustrates a suitable apparatus. There are various commercial instruments available but some require modification to meet the requirements of these test methods.

15. Preparation of Apparatus

15.1 Preparation of Sled Facing:

15.1.1 If a synthetic rubber facing is used on the test sled, it must be preconditioned by light sanding before each determination.

15.1.2 Place a sheet of 400A wet or dry silicon carbide abrasive paper on a plane surface. Sand the synthetic rubber facing by rubbing it gently back and forth over the paper four

⁴ A facing of heel material such as Neolite, with a Shore "A" hardness of 95 ± 5 may be used if appropriate.

⁵ Chatillon dynamometer gages, available from Chatillon, Inc., 83-30 Kew Garden Rd., Kew Garden, NY 11415, have been found suitable for this purpose.

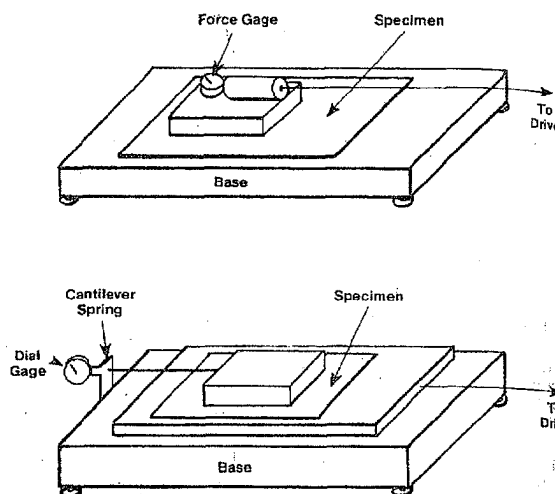


FIG. 2 Schematics for Two Horizontal Plane Instruments.

times. Repeat at 90° to the first direction. Wipe the surface of the synthetic rubber facing with a clean, dry cloth to remove dust or loose material from the surface.

16. Procedure

16.1 Select the test sled and traverse the speed appropriate for the type of test desired:

16.1.1 The test sled described in 14.2.2 with a traverse speed of 6 in. (150 mm)/min is recommended if the best precision of measurement on a smooth coating is desired.

16.1.2 The test sled described in 14.2.2 with a traverse speed of 6 in. (150 mm)/min is recommended if it is desired to measure the resistance of a smooth coating to the slipping of a rubber shoe heel or sole.

16.1.3 The test sled described in 14.2.3 with a traverse speed of 12 in. (300 mm)/min is recommended if it is desired to measure the resistance of a rough coating to the slipping of a rubber shoe heel or sole.

16.2 Clamp the test specimen to the base. Position the test sled and connect it to the power unit or force gage. Fasten the inelastic linkage (cable or chain) to the test sled and connect to the power unit or the force gage.

16.3 As soon as possible after positioning the sled, start the power unit, taking care to maintain tautness in the linkage as the power unit takes up the load.

16.4 Record the force required to just begin motion of the test slide; that is, the maximum force value recorded.

16.5 For the evaluation of smooth coatings, make two additional determinations on the test specimen as described in 16.1 through 16.4. For the evaluation of rough coatings, make four additional determinations on the test specimen.

17. Calculation

17.1 Calculate the mean of the force value obtained for the replicate determinations.

17.2 Divide the mean force by the mass of the test sled. This is the static friction value.

18. Report

18.1 Report the following information for the coated panels tested:

- 18.1.1 Temperature and humidity during curing and testing,
- 18.1.2 Whether facing and coating surfaces were dry or wet,
- 18.1.3 Mass of test sled used,
- 18.1.4 Type of facing used on the test sled,
- 18.1.5 Traverse speed of sled used,
- 18.1.6 Static friction value for each coated panel, and

18.1.7 Mean static friction value for the replicate panels.

19. Precision and Bias

19.1 An interlaboratory test will be conducted to determine the precision and bias of this test method. Tests conducted by several laboratories have shown that this measuring technique has sufficient sensitivity and precision to differentiate coatings exhibiting significantly different sliding friction characteristics.

20. Keywords

20.1 coating surface slipperiness; horizontal pull slipmeter test; inclined plane; static friction

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Guide for Establishing Procedures to Qualify and Certify Inspection Personnel for Coating Work in Nuclear Facilities¹

This standard is issued under the fixed designation D 4537; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide delineates the requirements for development of procedures for the qualification of personnel who perform inspection of coating work. These activities are accomplished to verify conformance to specified requirements for nuclear facility coatings work whose satisfactory performance is required in order not to compromise systems used to mitigate the consequences of postulated accidents.

1.2 This guide provides a uniform interpretation of the requirements in ANSI/ASME N45.2.6-1978 for the inspection of coating work in nuclear facilities.

1.3 This guide meets the intent of ANSI/ASME NQA-1.

1.4 It is the intent of this guide to provide a recommended basis for qualification, not to mandate a singular basis for all qualifications. Variations or simplifications of the qualifications described in this guide are appropriate for special coating work outside of safety-related areas.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ANSI/ASME Standards:

ANSI/ASME N45.2.6 Qualifications of Inspection, Examination, and Testing Personnel for Nuclear Power Plants²

ANSI/ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facilities.²

3. Terminology

3.1 Description of Terms Specific to This Standard

3.1.1 *certification*—written documentation of qualification.

3.1.2 *coating work inspection*—a phase of quality control which, by means of examination, observation, or measurement, determines the conformance of coating work to predetermined quality requirements.

3.1.3 *qualifications*—skills, training, and experience required for personnel to perform properly the duties and

execute the responsibilities of the appropriate certification level.

3.1.4 *training*—the program developed to ensure that personnel receive the knowledge and skills necessary for qualification.

4. Significance and Use

4.1 The requirements of this guide apply to personnel who perform inspections of coating work during (1) fabrication, (2) receipt of items at the construction site, (3) construction, (4) pre-operational and startup testing, and (5) operational phases of nuclear facilities.

4.2 It is the responsibility of each organization participating in the project to ensure that only those personnel within their respective organizations who meet the requirements of this guide are permitted to perform inspection activities covered by this guide.

4.3 The organization(s) responsible for establishing the applicable requirements for activities covered by this guide shall be identified, and the scope of their responsibility shall be documented. Delegation of this responsibility to other qualified organizations is permitted and shall be documented.

4.4 It is the responsibility of the organization performing these activities to specify the detailed methods and procedures for meeting the requirements of this guide, unless they are otherwise specified in the contract documents.

5. General Requirements for Inspection Personnel

5.1 Provisions shall be made for the indoctrination of personnel as to the technical objectives of the project, the codes and standards that are to be used, and the quality assurance elements that are to be employed.

5.2 The need for formal training programs shall be determined, and such training activities shall be conducted as required to qualify personnel who perform inspections. On-the-job participation shall also be included in the program, with emphasis on first-hand experience gained through actual performance of inspections.

5.3 A candidate's qualifications for certification shall be initially determined by a suitable evaluation of the candidate's education, experience, training, examination results, and capability demonstration.

5.4 The job performance of coating work inspection personnel shall be reevaluated at periodic intervals not to exceed three years. Reevaluation shall be by evidence of continued satisfactory performance or redetermination of capability in accordance with 5.3. If, during this evaluation or at any other time, it is determined by the responsible organization that the capabilities of an individual are not in

¹ This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

Current edition approved Aug. 15, 1991. Published October 1991. Originally published as D 4537 - 86. Last previous edition D 4537 - 86.

² Available from American National Standards Institute, 11 W. 42nd Street, 13th Floor, New York, NY 10036.

VISION TEST RECORD

Expires _____

NAME: _____
(Last) (First) (Middle Initial)

JOB NUMBER: _____ JOB NAME: _____

NEAR DISTANCE VISUAL ACUITY

1. Jaeger J-1 letters		
Uncorrected, normal	YES	NO
Corrected, normal	YES	NO
-OR-		
2. Alternative Method: _____		

Uncorrected, normal	YES	NO
Corrected, normal	YES	NO

COLOR VISION

1. Ishihara's test chart	YES	NO
2. Alternative Method: _____		

Color vision test findings: _____

The above-named individual has Passed/Failed the vision test as specified in Section 7 of this Guide.

THIS CERTIFICATION IS VALID FOR ONE YEAR FROM THE DATE OF EXAMINATION.

EXAMINER: _____
Type or print name followed by signature

TITLE: _____ DATE: _____

FIG. 1 Sample Vision Test Record

accordance with the qualifications specified for the job, that person shall be removed from that activity until the required capability has been demonstrated.

5.5 Any person who has not been actively engaged in the performance or supervision of coating work inspection for a period of one year shall be reevaluated in accordance with 5.3.

6. Functional Qualifications of Inspectors

6.1 All physical inspection activities can be performed by certified Level I, Level II, or Level III inspectors.

6.2 *Level I Coatings Inspector*, shall be capable of the following:

6.2.1 Implementing and recording all inspections required by the applicable procedures.

6.2.2 Verifying instrument calibration.

6.2.3 Performing hold point inspections in accordance with the applicable procedures.

6.3 *Level II Coatings Inspector*, shall be capable of the following:

6.3.1 Performing all of the duties and responsibilities of a Level I coatings inspector.

6.3.2 Planning and supervising inspections, initiating and reviewing inspection procedures, and evaluating the adequacy of activities.

6.3.3 Reviewing, organizing, and approving results of inspections.

6.3.4 Monitoring the performance of and supervising the work of Level I coatings inspectors.

6.3.5 Training and verifying the qualifications of Level I coatings inspectors for certification.

6.3.6 Initiating changes to quality procedures.

6.3.7 Implementing the Quality Assurance Program if assigned that authority by company policy or the Quality Assurance Program.

6.4 *Level III Coatings Inspector*, shall be capable of the following:

6.4.1 Carrying out all of the duties and responsibilities of a Level II coatings inspector.

6.4.2 Certifying Level I, Level II, and other Level III coatings inspectors.

EDUCATION:

EXPERIENCE:

PROFESSIONAL DATA:

I certify that the above information is accurate.

SIGNED: _____ DATE: _____

Candidate

FIG. 2 Sample Education and Experience Record

6.4.3 Responsible for evaluating the adequacy of programs used to train coatings inspectors.

6.4.4 Responsible for authorizing Level II coatings inspectors to carry out training and examination duties:

6.4.5 Responsible for approving all safety-related inspection procedures.

7. Physical Qualifications of Inspectors

7.1 Each inspector shall be examined annually to ensure natural or corrected near-distance visual acuity in at least one eye. The individual shall read the J-1 letters on a Standard Jaeger Test Chart, or equivalent, at a distance of not less than 12 in. with one or both eyes, uncorrected or corrected.

7.2 Each candidate shall be examined for color preception using the Ishihara Test or the Farnsworth D-15 Test when being certified or recertified. If a candidate does not pass the Red/Green Sensitive Ishihara Test, the candidate may take the Farnsworth D-15 Test.

7.3 If a candidate does not pass the Farnsworth D-15 Test the candidate may be evaluated by a licensed medical practitioner to provide the necessary data to determine the candidate's color perception. Individuals certified after an

evaluation by a licensed medical practitioner may only be certified to perform inspection work that is within the candidate's color perception capability.

7.4 The examinations required by 7.1 and 7.2 shall be administered by a licensed medical practitioner or a person familiar with the tests involved. The results of vision tests shall be documented on a Vision Test Record (Fig. 1 or equivalent form).

7.5 The responsible organization shall identify any other physical qualifications required to perform the assigned inspection duties. Inspectors requiring the identified physical qualifications shall have them confirmed by examinations at intervals not to exceed one year.

8. Education, Training, and Experience Qualifications

8.1 Candidates for certification as coatings inspectors shall have sufficient education, experience, and training to ensure an understanding of the principles and procedures in those areas of inspection, examination, and testing activities for which they are being considered for certification.

8.2 *Level I Coatings Inspectors*, shall, as a minimum, meet one or more of the following requirements:

ORGANIZATIONAL TRAINING RECORD

NAME: _____
(Last) (First) (Middle Initial)

TRAINING COURSES, SEMINARS, LECTURES, ETC.

Given by and Date	Description of Training	Test Score	Pass/Fail	Initials

FIG. 3 Sample Organization Training Record

8.2.1 High school graduation plus six months of related experience in equivalent inspection activities.

3.2.2 Completion of college level work leading to an associate Degree or higher, plus three months of related experience in equivalent inspection activities.

8.3 *Level II Coatings Inspectors*, shall, as a minimum, meet one or more of the following requirements:

8.3.1 High school graduation plus one year of satisfactory performance as a Level I coating inspector in the corresponding inspection activity.

8.3.2 High school graduation plus three years of related experience in equivalent inspection activities.

8.3.3 Completion of college level work leading to an associate Degree plus one year of related experience in equivalent inspection activities.

3.3.4 Four-year college graduation plus six months of related experience in equivalent inspection activities.

8.4 *Level III Coatings Inspectors*, shall meet one or more of the following requirements:

8.4.1 High school graduation plus six years of satisfactory performance as a Level II coatings inspector in the corresponding inspection activity.

8.4.2 High school graduation plus ten years of related experience in equivalent inspection activities; or high school graduation plus eight years experience in equivalent inspection activities, with at least two years as a Level II coatings inspector. The candidate shall have at least two years associated with nuclear facilities or sufficient training to be knowledgeable of the quality assurance requirements for nuclear coating work.

8.4.3 Completion of college level work leading to an Associate Degree and seven years of related experience in equivalent inspection activities. The candidate shall have at least two years of this experience associated with nuclear

RECORD OF CERTIFICATION

Date Certified: _____
Expiration: _____

TO WHOM IT MAY CONCERN:

SUBJECT: Coatings Inspector Certification, Level _____

REFERENCE: ASTM Standard Guide _____ Rev. _____

_____ has complied with, and successfully passed, all of the applicable requirements of the referenced Standard Guide.

EXAMINATION SCORES

GENERAL PORTION _____
SPECIFIC PORTION _____
PRACTICAL PORTION _____

I certify that the above statements are correct and recommend certification as a Level _____ Coatings Inspector.

RECOMMENDED BY

NAME: _____
Type or print followed by signature
TITLE: _____

The above statement and recommendation have been reviewed for conformance to the requirements of the referenced Standard, and are correct. This individual is certified as Level _____ coatings inspector in accordance with the referenced Standard Guide.

CERTIFIED BY

Type or print name followed by signature
(must comply with 11.3 of the Standard Guide)

FIG. 4 Sample Record of Certification

facilities or sufficient training to be knowledgeable of the quality assurance requirements for nuclear coating work.

8.4.4 Four-year college graduation plus five years of related experience in equivalent inspection activities. The candidate shall have at least two years of this experience associated with nuclear facilities or sufficient training to be knowledgeable of the quality assurance requirements for nuclear coating work.

8.5 Compliance with the requirements of 8.1, 8.2, 8.3, and 8.4 shall be documented on an Education and Experience Record (Fig. 2 or equivalent form).

8.6 Training leading to certification or recertification as a qualified coatings inspector shall be documented on an Organizational Training Record (Fig. 3 or equivalent form).

9. Examination

9.1 Each candidate for coatings inspector shall be given an

examination covering the general, specific, and practical aspects of coatings inspection. The general and specific portions of the examination may be written, in the form of a personal interview, or a combination of both. The examination results shall be documented in accordance with 11.1.

9.1.1 The general portion of the examination shall cover the basic principles of quality assurance and coating work inspection.

9.1.2 The specific portion of the examination shall cover specific coating work requirements and inspection procedures.

9.1.3 The practical portion of the examination shall cover the use of coating work inspection equipment and inspection procedures.

9.1.4 All parts of the examination for a Level I or Level II coatings inspector shall be administered by a Level III

coatings inspector or a duly authorized Level II coatings inspector.

9.1.5 Examinations for a Level III coatings inspector shall be administered by a certified Level III coatings inspector or by the responsible organization's management.

10. Performance

10.1 Personnel assigned the responsibility and authority to perform functions covered by this guide shall have, as a minimum, the appropriate level of capability given in Section 6. When a single inspection requires implementation by a team or group, personnel not meeting the requirements of this Guide may be used in data-taking assignments or in plant or equipment operation provided they are supervised or overseen by a qualified individual participating in the inspection.

10.2 A certified coatings inspector shall submit annually a summary of coatings related inspection/testing activities performed between certification anniversary dates in order to maintain the validity of the certification. This summary for a Level I or Level II coatings inspector shall be reviewed by a certified Level III coatings inspector or his designee, signed and placed in the coatings inspector's certification file. The summary review for a Level III coatings inspector shall be conducted by another certified Level III coatings inspector or the responsible organization's management, signed and placed in the inspector's certification file.

10.3 Any certified coatings inspector not performing coatings related inspection/testing work between certification anniversary dates shall be recertified in accordance with 11.2, after it is reconfirmed that the inspector's capabilities meet the requirements of this guide.

11. Certification of Inspectors

11.1 Level I and Level II coatings inspection certifications are valid for a period not to exceed three years. Level III coatings inspector certifications are valid for a period not to exceed five years. Coatings inspectors are certified after review of the candidate's qualifications for compliance with this guide. The examiner shall document the interview of the candidate and the review of the candidate's capabilities, education, experience, vision records, training records, and applicable examination records. Certifications are documented on a Record of Certification (Fig. 4 or equivalent form).

11.2 Coatings inspectors shall be recertified as required by 11.1 after evaluation of their work record. Individual work records shall show evidence of continuing satisfactory performance.

11.3 Level I and Level II coatings inspectors are certified or recertified by a certified Level III coatings inspector. Level III coatings inspectors are certified or recertified by the responsible organization's management or by a certified Level III coatings inspector.

11.4 Certification by a given employer shall be considered revoked when employment is terminated.

12. Records

12.1 A personnel qualification records file shall be established and maintained by the employer. Collection, storage, and control of records required by this guide shall be in accordance with the requirements of the responsible organization and appropriate specifications.

13. Keywords

13.1 certified coatings inspector; coatings inspection; coatings inspector; inspector certification; Level II Coatings Inspector; Level III Coatings Inspector; nuclear coatings inspector; qualified coatings inspector

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Standard Terminology Relating to Protective Coating and Lining Work for Power Generation Facilities¹

This standard is issued under the fixed designation D 4538; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

- alligatoring**—*in protective coatings*, surface cracking of coating film having an appearance similar to alligator hide.
- certification**—*in protective coatings*, the written documentation of the qualification of personnel or material.
- checking**—*in protective coatings*, the formation of slight breaks in a coating film that do not penetrate to the underlying surface.
- coating applicator**—*in protective coatings*, an organization or individual responsible for applying a protective or decorative coating.
- coating system**—*in protective coatings*, a protective film consisting of one or more coats, applied in a predetermined order by prescribed methods.
- coating work**—*in protective coatings*, an all-inclusive term to define all operations required to accomplish a complete coating job; construed to include materials, equipment, labor, preparation of surfaces, control of ambient conditions, application of coating systems, and inspection.
- cobwebbing**—*in protective coatings*, the formation of fine filaments (cobwebs) or partly dried coating, during spray application.
- cracking**—*in protective coatings*, the formation of breaks in a coating film that extend through to the underlying surface.
- cratering**—*in protective coatings*, the formation of round depressions in a coating film that do not expose the previous coat or the substrate.
- crawling**—*in protective coatings*, a defect in which a wet coating film recedes from a small area to form an uneven surface shortly after application.
- crazing**—*in protective coatings*, the formation of a criss-cross pattern of minute cracks on the surface of a coating film.
- delamination**—*in protective coatings*, a separation of one coat from another coat within a coating system; or from the substrate.
- deviation**—*in protective coatings*, a departure of a characteristic from established procedures or from specified requirements.
- drips**—*in protective coatings*, the small drops of coating that collect on the edge of the coated work.
- dry spray**—*in protective coatings*, a rough, powdery, non-coherent film produced when an atomized coating partially dries before reaching the surface.
- flaking**—*in protective coatings*, the detachment of small pieces of the coating film.
- foreign matter**—*in protective coatings*, insoluble foreign particles such as sand, lint, dust, and dirt that get mixed with the coating material before, during, or after application; causing the formation of raised specks in the dried film.
- hairline crack**—*in protective coatings*, a very fine crack (having a hairlike appearance) that is visible on the surface of a dried coating film.
- heavy-centered spray pattern**—*in protective coatings*, an uneven spray pattern having more coating in the center, and less at the edges.
- intercoat contamination**—*in protective coatings*, the presence of foreign matter between successive coats.
- mudcracking**—*in protective coatings*, a particular pattern of cracking in a coating with the appearance of a dried mud puddle (see cracking and checking).
- orange peel**—*in protective coatings*, the dimpled appearance of a dried coating film resembling the surface of an orange.
- pinhole**—*in protective coatings*, minute holes through a coat or coats that expose an underlying coat or the substrate.
- pinholes**—small pore-like flaws in a coating that extend entirely through the applied film and have the general appearance of pin pricks when viewed by reflected light (see Definitions D 16²).
- qualification**—*in protective coatings*: The characteristics or abilities gained through training or experience, or both, that enable an individual to perform a required function.
- wrinkling**—*in protective coatings*, the formation of a surface appearance in a coating film resembling the skin of a prune.

¹ These definitions are under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and are the direct responsibility of Subcommittee D 33.92 on Definitions.

Current edition approved Nov. 30, 1990. Published January 1991. Originally published as D 4538 - 86a. Last previous edition D 4538 - 90.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

² Definitions D 16, of Terms Relating to Paint, Varnish, Lacquer, and Related Products (Committee D - 1 on Paint and Related Coatings and Materials).



Standard Guide for Testing Interior Latex Semigloss and Gloss Paints¹

This standard is issued under the fixed designation D 4540; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

Scope

1.1 This guide covers the selection and use of procedures for testing latex semigloss or gloss paints intended for use on interior walls or trim. The test methods included are listed in Tables 1 and 2.

1.2 The latex semigloss and gloss paints covered by this guide are intended for application by brushing, rolling, spraying, or other means, on plaster, masonry surfaces, wood, wallboard, previously painted surfaces and other interior architectural surfaces.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts³
- D 522 Test Methods for Mandrel Bend Test of Attached Organic Coatings³
- D 523 Test Method for Specular Gloss³
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer³
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³
- D 869 Test Method for Evaluating Degree of Settling of Paint³
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems³
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products³
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature³
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³

- D 1736 Test Method for Efflorescence of Interior Wall Paints³
- D 1849 Test Method for Package Stability of Paint³
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer³
- D 2243 Test Method for Freeze-Thaw Resistance of Water-Borne Paints³
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates³
- D 2369 Test Method for Volatile Content of Coatings³
- D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints³
- D 2574 Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms³
- D 2805 Test Method for Hiding Power of Paints by Reflectometry³
- D 2831 Test Method for Evaluating the Ability of a Latex Paint to Resist Efflorescence from the Substrate³
- D 3258 Test Method for Porosity of Paint Films³
- D 3450 Test Method for Washability Properties of Interior Architectural Coatings³
- D 3793 Test Method for Low-Temperature Coalescence of Latex Paint Films³
- D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings³
- D 3928 Test Method for Evaluation of Gloss or Sheen Uniformity³
- D 3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings³
- D 4062 Test Method for Leveling of Paints by Draw-Down Method³
- D 4213 Test Method for Wet Abrasion Resistance of Interior Paints³
- D 4287 Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer³
- D 4400 Test Methods for Sag Resistance of Paints Using a Multinotch Applicator³
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴
- E 105 Practice for Probability Sampling of Materials⁵
- 2.2 *U.S. Federal Test Methods Standard No. 141.*⁶
 - 2112 Application by Roller
 - 2131 Application of Sprayed Films
 - 2141 Application of Brushed Films
 - 3011 Condition in Container

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct 15, 1991. Published December 1991. Originally published as D 4540 - 85. Last previous edition D 4540 - 90.

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 15.05.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

4061 Drying Time
4321 Brushing Properties
4541 Working Properties and Appearance of Dried Film

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this guide, see Terminology D 16.

4. Conditions Affecting Latex Semigloss and Gloss Paints

4.1 *Substrate Type*—The substrate to be painted can affect not only the application properties and the physical properties of a latex paint, such as gloss and uniformity, but may also be a factor in determining the type of latex paint to be used.

4.2 *Substrate Condition*—Dirty, alkaline, or water-soluble surfaces may affect the practical requirements and performance of these paints.

4.3 *Application Properties*—The application properties of paints are affected by temperature, humidity, and the texture and porosity of the surface to be painted. Application is also affected by the type and quality of equipment used.

5. Selection of Tests

5.1 Many conditions affect interior latex semigloss and gloss paints, so that different types of latex paints have been developed specifically to meet the requirements of these various conditions. Although the recommended test methods in Tables 1 and 2 cover most of the properties of such paints, all of the tests may not be required for each paint. If a paint is to be used only in a warm climate for instance, then freeze-thaw tests or low temperature coalescence tests need not be considered.

5.2 The purchaser should first determine which properties a latex paint must have and then select only the test methods that will measure or evaluate those properties. After selecting the desired tests, the purchaser should determine which of these properties are the most important and establish the appropriate requirements or specifications for obtaining the desired properties. Since paint properties frequently tend to oppose each other, some properties may need to be attenuated if others are to be accentuated. This balance of properties must be considered when selecting the tests and establishing their requirements. A normal range of values is indicated in many of the tests methods.

5.3 This guide does not indicate relative importance of the various tests nor does it recommend specific values for each test, since the properties that are important to one purchaser may not be important to another.

6. Sampling

6.1 Prior to sampling, the condition of the container should be established since damage to it may cause evaporation or skinning of the pigment or other undesirable effects in the coating. Determine the condition of the coating in accordance with 7.1 and 7.2.

6.2 Sample in accordance with Practice D 3925. Determine the weight per gallon in accordance with Test Method D 1475. Repeat this procedure until successive readings agree within 0.2 lb (90 g) or as agreed upon between purchaser and seller. Samples for testing may then be taken.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
Liquid Paint Properties:			
Condition in container	7.1		3011
Coarse particles and foreign matter	7.2	D 185	
Density (weight per gallon)	7.3	D 1475	
Fineness of dispersion	7.4	D 1210	
Odor	7.5		
Color compatibility	7.6		
Package Stability:	7.7	D 1849	
Accelerated heat-aged stability	7.7.1		
Freeze-thaw stability	7.7.2	D 2243	
Microorganism resistance	7.7.3	D 2574	
pH stability	7.7.4	E 70	
Sampling	6	D 3925	
Settling	7.7.5	D 869	
Paint Application and Film Formation:			
Application properties	8.1		4541
Application by brush	8.1.1		2141
Application by roller	8.1.2		2112
Application by spray	8.1.3		2131
Drying time	8.2	D 1640	
Low-temperature coalescence of paints	8.3	D 3793	
Producing uniform thickness of films	8.4	D 823	
Touch-up uniformity	8.5	D 3928	
Rheological Properties:			
Leveling	9.1	D 4062	
Low shear viscosity (consistency)	9.2	D 562	
High shear viscosity (ICI cone and plate viscosity)	9.3	D 4287	
Sag resistance	9.4	D 4400	
Rheological properties, non-Newtonian liquids	9.5	D 2196	
Appearance of Dry Paint Film:			
Color difference by visual evaluation	10.1	D 1729	
Color difference using instrumental measurements	10.2	D 2244	
Hiding power	10.3	D 344, D 2805	
Specular gloss	10.4	D 523	
Properties of Dry Paint Film:			
Block Resistance	11.1		
Efflorescence of paint film	11.2	D 1736	
Efflorescence from substrate	11.3		
Flexibility	11.4	D 522	
Film porosity	11.5	D 3258	
Print resistance	11.6		
Scrubability (wet abrasion)	11.7	D 4213 or D 2486	
Stain removal (washability)	11.8	D 3450	
Wet adhesion	11.9		
Analysis of Paint:			
Chemical analysis	12.1		
Nonvolatile content	12.2	D 2369	
Volatile organic compound (VOC)	12.3	D 3960	

6.3 Specify the identification codes. A 1 U.S. gallon (or 4-L) sample is usually sufficient for the recommended tests but for guidance in selecting a sampling plan, consult Practice E 105.

7. Liquid Paint Properties

7.1 *Condition in the Container*—Thickening, settling, and separation are undesirable and objectionable if the paint cannot be reconditioned with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties of paints after storage. Deter

TABLE 2 Alphabetical List of Test Methods

Test Method	Section	ASTM Test Method	Federal Test Method Standard No. 141
Accelerated heat-aged stability	7.1.1		
Application properties	8.1		4541
Application by brush	8.1.1		2141
Application by roller	8.1.2		2112
Application by spray	8.1.3		
Block resistance	11.1		
Brushing properties	8.1.1		4321
Chemical analysis	12.1		
Coarse particles and foreign matter	7.2	D 185	
Color compatibility	7.6		
Color difference—using instrumental measurements	10.2	D 2244	
Color difference by visual evaluation	10.1	D 1729	
Condition in container	7.1		3011
Consistency (viscosity)	9.2	D 582	
Density (weight per gallon)	7.3	D 1475	
Drying time	8.2	D 1640	4061
Efflorescence of paint film	11.2	D 1736	
Efflorescence from substrate	11.3	D 2831	
Film Porosity	11.5	D 3258	
Fineness of dispersion	7.4	D 1210	
Flexibility	11.4	D 522	
Freeze-thaw stability	7.7.1	D 2248	
Fading power	10.3	D 344, D 2805	
High shear viscosity	9.3	D 4287	
Leveling	9.1	D 4062	
Low temperature coalescence of paints	8.3	D 3793	
Microorganism resistance	7.7.3	D 2574	
Nonvolatile content (see volatile content)	12.2		
Odor	7.5		
Package stability	7.7	D 1849	
Paint resistance	7.7.4	E 70	
Producing uniform thickness films	11.6		
Rheological properties of non-Newtonian liquids	9.5	D 823	
Sag resistance	9.4	D 4400	
Sampling	6	D 3925	
Scrubability (wet abrasion, scrub resistance)	10.7	D 2486, D 4213	
Settling	7.7.5	D 869	
Specular gloss	10.4	D 523	
Stain removal (washability)	11.8	D 3450	
Touch-up uniformity	8.5	D 3928	
Volatile content	12.2	D 2369	
Volatile organic compound (VOC)	12.3	D 3960	
Wet adhesion	11.9		

mine the condition in the container in accordance with Method 3011 of Federal Test Method Standard No. 141. (See also 7.7 on Package Stability.)

7.2 Coarse Particles and Foreign Matter:

7.2.1 Paints must be free of coarse particles to form uniform films of good appearance, a typical maximum being weight % of total paint. The specified test with a 325-mesh (45- μ m) screen and water as the wash liquid gives the percent of these particles in a latex paint. Determine coarse particles and foreign matter in accordance with Test Methods D 185.

7.2.2 Another test method used in industry to determine whether coarse particles are present in a dry film is to scrape the surface of the film with a spatula or metal edge of a ruler. Any particles larger than 325 mesh (45 μ m) can be clearly seen after the surface has been scraped.

7.3 Density (Weight per Gallon)—The density as mea-

sured by weight per gallon (kilograms per litre) is used to assure product uniformity from batch to batch. It does not necessarily measure the quality of a paint. In the referenced method, the density is expressed as the weight in pounds per U.S. gal (or kg/L) of the paint at a specified temperature. Most interior semigloss and gloss paints have densities of about 10 to 12 lb/gal (1.2 to 1.4 kg/L). Determine density in accordance with Test Method D 1475.

7.4 Fineness of Dispersion:

7.4.1 The more finely a pigment is dispersed, the more efficiently it is being used. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the material down a calibrated tapered groove varying in depth from 4 to 0 mils (100 to 0 μ m). The Hegman scale is one commonly accepted measurement method. The point at which the continuous groupings of particles or agglomerates, or both, protrude through the surface of the liquid is taken as the fineness reading. Lower readings in mils or micrometres or higher readings in Hegman units indicate better fineness dispersion. Measure fineness of dispersion in accordance with Test Method D 1210.

7.4.2 The referenced method was designed primarily for coatings with good fineness of dispersion such as enamels. Most interior semigloss and gloss latex paints have a fineness of about 7.5 to 5 Hegman (6 to 40 μ m).

7.5 Odor—One of the advantages of latex paints is that they do not have odors characteristic of solvent-reducible coatings. However, other ingredients, such as ammonia, may be used that might also be objectionable in confined spaces. Thus, interior latex paints should be tested for odor acceptability. Determine whether the paint has an unpleasant or irritating odor during application or drying.

7.6 Color Compatibility—A test method to determine how well colorants can be dispersed in paints so that the paint will have uniformity of color when applied is in preparation and will be included in the guide when adopted by ASTM.

7.7 Package Stability—Since paints cannot normally be used immediately after manufacture they must remain stable in the can for some time. At normal temperatures, most latex paints can be stored for over a year with little change in properties. Although indications of long term package stability can usually be obtained in several days or weeks at an elevated temperature, such as 140°F (60°C) or 122°F (50°C), occasionally the results of the accelerated test do not agree with those at prolonged normal storage conditions. The referenced method predicts the change in consistency and certain other properties in packaged latex paint when stored at temperatures above freezing. Determine package stability in accordance with Test Method D 1849.

7.7.1 Heat Stability—Heating is used to accelerate changes in viscosity with time and to predict their affect on other properties. An excessive increase or decrease in viscosity after heat-aging is unacceptable. There is a general correlation between short-term high temperature oven stability and long-term shelf storage at ambient temperature. This test is meant to predict which paints will not be stable when stored by customers or consumers under elevated temperature for a short period of time or at room temperature for a longer period of time. After heat-aging, such

properties as viscosity, flow, gloss, pH drift, foam resistance, color uniformity, and wet adhesion are usually rechecked versus room temperature-aged controls.

7.7.2 Freeze-Thaw Stability—Water-reducible paints may be subjected to freezing conditions during shipping and storage. Suitably stabilized paints will resist several cycles of freezing and thawing without showing deleterious changes such as coagulation, graininess, or excessive viscosity increase. Many latex paints will increase in viscosity but can still be considered satisfactory if other properties that may be affected by a higher viscosity, such as leveling and brushability, are satisfactory. Determine freeze-thaw stability in accordance with Test Method D 2243.

7.7.3 Microorganism Resistance—Bacteria in a latex paint can cause gassing, putrefactive or fermentative odors, and loss of viscosity. Determine if the paint contains living bacteria or if it is resistant to attack by bacteria in accordance with Test Method D 2574.

7.7.4 pH—Latex paints with low (acidic) pH can corrode metal containers. pH may vary from about 5 to 10 depending upon the type of latex used and general formulation. pH does not determine the quality of a latex paint and should only be used to assure product uniformity. However, a change in pH during storage may indicate poor stability and unacceptable change in the properties of a paint. Determine pH in accordance with Test Method E 70.

7.7.5 Settling—Latex paints are generally resistant to hard settling, but do at times show separation and soft settling. The referenced method covers the degree of pigment suspension and ease of remixing a shelf-aged sample of paint to a homogeneous condition suitable for the intended use. Determine settling in accordance with Method D 869.

8. Paint Application and Film Formation

8.1 Application Properties—Determine the ease with which a paint can be applied to various wall surfaces with brush, roller, or spray equipment, in accordance with Method 4541 of Federal Test Method Standard No. 141. Application properties are generally compared to a standard, or described by requirements in a product specification.

8.1.1 Brush Application—Brushed films should be smooth and free of seeds and on vertical surfaces should show no sagging, color streaking, or excessive brush marks. The specified method covers the determination of the brushing properties of coatings. The test is subjective although those experienced in the art can produce quite consistent results, particularly in the evaluation of "drag" properties. Determine brushing properties in accordance with Method 4321 of Federal Test Method Standard No. 141.

8.1.2 Roller Application—Walls are frequently painted with rollers that produce slight stipple effects. Determine the ease with which a paint can be roller applied in accordance with Method 2112 of Federal Test Method Standard No. 141. Since roller foam and roller spatter are often serious problems when latex paints are roller applied, these properties can also be determined in the test method for roller application. The amount of foam produced, the time that it takes for the bubbles to break, and the number of craters that remain after the bubbles have broken can be determined by visual examination of the test panel. Roller spatter can be

determined by placing a strip of paper or a panel at the bottom of the test panel to catch the paint spatters from the roller. The degree to which a paint will spatter when roller applied can be determined by the density of the paint spatter.

8.1.3 Spray Application—Interior coatings are sometimes applied by spray. Both air and airless spray are used on commercial work. Determine the spray application properties in accordance with Method 2131 of Federal Test Method Standard No. 141. The method can be modified to include application by airless spray equipment.

8.2 Drying Properties—The drying time of an interior latex paint is important in determining when a freshly painted room can be put back to use. Under average conditions most semigloss and gloss latex paints are dry to touch in 1 to 2 h when the water has evaporated from the film. Because of the glycols usually present in these paints, it is prudent to recoat after at least an 18-h dry. Ultimate properties may take a few days to develop for some latex paints while others may require a few weeks depending on the composition. Determine the drying time in accordance with Test Methods D 1640.

8.3 Low-Temperature Coalescence of Paints—The referenced test method determines how well the latex particles in a paint will fuse together or coalesce, to form a continuous film at low temperatures. Determine low-temperature coalescence in accordance with Test Method D 3793.

8.4 Producing Films of Uniform Thickness—The following method covers the preparation of various films of uniform thickness essential in conducting tests. Prepare films in accordance with Test Methods D 823.

8.5 Touch-Up Uniformity—After paint has dried, areas where less material was applied sometimes become noticeable. If the paint has suitable touch-up properties, additional paint can be applied to these areas only, instead of refinish the complete wall. The color, gloss, and leveling of the touched-up areas and the previously painted area should be uniform. Differences in these properties are often caused by short wet edge time, poor leveling on recoat and pigment orientation or floatation during and after application. Determine touch-up properties in accordance with Test Method D 3928.

9. Rheological Properties

9.1 Leveling—Leveling is an important factor when uniform surfaces are to be produced as it affects hiding power and appearance. Brush marks and imperfections are much more conspicuous in semigloss and gloss paints than they are in flat paints. The referenced method covers the leveling characteristics of liquid coatings. Evaluate leveling in accordance with Test Method D 4062.

9.2 Low Shear Viscosity (Consistency)—Paints of a given type should fall within a stated consistency range for satisfactory reproduction of a specific formula. While consistency is an important property, it does not determine the quality of a paint and should be used mainly to ensure product uniformity. In the referenced method, consistency is defined as the load in grams to produce a specified rate of shear. Although the consistency of most latex wall paint is about 150 to 300 g, a much wider range is possible because of the wide variations in rheological properties of these paints. Two paints of the same consistency may have quite different

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ological properties since this is only a measure of their shear viscosity. Optimized application properties are generally used to determine the optimum consistency. Measure the consistency in accordance with Test Method D 562.

10.3 High Shear Viscosity (ICI Cone and Plate Viscosity)—The shear rate for this measurement (10 000 reciprocal seconds) is similar to that occurring during brush application, and the viscosity measured at this shear rate therefore is related to brush drag, which is in turn related to spreading and film build. Measure the high shear viscosity in accordance with Test Method D 4287.

10.4 Sag Resistance—This is an important property particularly for semigloss and gloss latex paints because sagging results in unsightly film appearance. Measure the sag resistance in accordance with Test Methods D 4400.

10.5 Rheological Properties, Non-Newtonian Liquids—Rheological properties are related to application and leveling properties of the liquid paint. The referenced methods cover the determination of rheological properties and are particularly suited for use with paints that display thixotropic characteristics. They actually measure viscosity under different shear rates. Determine rheological properties in accordance with Test Methods D 2196 or D 4287, or both.

10. Appearance of Dry Film

10.1 Color Differences by Visual Evaluation—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, size of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color differences in accordance with Practice D 1729.

10.2 Color Differences Using Instrumental Measurements—The differences in color between a product and its standard can be measured by instrument. Generally, the standard is agreed upon by the purchaser and the seller and may also be required if a product specification is involved. Color instruments provide numerical values that can be compared to subsequent measurements. The referenced method covers the instrumental determination of small color differences observable in daylight illumination between nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (see 10.1) should be used to verify the results. Calculate in accordance with Test Method D 2244 the color differences that have been measured instrumentally.

10.3 Hiding Power (Dry Opacity)—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness that is influenced by flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, opacity is evaluated and usually compared to a standard paint, and results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test that does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Deter-

mine hiding power in accordance with Test Methods D 344 or D 2805.

10.4 Specular Gloss—The method given, using the 20° geometry for high gloss paints and 60° geometry for semigloss paints, is useful in characterizing the direct appearance of gloss and semigloss paints. According to the *Paint/Coatings Dictionary*,⁷ the semigloss range is usually 35 to 70 using the 60° geometry. Full gloss is usually above 70 using the 60° geometry. Although paints with good uniformity of appearance are often paints of lower gloss and paints with good cleanability are often ones with higher gloss, this is not always the case, and the gloss of a paint should not be used as a measure of other paint properties. Determine the gloss at 20° and 60°, as appropriate, gloss in accordance with Test Method D 523.

11. Properties of Dry Film

11.1 Block Resistance—This is an important property of an interior semigloss or gloss since it is the resistance of the painted surfaces to stick together when stacked or placed in contact with each other. An interior paint often comes in contact with itself especially in the cases of doors, windows and drawers and sometimes sticks to itself (blocks) depending on the hardness of the coating, the pressure, temperature, humidity, and duration of time the surfaces are in contact.

11.2 Efflorescence of the Paint Film—The referenced method measures efflorescence that comes from the paint itself, not from the substrate. Few interior latex paints effloresce due to improvements in latex and latex paint formulations. Salt formation is produced by specific conditions of temperature and humidity if a paint contains sufficient solid water-soluble material to cause a noticeable deposit on the film. Determine efflorescence resistance in accordance with Test Method D 1736.

11.3 Efflorescence from Substrate—Cementitious substances may contain sufficient solid water-soluble materials to cause a surface deposit through leaching and evaporation.

11.4 Flexibility—Elongation is a measure of the flexibility of a paint film. Most interior latex paints can be bent over a 1/8-in. (3.2-mm) mandrel without affecting the film. Determine elongation in accordance with Test Methods D 522.

11.5 Film Porosity—The more porous a paint is, the worse will be its cleanability and enamel holdout. Determine film porosity in accordance with Test Method D 3258.

11.6 Print Resistance—The ability of a coating to resist printing is important because its appearance is adversely affected if the surface texture is modified by contact with another surface particularly one with a pattern. Interior gloss and semigloss systems on window sills and other horizontal surfaces often have flower pots placed on them that may tend to leave a permanent impression from the pressure. This tendency for a paint film to "print" is often a function of the hardness of the coating, the pressure, temperature, humidity, and duration of time that the painted surface is in contact with the object.

⁷ Available from Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

11.7 Scrubbability—The ability of an interior finish to resist scrubbing is an important property. The referenced method provides a measure of the wet abrasion resistance of a film. However, wet abrasion resistance is not necessarily a measure of how well soils or stains can be removed since some paints have good scrubability but poor stain cleanability because they are porous. Determine the scrubability in accordance with Test Method D 4213 or D 2486. A control paint should always be tested at the same time because of the variability of the method.

11.8 Stain Removal (Cleanability)—The ability to remove marks satisfactorily without damaging the film is an important property of interior finishes. Determine stain removal in accordance with Test Method D 3450.

11.9 Wet Adhesion—It is essential that an interior finish adhere tightly to a given substrate or primer under the wet conditions of washing or scrubbing.

12. Analysis of Paint

12.1 Chemical Analysis—If a specification requires certain raw materials or certain components in a given amount, then chemical analysis is required to determine whether the specified materials are present and in what amounts. Analysis does not necessarily establish paint quality that can also be greatly affected by manufacturing techniques. Most ASTM analytical methods apply to solvent-based coatings. However, some of them can be adapted for analysis of latex paints.

12.2 Volatile Content—The percent of volatile matter is a

measure of the amount of a liquid coating lost as it dries. This quantity is not necessarily indicative of the quality of a coating. It is useful, however, for determining the similarity of two batches. The referenced method covers the determination of the volatile content by weight of solvent- and water-reducible coatings. The quantity determined subtracted from 100 % gives the nonvolatile content. Determine volatile content in accordance with Test Method D 2369.

12.3 Volatile Organic Compound (VOC) Content—Several local jurisdictions in California have adopted air pollution controls that severely limit the amount of solvent (Volatile Organic Compound (VOC) content) permitted in architectural coatings, including interior latex gloss and semigloss paints. Since these paints may contain solvents such as coalescents and cosolvent wet edge aids, it is essential that these products not exceed the established VOC limits. Determine VOC content in accordance with Practice D 3960.

13. Field Testing

13.1 Although many of the recommended test methods attempt to simulate conditions under which latex semigloss and gloss wall paints are applied, it is not possible to accurately simulate all possible conditions. Testing latex semigloss and gloss wall paints under field conditions is recommended for the final evaluation of suitability.

14. Keywords

14.1 architectural coatings; gloss paints; interior paints; latex paints; semigloss paints

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers¹

This standard is issued under the fixed designation D 4541; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Editorial changes were made throughout in April 1989.

Scope

1.1 This test method covers a procedure and apparatus for evaluating the pull-off strength (commonly referred to as "adhesion") of a coating by determining either the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached, or whether the surface remains intact at a prescribed force (pass/fail). Failure will occur along the weakest plane within the system comprised of the test fixture, adhesive coating system, and substrate and will be exposed by the fracture surface. This test method maximizes tensile stress as compared to the shear stress applied by other methods such as scratch or knife adhesion and results may not be comparable.

1.2 This test method uses a class of apparatus known as pull-off adhesion testers.² They are portable and capable of applying a concentric load and counter load to a single surface so that coatings can be tested even though only one side is accessible. Measurements are limited by the strength of adhesion bonds between the loading fixture and the specimen surface or the cohesive strength of the substrate.

1.3 This test can be destructive and spot repairs may be necessary.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Notes 2 and 3.*

Referenced Documents

2.1 ASTM Standards:

D 2651 Practice for Preparation of Metal Surfaces for Adhesive Bonding³

D 3933 Practice for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing)³

2.2 ANSI Standard:

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coating.

Current edition approved Nov. 29, 1985. Published January 1986.

² The term adhesion tester may be somewhat of a misnomer, but its adoption by two manufacturers and at least two patents indicates continued usage.

³ Annual Book of ASTM Standards, Vol 15.06.

N 512 Protective Coatings (Paints) for the Nuclear Industry⁴

2.3 ISO Standard:

4624 Paints and Varnish—Pull-Off Test for Adhesion⁴

3. Summary of Test Method

3.1 The general pull-off test is performed by securing a loading fixture (dolly) normal (perpendicular) to the surface of the coating with an adhesive. After the adhesive is cured, a testing apparatus is attached to the loading fixture and aligned to apply tension normal (perpendicular) to the test surface. The force applied to the loading fixture is then gradually increased and monitored until either a plug of coating material is detached, or a specified value is reached. When a plug of material is detached, the exposed surface represents the plane of limiting strength within the system. The nature of the failure is qualified in accordance with the percent of adhesive and cohesive failures, and the actual interfaces and layers involved. The pull-off strength is computed based on the maximum indicated load, the instrument calibration data, and the original surface area stressed (see Fig. 1).

4. Significance and Use

4.1 The pull-off strength (commonly referred to as adhesion) of a coating is an important performance property that has been used in specifications. This test method serves as a means for uniformly preparing and testing coated surfaces, and evaluating and reporting the results. This test method is applicable to any portable apparatus meeting the basic requirements for determining the pull-off strength of a coating.

5. Apparatus

5.1 *Adhesion Tester*, commercially available, or comparable apparatus specific examples of which are listed in Annexes A1 and A2 (see Fig. 1):

5.1.1 *Loading Fixtures*, having a flat surface on one end that can be adhered to the coating and a means of attachment to the tester on the other end.

5.1.2 *Detaching Assembly* (adhesion tester), having a central grip for engaging the fixture.

⁴ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

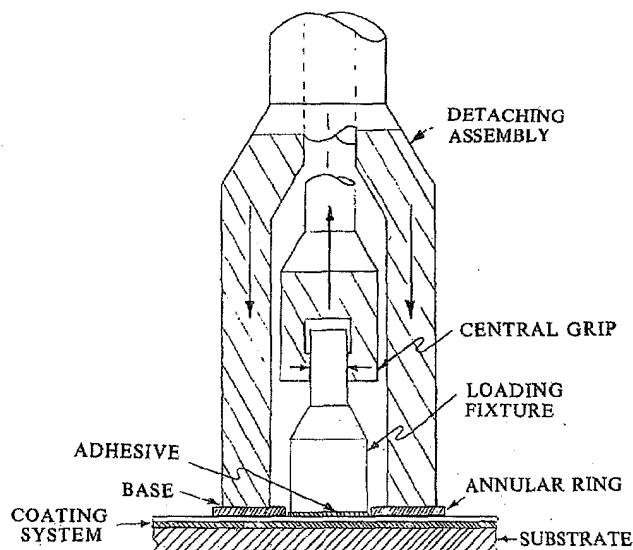


FIG. 1 Schematic of Pull-Off Adhesion Tester

5.1.3 *Base*, on the detaching assembly, or an annular bearing ring if needed for uniformly pressing against the coating surface around the fixture either directly, or by way of an intermediate bearing ring. A means of aligning the base is needed so that the resultant force is normal to the surface. If shims are required when a bearing ring is employed, place them between the tester base and bearing ring rather than on the coating surface.

5.1.4 Means of moving the grip away from the base in as smooth and continuous a manner as possible so that a torsion free, co-axial (opposing pull of the grip and push of the base along the same axis) force results between them.

5.1.5 *Timer*, or means of limiting the rate of stress to less than 150 psi/s (1 MPa/s) so that the maximum stress is obtained in less than about 100 s. A timer would be the minimum equipment when used by the operator along with the force indicator in 5.1.6.

5.1.6 *Force Indicator and Calibration Information*, for determining the actual force delivered to the loading fixture.

5.2 *Solvent*, or other means for cleaning the loading fixture surface. Finger prints, moisture, and oxides tend to be the primary contaminants.

5.3 *Fine Sandpaper*, or other means of cleaning the coating that will not alter its integrity by chemical or solvent attack. If any light sanding is anticipated, choose only a very fine grade abrasive that will not introduce flaws or leave a residue.

5.4 *Adhesive*, for securing the fixture to the coating that does not affect the coating properties. Two component

epoxies⁵ and acrylics⁶ have been found to be the most versatile.

5.5 *Magnetic or Mechanical Clamps*, if needed, for holding the fixture in place while the adhesive cures.

5.6 *Cotton Swabs*, or other means for removing excess adhesive and defining the adhered area. Any method for removing excess adhesive that damages the surface, such as scoring, must generally be avoided since induced surface flaws may cause premature failure of the coating.

5.7 *Circular Hole Cutter* (optional), to score through to the substrate around the loading fixture.

6. Test Preparation

6.1 The method for selecting the coating sites to be prepared for testing depends upon the objectives of the test and agreements between the contracting parties. There are, however, a few physical restrictions imposed by the general method and apparatus. The following requirements apply to all sites:

6.1.1 The selected test area must be a flat surface large enough to accommodate the specified number of replicate tests. The surface may have any orientation with reference to gravitational pull. Each test site must be separated by at least the distance needed to accommodate the detaching appa-

⁵ Araldite Adhesive, available from Ciba-Geigy Plastics, Duxford, Cambridge, CB2 4QA, England, Scotch Weld Adhesive 1838B/A, available from 3M, Adhesive Coatings and Sealers Div., 3M Center, St. Paul, MN 55144, and Hysol Epoxy Patch Kit 907, available from Hysol Div., The Dexter Corp., Willow Pass Rd., Pittsburg, CA 94565, have been found satisfactory for this purpose.

⁶ Versilac 201 and 204 with accelerator, available from Lord Corp., Industrial Adhesive Div., 2010 W. Grandview Blvd., P.O. Box 10038, Erie, PA 16514, have been found satisfactory for this purpose.

status. The size of a test site is essentially that of the secured loading fixture. At least three replications are usually required in order to statistically characterize the test area.

6.1.2 The selected test areas must also have enough perpendicular and radial clearance to accommodate the apparatus, be flat enough to permit alignment, and be rigid enough to support the counter force.

6.2 Since the rigidity of the substrate affects pull-off strength results and is not a controllable test variable in field measurements, some knowledge of the substrate thickness and composition should be reported for subsequent analysis of laboratory comparisons. For example, steel substrate of less than 1/8-in. (3.2-mm) thickness may reduce pull-off strength results compared to 1/4 in. (6.4 mm) thick panels.

6.3 Subject to the requirements of 6.1, select representative test areas and clean the surfaces in a manner that will not affect integrity of the coating or leave a residue. Surface abrasion may introduce flaws and should generally be avoided. A light abrasive should only be used if needed to remove loose or weakly adhered surface contaminants.

6.4 Clean the loading fixture surface as indicated by the apparatus manufacturer. Failures at the fixture-adhesive interface can often be avoided by treating the fixture surfaces in accordance with an appropriate ASTM standard practice for preparing metal surfaces for adhesive bonding.

NOTE 1—Practices D 2651 and D 3933 are typical of well-proven methods for improving adhesive bond strengths to metal surfaces.

6.5 Mix the adhesive in accordance with the adhesive manufacturer's recommendations, applying the adhesive between the fixture and the surface to be tested using a method preferred by the adhesion-tester manufacturer. Carefully remove the excess adhesive from around the fixture.

NOTE 2: Caution—Movement, especially twisting, can cause tiny bubbles to coalesce into large holidays that constitute stress discontinuities during testing.

6.6 Based on the adhesive manufacturer's recommendations and the anticipated environmental conditions, allow enough time for the adhesive to set up and reach the recommended cure. During the adhesive set and early cure stage, a constant contact pressure should be maintained on the fixture. Magnetic or mechanical clamping systems work well, but systems relying on tack, such as masking tape, should be used with care to ensure that they do not relax with time and allow air to intrude between the fixture and the test area.

6.7 Scoring around the fixture violates the fundamental in-situ test criterion that an unaltered coating be tested. If scoring around the test surface is employed, extreme care is required to prevent micro-cracking in the coating. Such cracks may cause failures at diminished strengths. Scored samples constitute a different test, and this procedure should be clearly reported with the results.

6.8 Note the approximate temperature and relative humidity during the time of test.

Test Procedure

7.1 Select an adhesion-tester with a detaching assembly having a force calibration spanning the range of expected values along with its compatible loading fixture. Mid-range measurements are usually the best, but before proceeding,

read the manufacturer's operating instructions.

7.2 If a bearing ring or comparable device (5.1.3) is to be used, place it concentrically around the loading fixture on the coating surface.

7.3 Carefully connect the central grip of the detaching assembly to the loading fixture without bumping, bending, or otherwise prestressing the sample and connect the detaching assembly to its control mechanism, if necessary. For nonhorizontal surfaces, the detaching assembly should be manually supported so that its weight does not contribute to the force exerted in the test.

7.4 Align the device according to the manufacturer's instructions and set the force indicator to zero.

NOTE 3: Caution—Proper alignment is critical. If shims are needed for fixed alignment devices, report the manner in which used.

7.5 Increase the load to the fixture in as smooth and continuous a manner as possible, at a rate of less than 150 psi/s (1 MPa/s) so that failure occurs or the maximum stress is reached in about 100 s or less.

7.6 Record the force attained at failure or the maximum force applied.

7.7 If a plug of material is detached, label and store the fixture for qualification of the failed surface in accordance with the procedure in 8.3.

7.8 Report any departures from the procedure such as possible misalignment, hesitations in the force application, etc.

8. Calculation and Interpretation of Results

8.1 Use the instrument calibration factors to convert the indicated force for each test into the actual force applied in units of pounds-force (1 N = 0.1 kgf).

8.2 Compute the relative stress applied to each coating sample as follows:

$$X = 4F/d^2\pi$$

where:

X = greatest mean pull-off stress applied during a pass/fail test, or the pull-off strength achieved at failure. Both have units of psi (MPa = 1 N/mm²).

F = highest force applied to the test surface as determined in procedure in 8.1, and

d = equivalent diameter of the original surface area stressed having units of inches (or millimetres). This is usually equal to the diameter of the loading fixture.

8.3 For all tests to failure, estimate the percent of adhesive and cohesive failures in accordance to their respective areas and location within the test system comprised of coating and adhesive layers. A convenient scheme that describes the total test system is outlined in procedures in 8.3.1 through 8.3.3. (See ISO 4624.)

8.3.1 Describe the specimen as substrate A , upon which successive coating layers B , C , D , etc., have been applied, including the adhesive, Y , that secures the fixture, Z , to the top coat.

8.3.2 Designate cohesive failures by the layers within which they occur as B , C , etc., and the percent of each.

8.3.3 Designate adhesive failures by the interfaces at which they occur as A/B , B/C , C/D , etc., and the percent of each.

8.4 Erratic errors should be dropped. Unpredictable errors

may result if alignment of the apparatus is not normal to the surface, from poor definition of the area stressed due to improper preparation of the adhesive, poorly defined glue lines and boundaries, holidays in the adhesive caused by voids, inclusions, improperly prepared surfaces, or sliding or twisting the fixture during the initial cure. Scratched or scored samples may contain stress concentrations leading to premature fractures. These should be retested at adjacent areas.

8.5 Further information relative to the interpretation of the test results is given in Appendix A1.

9. Report

9.1 Report the following information:

9.1.1 Brief description of the general nature of the test, such as, field or laboratory testing of the general type of coating as applied to a given substrate, etc.

9.1.2 Temperature and relative humidity and any other pertinent environmental conditions during the test period.

9.1.3 Apparatus used, fixed or self-aligning, which includes: apparatus manufacturer and model numbers, loading fixture type and dimensions, and bearing ring type and dimensions.

9.1.4 Description of the test system, if possible, by the indexing scheme outlined in 8.3 including: product identity and generic type for each coat and any other information supplied, the substrate identity (thickness, type, orientation, etc.), and the adhesive used.

9.1.5 Test results.

9.1.5.1 Date, test location, testing agent.

9.1.5.2 For pass/fail tests, stress applied along with the result, for example, pass or fail and note the plane of any failure (see ANSI N512).

9.1.5.3 For tests to failure, all values computed in 8.2 along with the nature and location of the failures as specified in 8.3, or, if only the average strength is required, report it along with the statistics.

9.1.5.4 If corrections of the results have been made, or if certain values have been omitted such as the lowest or highest values or others, reasons for the adjustments and criteria used.

9.1.5.5 For any test where scoring was employed, indicate it by placing a footnote superscript beside each data point affected and a footnote to that effect at the bottom of each page on which such data appears. Note any other deviations from the procedure.

10. Precision and Bias

10.1 The precision and bias is primarily dependent upon the accuracy of the force measurement, the alignment of the device, and the care exercised in preparation and testing.

10.2 Since no standard for pull-off strength of coatings has been developed prior to this test method, precision and bias statements will be developed in round-robin testing.

11. Keywords

11.1 adhesion; adhesive strength; bond strength; coatings cohesion; cohesive strength; paints; pull-off strength

ANNEXES

(Mandatory Information)

A1. FIXED-ALIGNMENT ADHESION TESTERS—ELCOMETER MODEL 106 ADHESION TESTER (Fig. A1.1)⁷

A1.1 Apparatus:

A1.1.1 This is a fixed-alignment portable tester.

A1.1.2 The tester is comprised of detachable aluminum loading fixtures having a flat conic base that is 0.8 in. in diameter on one end for securing to the coating, and a circular T-bolt head on the other end, a central grip for engaging the loading fixture that is forced away from a tripod base by the interaction of a handwheel (or nut), and a co-axial bolt connected through a series of Belleville washers, or springs in later models, that acts as both a torsion relief and a spring that displaces a dragging indicator with respect to a scale.

A1.1.3 The force is indicated by measuring the maximum spring displacement when loaded. Care should be taken to see that substrate bending does not influence its final position or the actual force delivered by the spring arrangement.

A1.1.4 The devices are available in four ranges: from 0 to 500, 0 to 1000, 0 to 2000, and 0 to 4000 psi (35, 70, 140 and 280 kgf/cm²).

A1.2 Procedure:

A1.2.1 Center the bearing ring on the coating surface concentric with the loading fixture. Turn the hand wheel or nut of the tester counter-clockwise, lowering the grip so that it slips under the head of the loading fixture.

A1.2.2 Align or shim the three instrument swivel pads of the tripod base so that the instrument will pull perpendicularly to the surface at the bearing ring.

A1.2.3 Take up the slack between the various members and slide the dragging (force) indicator located on the tester to zero.

A1.2.4 Firmly hold the instrument with one hand. Do not allow the base to move or slide during the test. With the other hand, turn the handwheel clockwise using as smooth and constant a motion as possible. Do not jerk or exceed a stress rate of 150 psi/s (1 MPa/s) which is attained by allowing in excess of 7 s/1000 psi stress. If the 2000 or 4000 psi models are used, the handwheel is replaced with a nut requiring a wrench for tightening. The wrench must be used

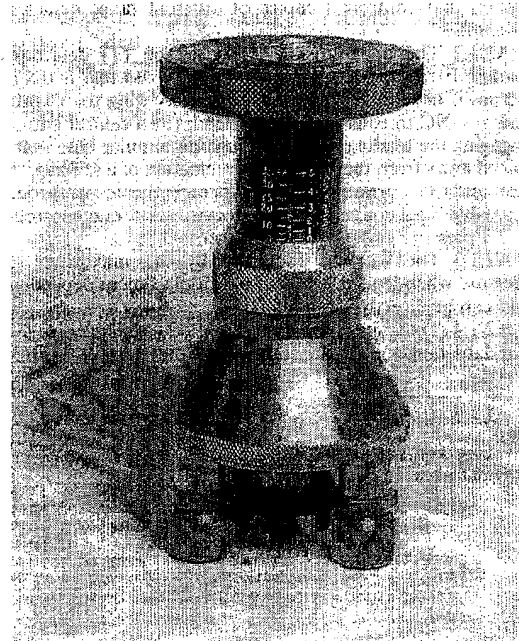


FIG. A1.1 Elcometer Model 106 Adhesion Tester

in a plane parallel to the substrate so that the loading fixture will not be removed by a shearing force or misalignment thus negating the results. The maximum stress must be reached within about 100 s.

A1.2.5 The pulling force applied to the loading fixture is increased to a maximum or until the system fails at its weakest locus. Upon failure, the scale will rise slightly, while the dragging indicator retains the apparent load. The apparatus scale indicates an approximate stress directly in pounds per square inch, but may be compared to a calibration curve.

A1.2.6 Record the highest value attained by reading along the bottom of the dragging indicator.

⁷ Manufactured by Elcometer Instruments, Ltd., Edge Lane, Droylston, Manchester M35 6UB, England.

A2. SELF-ALIGNING ADHESION TESTERS—SEMICRO PNEUMATIC ADHESION TESTER (Fig. A2.1)⁸

A2.1 Apparatus:

A2.1.1 This is a self-aligning tester, although a non-self-aligning pneumatic apparatus is in use in Europe.⁹ It is portable and has a common pressure source and measuring system that controls a choice of different range detaching assemblies.

A2.1.2 The apparatus is comprised of: (1) detachable loading fixtures having a flat cylindrical base that is 0.5 in. (13 mm) in diameter on one end, for securing the coating, and $\frac{3}{8}$ UNC threads on the other end; (2) a central grip for engaging the loading fixture through an annular base that is forced away from the grip by the interaction of a self-aligning seal; and (3) a pressurized gas that enters the device through a flexible hose connected to a pressurization rate controller and a pressure gage (or electronic sensor).

A2.1.3 The force is indicated by the maximum gas pressure when loaded, which is not displacement dependent, and can be directly calibrated.

A2.1.4 The detaching assemblies are available in four standard ranges in multiples of two from 0 to 500 psi (3.5

MPa) to 0 to 4000 psi (28 MPa). Special ranges to 10 000 psi (70 MPa) are available.

A2.1.5 The standard System 2000 is a fully automated electronic monitoring and control system of briefcase size that can be used with any detaching assembly. Special manually controlled pneumatic devices can be ordered.

A2.2 Procedure:

A2.2.1 Position the annular ring on the coating concentric with the fixture, and loosely engage the fixture via the central threaded grip. Leave at least $\frac{1}{32}$ -in. (0.8-mm) clearance between the detaching assembly and the annular ring so that the seal can protrude enough to align itself when pressurized.

A2.2.2 Make the appropriate pneumatic connections, and adjust the pressure.

A2.2.3 Initialize the system by nulling the force indicator and introducing a small amount of gas in order to set the seal and align the device.

A2.2.4 If the device is fully automated, select the desired rate of load and set the mode switch to its run position; otherwise, manually control the gas pressure to the device so that the rate of stress does not exceed 150 psi/s (1 MPa/s) yet reaches its maximum within 100 s.

A2.2.5 Record both the maximum pressure attained and the appropriate force or stress multiplier for the specific detaching assembly.

⁸ Manufactured by SEMicro Corp., 15817 Crabbs Branch Way, Rockville, MD 20855.

⁹ Saberg Apparatus, available from Scandinavian Paint and Printing Inc., Research Institute, Copenhagen, Denmark.

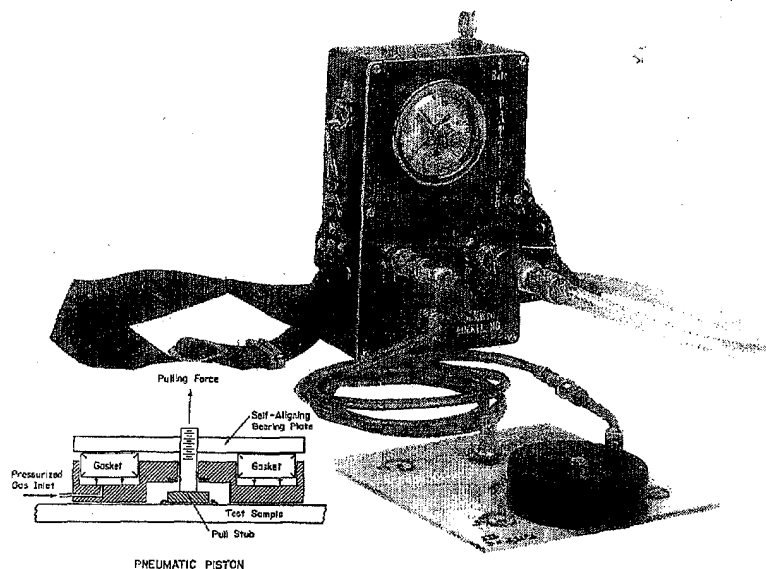


FIG. A2.1 SEMicro Pneumatic Adhesion Tester

APPENDIX

(Nonmandatory Information)

X1. STRESS CALCULATION

X1.1 The stress computed in 8.2 is equal to the uniform pull-off strength of the analogous rigid coating system if the applied force is distributed uniformly over the critical locus at the instant of failure. For any given continuous stress distribution where the peak-to-mean stress ratio is known, the uniform pull-off strength may be approximated as:

$$U = XR$$

where:

U = uniform pull-off strength, representing the greatest force that could be applied to the given surface area, psi (MPa),
 X = measured in-situ pull-off strength calculated in 8.2, psi (or MPa) and
 R = peak-to-mean stress ratio.

It is important to note that a difference between these pull-off strengths does not necessarily constitute an error;

rather the in-situ measurement simply reflects the actual character of the applied coating system with respect to the analogous "ideal" rigid system.

X1.2 An error is introduced if the alignment of the apparatus is not normal to the surface. An approximate correction by the peak-to-mean stress ratio is:

$$R = (1 + 0.14 az/d),$$

where:

z = distance from the surface to the first gimbal or the point at which the force and counter force are generated by the action of the driving mechanism, in. (mm),
 d = diameter of the loading fixture, in. (mm),
 a = angle of misalignment, degrees (less than 5), and
 R = maximum peak-to-mean stress ratio for the misaligned rigid system.

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Standard Test Method for Determination by Atomic Absorption Spectroscopy of Titanium Dioxide Content of Pigments Recovered From Whole Paint¹

This standard is issued under the fixed designation D 4563; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Keywords were added editorially in February 1991.

1. Scope

1.1 This test method covers the atomic absorption (AA) analysis of titanium dioxide content in pigments recovered from whole paint. It is applicable to quality control situations where the same type of product is repeatedly analyzed.

1.2 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1193 Specification for Reagent Water²
- D 1394 Test Methods for Chemical Analysis of White Titanium Pigments³
- E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁴
- E 288 Specification for Volumetric Flasks⁵

3. Significance and Use

3.1 This test method may be used in quality control laboratories when the repeated analysis of titanium dioxide in similar paints may be required. Reagents and time are kept to a minimum when this test method is used in place of wet chemical analysis such as in Test Methods D 1394. However, reproducibility and repeatability are not as good as in Test Methods D 1394.

4. Summary of Test Method

4.1 The specimen is prepared for analysis by ashing at 450°C followed by digestion with sulfuric acid and ammo-

nium sulfate as in Methods D 1394. The titanium content is determined by atomic absorption spectroscopy using a specimen similar to that previously analyzed by the Aluminum Reduction Method in Test Methods D 1394.

4.2 By utilizing the pigment analyzed in Test Methods D 1394 as an atomic absorption standard, several hundred TiO₂ determinations can be made. The AA technique is much faster than the technique in Test Methods D 1394 for multiple determinations and uses only acids. This keeps reagents and time to a minimum.

5. Apparatus

- 5.1 *Atomic Absorption Spectrophotometer*, consisting of:
 - 5.1.1 Atomizer and nitrous oxide burner,
 - 5.1.2 Gas-pressure regulator and metering devices for nitrous oxide and acetylene,
 - 5.1.3 Titanium hollow cathode lamp with regulated constant-current supply,
 - 5.1.4 Monochromator and associated optics,
 - 5.1.5 Photosensitive detector connected to an electronic amplifier,
 - 5.1.6 Readout device.
- 5.2 *Muffle Furnace*, capable of maintaining 450 ± 25 °C.
- 5.3 *Circulating Oven*, maintained at 105 ± 2°C.
- 5.4 *Porcelain Dishes*, 90-mm diameter.
- 5.5 *Plastic Disposable Syringe*, 10-mL capacity.
- 5.6 *Agate Mortar and Pestle*, 95-mm outside diameter.
- 5.7 *Wide-Mouth Erlenmeyer Flask*, 500-mL capacity.
- 5.8 *Hot Plate*, with variable surface temperature control from 10°C above ambient to 370°C accurate to within ±5°C.
- 5.9 *Burner*.
- 5.10 *Volumetric Flask*, 1000 mL, plastic (see Specification E 288).
- 5.11 *Paint Shaker*.
- 5.12 *Weighing Bottles*, wide-mouth, with an external fitting cap, and no larger than necessary for required amount of sample.
- 5.13 *Desiccator*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chem-

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Jan. 31, 1986. Published March 1986.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without reducing the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.⁷

6.3 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

6.4 *Ammonium Sulfate* (NH₄)₂SO₄.

6.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

6.6 *Hydrofluoric Acid* (HF)—Approximately 49 %.

6.7 *Standard Pigment Solution*.

6.7.1 Following the procedure in Section 9 recover the pigment from a paint that is similar to the unknown specimen. Analyze the extracted pigment for TiO₂ content in accordance with the Aluminum Reduction Method in Test Methods D 1394 and record the percent TiO₂ found in the pigment.

6.7.2 Following the procedure in 12.1 through 12.5 take a specimen of the pigment extracted in 6.7.1 into solution. Store this solution for no more than 3 months in a plastic bottle marked with the percent TiO₂ as determined in 6.7.1.

6.8 *Sulfuric Acid* (sp gr 1.84), concentrated sulfuric acid (H₂SO₄).

6.9 *Toluene*.

Hazards

7.1 *Concentrated Hydrofluoric Acid*—Make certain to observe manufacturer's recommended precautions for handling.

7.2 *Warning*—Nitrous oxide and acetylene can cause explosions, if not used properly. See the supplier's manual of instructions for the atomic absorption instrument for proper operation with these gases.

Calibration and Standardization

8.1 Operational instructions for atomic absorption spectrophotometers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

8.2 Turn the instrument on and set the wavelength to the 353-nm titanium line. Apply the recommended current to the titanium hollow cathode lamp. Allow the instrument to warm up for about 15 min and set the proper slit width. Adjust the gas pressures and ignite the burner in accordance with instructions for using nitrous oxide and acetylene.

8.3 Aspirate the water to rinse the atomizer chamber from 10 to 15 min until the burner head achieves temperature equilibrium. Set the instrument reading to zero while doing this. While aspirating the working standard from 6.7.2 set the instrument to the percent TiO₂ (determined in 6.7.1). Aspirate the water and reset the instrument to ZERO. Repeat this procedure until the readings become stable.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, DC. For suggestions on testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards" by Joseph E. Ingham, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

8.3.1 Expanding the scale of a spectrophotometer increases the noise level of the readout system. Therefore, if expanding the scale to make the readout indicate 60 % TiO₂ has this effect, do not use any expansion. For instance, it is not necessary to make the instrument read 60 for 60 % TiO₂ standard. It may read 30. If 60 % equals 30, a pigment giving a reading of 25 would have a TiO₂ content of 50 %.

PIGMENT CONTENT

9. Procedure

9.1 Mix the samples until homogenous, preferably on a mechanical shaker. If air bubbles become entrapped in the sample, stir by hand.

9.2 Draw approximately 5 g of the paint under test into a 10-mL syringe and weigh to 1 mg. Transfer, by dropwise addition, between 2.0 and 4.0 g of the specimen to a tared porcelain dish containing either 2 mL of water (for water-borne paint) or 2 mL of toluene (for solvent-borne paint). Reweigh the syringe to 1 mg. Swirl the dish during the addition of the paint and continue to swirl until the specimen is completely dispersed. If a water-borne paint tends to agglomerate or form lumps that cannot be dispersed, a drop or two of concentrated NH₄OH may help the dispersement. If the lumps persist, discard the specimen, and prepare a new one. Prepare a duplicate specimen in the same manner.

9.3 Dry the specimens at 110°C for 30 min and then drive off the remaining solvent or water at the lowest temperature possible using a Meker burner (under a hood). Do not leave the dishes on the burners after the flames have subsided.

9.4 Transfer the dried specimens to a muffle furnace and heat at 450 ± 25°C for at least 1 h and until no further char is evident. Leave the furnace door slightly open after first inserting the dishes to allow smoke and possibly flames to escape making certain the furnace is well vented.

9.5 Remove the dishes from the muffle furnace, cool in a desiccator, and weigh.

9.6 Grind the pigment to pass through an 80-mesh screen.

10. Calculation

10.1 Calculate the percent pigment content as follows:

$$P = \frac{C - W_1}{W_2} \times 100$$

where:

P = pigment content, %

C = weight of the dish and specimen after ignition, g.

W_1 = weight of the dish alone, g.

W_2 = specimen weight used, g.

11. Precision and Bias (see Practice E 180)⁷

11.1 In an interlaboratory study of this test method in which operators in each of seven laboratories made duplicate analyses on different days and one operator in one laboratory made duplicate analyses on one day and a single analysis on the second day on a water-borne flat wall paint, a solvent-

⁷ Supporting data are available from ASTM Headquarters. Request RR: D01-1046.

borne semi-gloss enamel and a solvent-borne house paint containing 33, 38.5, and 37 % pigment, respectively, the pooled within-laboratory standard deviation was found to be 0.07 % absolute with 21 df. The pooled between-laboratories standard deviation was 0.22 % absolute with 21 df. One laboratory's results for one day were discarded because the duplicates differed significantly and another laboratory's results for one day because the range differed significantly from all other ranges for Coating 1 and a third laboratory's results for one day because it differed significantly from the other day's results and those from all other laboratories for Coating 2. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results, each the mean of duplicates, obtained by the same operator should be considered suspect if they differ by more than 0.21 % absolute at pigment contents of 33 to 39 wt %.

11.1.2 *Reproducibility*—Two results, each the mean of duplicates obtained by operators in different laboratories, should be considered suspect if they differ by more than 0.66 % absolute at the same pigment content levels.

TITANIUM DIOXIDE CONTENT

12. Procedure

12.1 Determine the dry weight of two weighing bottles with caps. In accordance with Table 1 select the appropriate specimen weight and place the approximate amount of recovered pigment in each weighing bottle.

12.2 Dry the specimens in the open weighing bottles for 2 h at 105°C. Cool in a desiccator. After cooling, cap the weighing bottles and weigh as rapidly as possible.

12.3 Transfer the dry specimens to 500-mL wide-mouth Erlenmeyer flasks. Reweigh bottles and caps and record weights of specimens transferred (W_4 in equation in 13.1 and 13.2).

12.4 To the 500-mL flasks, which now contain the specimens, add 8 g $\pm$ 0.001 g of $(\text{NH}_4)_2\text{SO}_4$, 50 mL of concentrated H_2SO_4 and 5 drops of concentrated HNO_3 . Mix well by swirling the flasks. Heat on a hot plate until dense white fumes are evolved. Continue heating over a hot flame until solutions are complete (usually requires not more than 5 min of boiling), or until it is apparent that the residues are composed of SiO_2 or siliceous matter. Cool and add 100 mL of water with extreme caution.

TABLE 1 Specimen Weight Requirement

Expected TiO_2 on pigment, %	Specimen Weight, mg ^a
0 to 19	1000
20 to 29	500
30 to 39	330
40 to 49	250
50 to 59	200
60 to 69	160
70 to 79	140
80 to 89	130
90 to 100	110

^a Equivalent to about 60 ppm Ti in the final solution.

12.5 Quantitatively transfer the contents of the flasks including any insoluble matter to 1000-mL plastic volumetric flasks (see Specification E 288). Add 50 mL of HCl and 30 mL of HF . Allow to cool to room temperature and then fill to the mark with water. Stopper and mix well. Transfer the solutions to plastic bottles (because of the HF) and store for no more than 3 months for analysis by AA. Solutions must be filtered through a Whatman 42 paper before being aspirated.

12.6 Calibrate the instrument in accordance with Section 8 and aspirate the solutions in the following order:

- 1—water (set to ZERO)
- 2—working standard (set to percent TiO_2)
- 3—specimen (read)
- 1—water (set to ZERO)
- 2—working standard (set to percent TiO_2)
- 3—specimen (read)

This MUST be repeated until the readings are stable.

13. Calculation

13.1 Calculate the percent TiO_2 in the pigment as follows:

$$\text{TiO}_2, \% = \frac{C \times W_3}{W_4}$$

where:

C = reading of specimen (number 3 in 12.6),

W_3 = standard pigment weight in 6.7.1, g, and

W_4 = specimen pigment weight in 12.3, g.

13.2 Calculate the percent TiO_2 in the whole paint as follows:

$$\text{TiO}_2, \% = \frac{P \times W_3 \times A}{W_4}$$

where:

A = pigment calculated in 10.1, %, and

P , W_3 , and W_4 = same as in 13.1.

14. Precision and Bias (see Practice E 180)⁷

14.1 In an interlaboratory study of this test method in which operators in each of eight laboratories analyzed on different days a water-borne flat wall paint, a solvent-borne semi-gloss enamel and a solvent-borne house paint, respectively containing 28.8, 56.8, and 42.8 weight % titanium dioxide in the pigment, the pooled within-laboratory standard deviation was found to be 0.65 % absolute with 22 degrees of freedom. The pooled between-laboratories standard deviation was 1.31 % absolute with 20 degrees of freedom. One duplicate value from one laboratory was discarded because the range differed significantly from all other duplicate ranges for Coating 1, one day's result from another laboratory because the range differed significantly from all other ranges and both day's results from a third laboratory were discarded because the mean differed significantly from all other laboratory means for Coating 2, and one duplicate value was discarded from the same laboratory as with Coating 1 because the range differed significantly from all other duplicate ranges for Coating 3. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

14.1.1
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contents
14.1.2
uplicate

14.1.1 *Repeatability*—Two results, each the mean of duplicates obtained by the same operator, should be considered suspect if they differ by more than 1.91 % absolute at TiO_2 contents of 28 to 57 weight % of the pigment.

14.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories

should be considered suspect if they differ by more than 3.68 % absolute at the same levels of TiO_2 content.

15. Keywords

15.1 atomic adsorption spectroscopy; percent pigment by ignition; titanium dioxide, analysis of

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Measuring Apparent pH of Electrocoat Baths¹

This standard is issued under the fixed designation D 4584; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^{e1}) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Keywords were added editorially in January 1991.

1. Scope

1.1 This test method covers the measurement of the free hydrogen ion concentration of electrocoat baths and their ultrafiltrates.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1293 Test Methods for pH of Water³

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴

E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁴

3. Summary of Test Method

3.1 A specimen of a well-agitated electrocoat bath is placed in a stirrer-equipped container and the pH measured with a pH meter and associated glass and reference electrodes.

4. Significance and Use

4.1 The pH is the measure of the free hydrogen ion concentration of a sample, and it indicates whether an electrocoat bath is acidic, neutral, or basic. Since pH measurements of good precision are made in aqueous solutions, it is suggested that the pH measurements of electrocoat baths are only semi-quantitative, and therefore such measurements should be referred to as apparent pH measurements.

4.2 The pH of electrocoat paints is used for research, production, and quality control or electrocoat bath process control.

4.3 Other related methods for determining the pH of water or aqueous systems are described in Test Methods D 1293 and E 70.

5. Apparatus

5.1 pH Meter,

5.2 Glass and Reference Electrodes.

NOTE—Due to the polarity of electrocoat materials it is desirable to use a separate set of electrodes for each bath polarity, cathodic and anodic, because a bath of opposite polarity poisons the electrodes. A desired practice is to rinse the electrodes after each measurement with an appropriate solvent for the electrocoat material.

5.3 Thermometer, capable of 0.5°C accuracy with a -2 to 32°C range.

6. Reagents

6.1 *Reference Standard Solutions*, commercial standards of pH 4.0, 7.0, and 10.0.

6.2 *Purity of Water*—References to water shall be understood to mean water conforming to Type II of Specification D 1193.

7. Sampling and Sample Preparation

7.1 The sample should be obtained while the electrocoat bath is under proper circulation so that a uniform material is obtained. In case of an ultrafiltrate, the material should be thoroughly mixed or stirred prior to sampling to assure uniformity.

7.2 After sampling and prior to removing a test specimen it is mandatory that the sample be shaken or stirred until it is homogeneous and free of any settled material. This is particularly important if there is any delay between the sampling of the bath and the preparation of the specimens for the test. The absence of settled material can be ascertained visually (in a transparent container) or by inserting a spatula, scraping the bottom of the container to make sure that there is no settled matter. Shake or stir the samples until specimens are taken for measurement; *this point is very important.*

8. Procedure

8.1 *Standardization*—Turn on the pH meter and allow it to warm up. Wash the electrodes with a stream of water. Use the pH meter manufacturer's instructions to calibrate the electrodes with buffer solutions of pH 4 and 7 for cationic systems and 7 and 10 for anionic systems. Rinsing the electrodes thoroughly after each calibration. The reference buffer solutions should thoroughly match the temperature of the samples to be measured within 2°C.

8.2 Apparent pH Measurement:

8.2.1 Place a suitable quantity of a well-mixed electrocoat specimen in a clean and dry container equipped with a stirrer (preferably magnetic). Operate the stirrer fast enough to avoid separation of the paint but slow enough to prevent splashing or vortexing. Allow the temperature of the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved April 23, 1986. Published June 1986.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

specimen to come to equilibrium (that is, the temperature should show a drift of less than 0.5°C/min). Record the temperature, and dial in the temperature correction on the pH meter. Place the electrodes in the specimen and determine the pH value in accordance with the pH meter manufacturer's instructions. Record the pH value and the temperature of the specimen.

8.2.2 Run two successive specimens of the electrocoat bath sample; the pH value should be within 0.1 units/min and should show drifts of less than 0.1 units/min. If these drift limits are not met, check the pH meter and repeat the whole procedure.

9. Precision and Bias

9.1 In an interlaboratory study of the test method, with five laboratories measuring five electrocoat bath samples with pH ranging from 2.6 to 8.0, the intralaboratory standard deviation (in accordance with Practice E 180) was

found to be 0.03 units at 25 df, and the interlaboratory coefficient of variation was 0.08 units at 20 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.1 units.

9.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.3 units.

9.1.3 *Bias*—Bias cannot be determined for this test method.

10. Keywords

10.1 electrocoat baths, pH; pH measurement; ultrafiltrates, pH

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Standard Practice for Testing Water Resistance of Coatings Using Controlled Condensation¹

This standard is issued under the fixed designation D 4585; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Paragraph 7.3 was corrected editorially in June 1988.

1. Scope

1.1 This practice covers basic principles and operating procedures for testing water resistance of coatings using controlled condensation. Condensation is produced by exposing one surface of a coated specimen to a heated, saturated mixture of air and water vapor, while the reverse side of the specimen is exposed to the cooling effect of room temperature air. This practice is derived from research of the Cleveland Society for Coatings Technology.²

1.2 This practice is limited to the methods of obtaining, measuring, and controlling conditions and procedures of controlled condensation tests. It does not specify specimen preparation, specific test conditions, or evaluation of results.

NOTE 1—Alternative practices for testing water resistance of coatings include Practices D 870, D 1735, and D 2247.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products³
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces³
- D 714 Test Method for Evaluating Degree of Blistering Paints³
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³
- D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion³

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Nov. 27, 1987. Published January 1988. Originally published as D 4585 - 86. Last previous edition D 4585 - 86a.

² Foecking, N. J., "Cleveland Condensing Type Humidity Cabinet," *Official Digest*, December 1963, Vol 35, No. 467, pp. 1318-1327; and Higgins, W. A., "Cleveland Condensing Type Humidity Cabinet: II," *Official Digest*, November 1965, Vol 37, No. 490, pp. 1392-1404.

³ *Annual Book of ASTM Standards*, Vol 06.01.

D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴

D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus³

D 2247 Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity³

D 2616 Test Method for Evaluation of Visual Color Difference With a Gray Scale⁵

D 3359 Test Methods for Measuring Adhesion by Tape Test³

D 3363 Test Method for Film Hardness by Pencil Test³

G 53 Practice for Operating Light- and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Nonmetallic Materials⁶

3. Summary of Practice

3.1 Water vapor is generated by heating a pan of water at the bottom of the test chamber. The specimens form the roof or walls of the test chamber so that the back sides of the specimens are exposed to the cooling effects of room temperature air. The resulting heat transfer causes vapor to condense on the test specimens as liquid water saturated with air.

3.2 The temperature and amount of condensate forming on the specimens is controlled by the test temperature and the room temperature. The test specimens are inclined so that condensate runs off the test surface by gravity and is replaced by fresh condensate in a continuous process during the condensate cycle.

3.3 Exposure conditions are varied by selecting: (a) the temperature of the test, (b) the duration of the test, and (c) periodic drying of the specimens. Testing may be conducted at temperatures from 100 to 180°F (38 to 82°C). Any effects such as color change, blistering, loss of adhesion, softening, or embrittlement are observed and reported.

4. Significance and Use

4.1 Water can cause degradation of coatings, so knowledge of how a coating resists water is helpful in predicting its service life. Failure in a condensation test may be caused by a number of factors including a deficiency in the coating itself, contamination of the substrate, or inadequate surface preparation. The test is therefore useful for evaluating

⁴ *Annual Book of ASTM Standards*, Vols 02.05 and 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 14.02.

⁶ *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

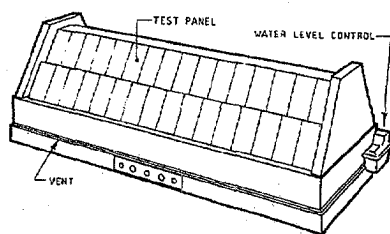


FIG. 1 Controlled Condensation Apparatus

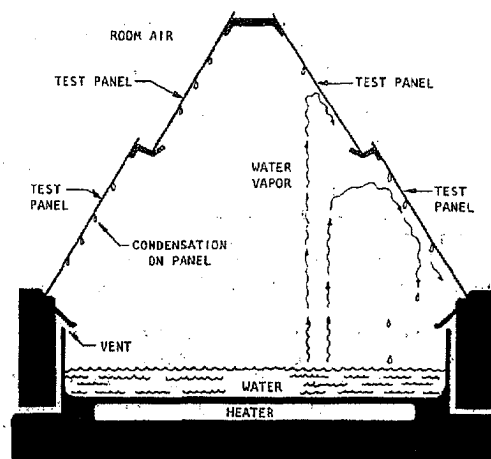


FIG. 2 Apparatus Cross Section

coatings alone or complete coating systems.

4.2 Condensation tests of coatings are used for specification acceptance, quality control, and research and development of coatings and substrate treatments. These tests usually result in a pass or fail determination but the degree of failure also may be measured. A coating system is considered to pass if there is no evidence of water-related failure after a specified period of time.

4.3 Results obtained from the use of condensation tests in accordance with this practice should not be represented as being equivalent to a period of exposure to water in the natural environment, until the degree of quantitative correlation has been established for the coating or coating system.

4.4 The test is usually conducted on metal or wood specimens with the coating facing the inside of the chamber. However, it is possible to test the blister resistance of house paints on wood specimens by mounting the uncoated wood surface facing the inside of the chamber.

4.5 This practice can be used for corrosion tests particularly if the specimens are periodically dried. While corrosion products will drain into the water bath, they are not carried into the vapor that condenses on the test specimens.

5. Apparatus⁷

5.1 *Test Chamber* (see Figs. 1 and 2), consisting of insulated side walls mounted on a base, test specimen racks attached to the side walls, a heated water pan, and provisions for controlling and indicating the vapor temperature within the chamber. Vents, approximately 0.10 to 0.20 in. (3 to 5 mm) wide, shall be provided to admit room air at the bottom of the test chamber.

NOTE 2—The apparatus described in Practice G 53 may be used if the ultraviolet lamps specified in Practice G 53 are turned off.

5.2 Specimens shall form the roof of the test chamber. If the specimens cannot completely fill all the openings, blank panels shall be used. Specimens shall be inclined from 15 to 75° from the horizontal and arranged so that condensate is returned to the water pan without dripping on other specimens.

5.3 *Water Supply*, with water level control.

5.4 *Water Heater*, preferably located under the water pan, controlled by a thermostat with the sensing element located in the water.

5.5 *Thermometer*, with the stem extending into the air-

water vapor mixture in the test chamber.

5.6 *Program Timer, Blower, and Air Heater*, (optional) fitted to the chamber to provide periods of drying on a fixed schedule.

6. Test Specimens

6.1 This practice does not cover the preparation of test specimens. The substrate composition and surface preparation, specimen preparation, and the number of specimens should be agreed upon prior to testing.

NOTE 3—Applicable methods for the preparation of test panels and substrates are given in Methods D 609 and Practices D 1730. Test Methods D 823 cover application techniques for the production of uniform films.

7. Procedure

7.1 Fill the water pan to a depth of approximately 1 in. (25 mm) with water. The quality of the water in the pan does not affect the test since the evaporation and condensation process yields distilled water, but the use of tap water can result in the accumulation of residues in the water pan.

7.2 Fill all spaces in the specimen holder rack with specimens or corrosion-resistant blank panels. Mount coated metal panels with the coating to be tested facing the inside of the chamber. Coated wood specimens may be mounted in the same way.

7.2.1 Blister tests to simulate the effects of water vapor migration from inside a frame house are mounted with the uncoated side of the wood specimen facing the inside of the test chamber.

7.2.2 Seal all cracks between specimens and holes in specimens with tape or metal strips. Condensate may seal small openings but cracks larger than 0.04 in. (1 mm) and holes larger than 0.08 in. (2 mm) in diameter can cause water vapor loss and local temperature variation.

7.3 Adjust the thermostat to maintain the desired temperature of the saturated air and water vapor mixture. Vapor temperatures of 100, 120, or 140°F (38, 49, or 60°C) are suggested. Other temperatures may be used provided that the

⁷ Apparatus from Q-Panel Co., 26200 First St., Cleveland, OH 44145 has been found satisfactory for this purpose. Equivalent apparatus may be used.

temperature is reported in conformance with Section 8. To ensure adequate condensation, maintain a 20°F (11°C) temperature differential between the room and the vapor.

7.4 Operate the chamber continuously unless otherwise specified or agreed. The removal of specimens for inspections during operation is permitted. When removing a specimen for inspection, replace it with a blank so that the test conditions are not altered.

7.5 Cyclic operation with alternating periods of condensation and drying may be used. Automatic drying requires the apparatus described in 5.6. For manual drying of specimens, remove them from the apparatus. Drying periods should be at least 4 h long.

7.6 Conclude the test after a specified period of time or after effects from exposure to water are noted.

7.7 Remove specimens at the conclusion of the test. Do not leave the specimens in the apparatus at the conclusion of the test as the specimens can remain wet for hours, or even days, when the apparatus is turned off.

7.8 Wipe the test specimens dry. Rate specimens for changes in color, blistering, etc. Evaluate specimens no less than 5 min and no more than 10 min after removal from test, as the effects from water exposure can change within a short time. Remove only as many specimens as can be rated within the specified time.

NOTE 4—The 0 to 10 scale described in ASTM STP 500⁸ is preferred for rating. Relevant procedures for evaluating water effects are described in Methods D 610 and D 2616, and Test Methods D 714, D 3359, and D 3363.

7.8.1 If possible, rate the specimens again after they have been removed from the test for a recovery period long enough that moisture absorbed within the specimen dries out and the specimens reach moisture equilibrium with room air. A recovery period from 12 to 24 h is generally sufficient. The post-recovery rating allows evaluation of the permanent effects of the exposure as distinct from the transient effects, and is especially important for evaluation of color and gloss.

8. Report

8.1 Report the following information:

- 8.1.1 Sample identification.
- 8.1.2 Results of the evaluation(s).
- 8.1.3 Reference to Practice D 4585.
- 8.1.4 Hours of test duration.
- 8.1.5 Description of any cyclic operations.
- 8.1.6 Condensation temperature.
- 8.1.7 Special conditions of test or any deviations in test procedure.

⁸ Paint Testing Manual, ASTM STP 500, ASTM, 1972.

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Standard Practice for Conducting Tests on Paint and Related Coatings and Materials Using a Fluorescent UV-Condensation Light- and Water-Exposure Apparatus¹

This standard is issued under the fixed designation D 4587; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1 This practice covers the selection of test conditions in Practice G 53 to be employed for exposure testing of paint and related coatings and materials.

2 *This standard does not purport to address all of the problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

ASTM Standards:

- D 358 Specification for Wood to Be Used As Panels in Weathering Tests of Coatings²
- D 523 Test Method for Specular Gloss²
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces²
- D 659 Method of Evaluating Degree of Chalking of Exterior Paints²
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²
- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints²
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Methods for Measurement of Dry Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²

This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.27 on Accelerated Testing.

Current edition approved Feb. 22, 1991. Published April 1991. Originally published as D 4587 - 86. Last previous edition D 4587 - 86.

¹ Annual Book of ASTM Standards, Vol 06.01.

D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments²

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³

D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁴

D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting⁴

D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting⁴

D 2092 Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting²

D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²

D 2616 Test Method for Evaluation of Visual Color Difference With a Gray Scale⁵

D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films²

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵

G 53 Practice for Operating Light- and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Nonmetallic Materials⁵

3. Significance and Use

3.1 Organic coatings on exterior exposure are subjected to attack by degrading elements of the weather, particularly ultraviolet light, oxygen, and water. This practice may be used for evaluating the behavior of films exposed in apparatus that produces ultraviolet radiation, high temperatures, and water condensation on the films. This apparatus is used to make an early materials comparison of the exterior exposure quality of paints. However, light sources, such as the fluorescent UV lamp, that emit a significant amount of radiation at wavelengths shorter than those in natural sunlight, may cause results that lead to unrealistic evaluations of weathering properties.

3.2 As no single light exposure apparatus, with or without water, can be specified as a direct simulation of natural exposure, this practice does not imply expressly, or otherwise, a specific correlation with outdoor exposure. It has, however, been useful in many instances.

3.3 Since climatic conditions vary with respect to time,

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

geography, and topography, it may be expected that the effects of natural exposure will vary accordingly. All materials are not affected equally by the same environment. Results obtained by use of this practice should not be represented as equivalent to those of any outdoor weathering test unless the degree of quantitative correlation has been established for the material in question.

3.4 Variations in results may be expected when operating conditions among similar type instruments vary within accepted limits of this standard procedure.

4. Test Specimens

4.1 Unless otherwise agreed upon, choose panels that meet the applicable base panel requirements specified in Standards D 358, D 609, D 1730, D 1731, D 1732, or D 2092. Select panel sizes suitable for exhibiting the failure mode to be observed.

4.2 Apply the coatings to flat panels with the base panel material, method of application, coating system, film thickness, and method of drying consistent with the anticipated end use, or as mutually agreed upon between the producer and the user. If it's not possible to test *flat* samples, you may need to take special precautions to ensure that (1) the sample holders seal behind the samples so that the water vapor does not escape from the test chamber, and (2) the closest part of the samples to the UV lamps is at the 50-mm distance specified in Practice G 53. If part of the sample is closer to the lamps, it will be subject to more intense UV exposure.

4.3 Unless otherwise agreed upon, coat test panels in accordance with Test Methods D 823 and measure the film thickness in accordance with an appropriate procedure selected from Test Methods D 1005, D 1186, or D 1400. Nondestructive methods are preferred because panels so measured do not need to be repaired.

4.4 Unless otherwise specified, before exposing coated panels in the apparatus, condition them at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity for one of the following periods in accordance with the type of coating:

Baked coatings	24 h
Radiation-cured coatings	24 h
All other coatings	7 days minimum

5. Apparatus

5.1 *Fluorescent UV/Condensation Apparatus*, complying with Practice G 53.⁶

6. Procedure

6.1 Place panels within the 8.25 by 35.35-in. (210 by 900-mm) area as described in Practice G 53. Reposition the panels on a regular schedule as described in Practice G 53 to minimize any effects from temperature or UV light variation. When the test specimens do not completely fill the racks, fill the empty spaces with blank non-rusting panels to maintain the test conditions within the chamber.

6.2 Use the test conditions specified by mutual consent or required by a product quality specification. Some test conditions in current use for testing paint and related

coatings and materials are:

A = 8 h UV/70°C followed by 4 h CON/50°C for automotive coatings,

B = 4 h UV/60°C followed by 4 h CON/50°C for general metal coatings,

C = 4 h UV/60°C followed by 20 h CON/60°C for exterior wood coatings,

D = 8 h UV/60°C followed by 4 h CON/45°C for industrial maintenance coatings,

E = other test temperatures and time cycles that conform to the Procedure section of Practice G 53.

where:

UV = ultraviolet light (lamps) only, and

CON = condensation conditions only.

NOTE 1—Temperatures are black panel temperatures measured in the panel rack.

6.3 Program the selected test conditions and operate the apparatus continuously within the limits specified in Practice G 53. Service the apparatus in accordance with Practice G 53.

NOTE 2—Variations in results can occur as the result of not changing lamps in accordance with the manufacturer's instructions.

7. Periods of Exposure

7.1 Use one of the following methods to determine the duration of the exposure under this practice:

7.1.1 A mutually agreed upon specified number of total hours.

7.1.2 The number of total hours of exposure required to produce a mutually agreed upon amount of change in either the test specimen or an agreed upon standard sample.

8. Evaluation of Results

8.1 Evaluate conditions of exposed test specimens by means of one or more of the following standards: D 523, D 610, D 659, D 660, D 661, D 662, D 714, D 772, D 1654, D 1729, D 2244, D 2616, D 4214 and E 97. Select methods in accordance with product use requirements.

8.2 Because of possible variations in results as described in 3.4, no reference should be made to results obtained from tests conducted in the apparatus using this practice unless accompanied by Section 9 or unless otherwise specified in a reference procedure.

9. Report

9.1 Report the following information:

9.1.1 Manufacturer and model of fluorescent UV/condensation apparatus.

9.1.2 Manufacturer's designation for the fluorescent UV lamp and the relative spectral energy distribution of the lamp. This may be accomplished by listing the manufacturer's designation, wavelength (nm) where peak emission occurs, and the wavelength near low cut-off where 1 % of peak emission occurs.

9.1.3 Exposure cycle, for example, 4 h UV/60°C, 4 h CON/50°C.

9.1.4 Total exposure time.

9.1.5 Results of panel evaluation (see 8.1).

9.1.6 Identification of standard used for comparative evaluation, if any.

⁶ Apparatus and lamps from Q-Panel Co., 26200 First St., Cleveland, OH 44145 and from Atlas Electric Devices Co., 4114 N. Ravenswood Ave., Chicago, IL 60613, have been found suitable for this purpose.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Guide for Determining the Presence of and Removing Microbial (Fungal or Algal) Growth on Paint and Related Coatings¹

This standard is issued under the fixed designation D 4610; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This guide describes techniques used for determining the presence of fungal or algal growth on paint and related coatings and methods for removal of such growth prior to recoating.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 2022 Method for Sampling and Chemical Analysis of Chlorine-Containing Bleaches²

D 3274 Test Method for Evaluating Degree of Surface Disfigurement of Paint Films by Microbial (Fungal or Algal) Growth or Soil and Dirt Accumulation³

3. Significance and Use

3.1 Microbial growth is a major cause of discoloration and deterioration of paint films. This guide describes techniques used to distinguish fungi and algae from other surface contaminants.

3.2 Repainting a surface contaminated with fungi or algae generally causes more rapid infestation of the new paint than repainting a surface from which fungal or algal growth has been removed. This guide describes methods for removing fungal or algal growth prior to repainting.

4. Reagents

4.1 *Sodium Hypochlorite*, approximately 5 % aqueous (NaOCl) as commercial household bleach. Because sodium hypochlorite decomposes on exposure to heat and sunlight and becomes ineffective, use only fresh material. See Method D 2022 for methods of sampling and chemical analysis.

4.2 *Trisodium Phosphate*, or non-ammonia-containing laundry detergent.

5. Procedures

5.1 *Determining the Presence of Fungal or Algal Growth on Paint:*

5.1.1 *Chemical*—Apply a drop of 5 % aqueous sodium hypochlorite solution (common household bleach) to the area suspected of being contaminated with fungal or algal growth. Fungal or algal discoloration will normally bleach within 60 s. Discoloration that does not bleach is probably dirt. For further confirmation do visual and subculture tests.

5.1.2 The following procedures should preferably be used by persons who have had basic microbiological training:

5.1.2.1 *Visual*—Examine the surface using magnification from 10 to 100 $\times$ to distinguish among fungal, algal, or dirt disfigurement in accordance with Test Method D 3274.

5.1.2.2 *Subculture*—Apply a prepared petri dish containing a raised convex surface of nutrient agar culture medium⁴ directly to the surface to be sampled and exert moderate pressure. Replace the cover and incubate for at least 72 h at 95°F (35°C). Examine the agar surface visually as in 5.1.2.1.

NOTE 1—The culture medium must contain the nutrients necessary for growth of algae and fungi.

5.2 *Removal of Fungal and Algal Growth on Paint:*

5.2.1 Wash the surface with a solution of approximately 5 % trisodium phosphate in water or a solution at approximately 2 % laundry detergent in water.

5.2.2 Thoroughly rinse the surface with water to remove residual trisodium phosphate or detergent.

5.2.3 Wash the surface with a solution of 1 part by volume of sodium hypochlorite 5 % aqueous solution and 3 parts by volume of water.

NOTE 2—The solution should be allowed to remain on the surface from 10 to 15 min.

NOTE 3—The sodium hypochlorite solution and the 2 % laundry detergent solution may be combined 1:1 by volume in one treatment if desired.

5.2.4 Thoroughly rinse the surface with water to remove residual sodium hypochlorite.

5.2.5 If agreed upon by the parties involved, reinspect the surface in accordance with 5.1 to ensure that there has been adequate removal. Refer to the coating manufacturer's literature for recommended drying time before recoating.

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.28 on Biodeterioration.

Current edition approved Aug. 29, 1986. Published October 1986.

² Annual Book of ASTM Standards, Vol 15.04.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Agar available from BBL Microbiology Systems, Div. of Becton, Dickinson and Co., P.O. Box 243, Cockeysville, MD 21030 or Scott Laboratories, Inc., Fiskeville, RI 02823, has been found suitable for this purpose.

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Standard Specification for Design and Fabrication of Flue Gas Desulfurization System Components for Protective Lining Application¹

This standard is issued under the fixed designation D 4618; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the design and fabrication of metal components for Flue Gas Desulfurization (FGD) equipment, including absorbers, tanks, chimney liners, ductwork and associated equipment that are to be lined for corrosion or abrasion resistance, or both.

1.2 *Limitations*—This specification does not cover structural performance of FGD components. It is intended only to define the design considerations for successful application and performance of protective linings for FGD system components.

1.3 This specification represents the minimum requirements for lining work. In cases where the manufacturer's instructions and recommendations differ from this specification, these differences must be resolved before fabrication is started.

1.4 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Design/Engineering Requirements

2.1 Rigidity:

2.1.1 The components shall be designed so that the interior metal surfaces are sufficiently rigid for the intended lining materials. Manufacturer's recommendations for maximum strains or deflection limits for the lining material shall be followed.

2.1.2 The weight of the lining system shall be considered in the structural design of the component.

2.1.3 The design shall consider the effects of pressure, wind, seismic and other design loads.

2.1.4 Vibration may cause flexing or high surface strains on the lining. This is of particular concern to rigid lining materials and shall be minimized.

2.1.5 Special consideration shall be given to all areas of potentially excessive strain such as unsupported bottom areas, oil-canning, out of roundness, sidewall-to-bottom joints, etc.

2.1.5.1 Where a component is on a concrete foundation, grouting shall be done if necessary to correct unsupported bottom areas.

2.1.5.2 Sand fill shall not be used for bottom support unless provisions are made to assure that the sand cannot be lost due to erosion.

2.2 Accessibility:

2.2.1 All interior surfaces of the components shall be designed to be readily accessible for surface preparation and lining application.

2.2.1.1 All fillets and corners shall be accessible for proper welding and grinding.

2.2.2 The minimum manway size for a working entrance during lining application shall be 36 in. (900 mm) in diameter or 24 in. (600 mm) width by 36 in. (900 mm) height.

2.2.2.1 Closed components shall have a minimum of two manways, one near the top and one near the bottom, preferably located 180° apart to facilitate adequate ventilation for workers.

2.2.2.2 Additional or larger openings may be required to facilitate ventilation and material handling. The lining material applicator should be consulted for specific requirements.

2.3 Shell Penetrations:

2.3.1 All connections or openings in the vessel or component shall be flush with the interior wall. Inlet nozzle shall extend into vessels if incoming fluids can be detrimental to lining materials. Any exterior or interior connection shall be flanged in order to facilitate linings.

2.3.2 The maximum length of flanged nozzles, 4 in. (100 mm) and greater in diameter, shall not exceed the dimensions in Table 1.

2.3.2.1 Only 4 in. (100 mm) diameter and larger nozzles shall be used for maximum reliability of the lining system.

2.3.2.2 As an alternative to lined nozzles, compatible prefabricated, reinforced plastic, ceramic or alloy metal inserts (sleeves) may be used if they offer superior corrosion and abrasion protection. Lining shall overlap onto prefabricated liners.

TABLE 1 Maximum Length of Nozzles

Nominal Nozzle Size, in. (mm)	Maximum Nozzle Length— Shell to Face of Flange, in. (mm)
4 (100)	8 (200)
6 (150)	12 (300)
8-24 (200-600)	16 (400)
24-36 (600-900)	24 (600)
Over 36 (900)	any length

¹ This specification is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.09 on Protective Linings for FGD Systems.

Current edition approved Feb. 20, 1987. Published April 1987.

2.3.3 Lining thickness may dictate changes in nozzle dimensions to achieve design flow rates.

2.4 Appurtenances Inside Components:

2.4.1 The requirements in Sections 2 and 3 apply to any appurtenances that are being lined and installed inside a lined component, such as agitators, anti-swirl baffles, gaging devices, internal piping, ladders, and support brackets.

2.4.2 If appurtenances inside the component cannot be lined, they shall be made of corrosion-resistant materials. If alloys are used, the lining shall carry over the welded area onto the alloy a minimum of 3 in. (76 mm). Some linings may require special designs to protect the edge of the lining. Bolted connections are used, dielectric insulation shall be provided.

2.4.3 Heating elements shall be attached with a minimum clearance of 6 in. (150 mm) from the surface of the lined component. Greater clearance may be required to protect the lining from excessive temperature conditions depending on

the temperature of the element.

2.4.4 Special precautions shall be taken in lined components where severe abrasion/impingement damage may occur. Precautionary design measures, such as wear plates, brick liners or added coating thickness, shall be considered when necessary.

2.5 Structural Reinforcement Members and Supports:

2.5.1 Structural reinforcement members (stiffeners) shall be installed on the vessel exterior, wherever necessary. However, if such members are installed internally they shall be fabricated of simple closed shapes such as round bars, pipe, or box beams for ease of applying the lining material.

2.5.2 The use of box beams or pipe for internal supports is recommended. The use of angles, channels, I-beams and other complex shapes shall be avoided wherever possible. If they must be installed internally, these members shall be fully seal welded and the edges ground to a 1/8 in. (3 mm) minimum radius.

2.5.3 If closed chambers are formed with internal box beams or pipes, they shall be vented to the atmosphere at the lowest point, so that pressures are not developed during operation and possible curing procedures and so that corrosion, due to localized lining failures, can be observed early.

3. Fabrication

3.1 Welds:

3.1.1 All internal welds to be lined shall be continuous without imperfections such as weld slag, weld splatter, rough surfaces, undercutting, high peaks, porosity, sharp corners, sharp edges and inadequate thickness (see Fig. 1).

3.1.2 The degree of weld preparation prior to lining depends on the type of lining to be applied. The lining manufacturer must be consulted for specific requirements for weld preparation during the design of the component and prior to start of fabrication.

3.1.3 Use of weld display samples before and after grinding may be of help to the component fabricator in supplying acceptable welds with a minimum required rework. All welds shall be inspected prior to lining. Whenever possible, shop welds shall be inspected in the fabricator's shop.

3.1.3.1 All weld areas shall be inspected before and after blast cleaning. Pinholes, pits, blind holes, porosity, undercutting or similar depressions are not permissible in the finished surface.

3.1.4 Weld splatter shall be removed. Chipping may be utilized only if followed by grinding for the required surface finish.

3.1.4.1 The use of non-silicone, anti-splatter coating applied adjacent to weld areas is suggested. This coating shall be of a type that can be removed by the final blast cleaning.

3.1.5 After inspection, all undercuts and pinholes shall be eliminated by welding or grinding. All rough welds shall be ground to remove sharp edges. Chipping may be used to remove sharp edges if followed by grinding.

3.1.6 All edges and similar abrupt contours shall be rounded off by grinding or machining to a 1/8-in. (3 mm) minimum radius.

3.1.7 Fillets and changes in contour shall be ground to a 1/8-in. (3-mm) minimum radius where required for the selected lining material. Any grinding done on welds, edges,

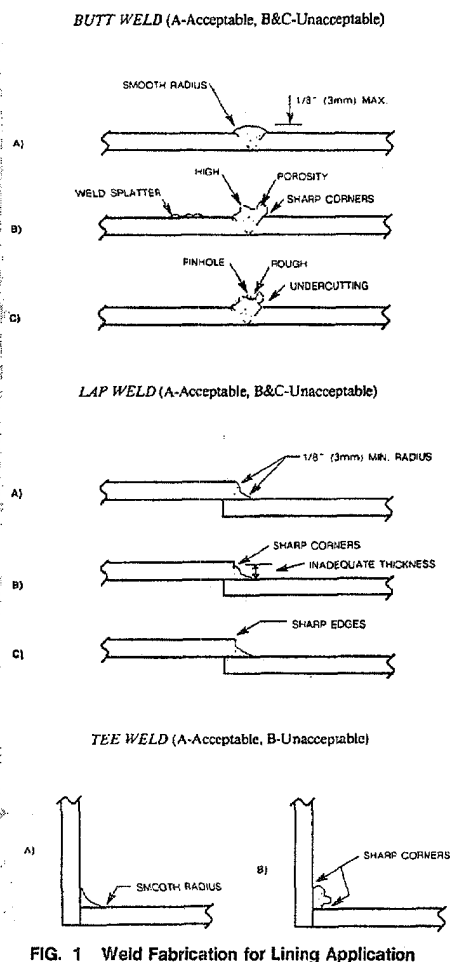


FIG. 1 Weld Fabrication for Lining Application

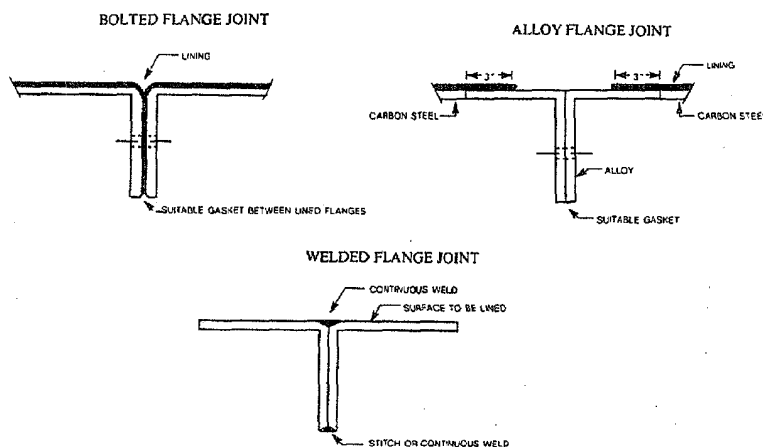


FIG. 2 Joint Fabrication for Lining Application

and fillets shall be done carefully to eliminate potential problems caused by gouging of the parent metal.

3.1.8 All internal and external welding shall be completed prior to any lining application.

3.2 Joints:

3.2.1 All welds shall be continuous. Intermittent or spot welding is not permitted (see Fig. 2).

3.2.2 Riveted joints shall not be used. Internal bolted joints shall not be used except to avoid welding on an already lined surface. In this case, corrosion resistant alloy or nonmetallic bolts shall be used.

3.2.3 If bolts are used to facilitate installation or welding, or both, of a component, they shall be removed and holes plug welded before lining application.

3.2.4 Lap welded joints shall be avoided whenever possible. Where they are necessary, the interior lap shall be a full fillet weld and finished as in accordance with 3.1.6.

3.2.5 Expansion joints and bolted flanged duct or shell joints require special lining consideration. Bolted flange joint surfaces shall be lined before assembly. Special consideration shall be given during erection and fit-up so as not to damage the lining.

3.2.5.1 If alloy flanges are used at expansion joints, the design must allow for the lining system to be applied over the alloy by 3 in. (75 mm). Some linings may require special designs to protect the leading edge.

3.3 During and after the lining of the equipment, no welding shall be allowed on the interior or exterior surfaces.

3.4 Signs shall be hung or stenciled on the exterior surface of the equipment designating the following: LINED EQUIPMENT, DO NOT BURN OR WELD. They shall be visible from all elevations and sides of the equipment.

4. Drawing Notes

4.1 All lined surfaces should be clearly identified on all detail and arrangement drawings.

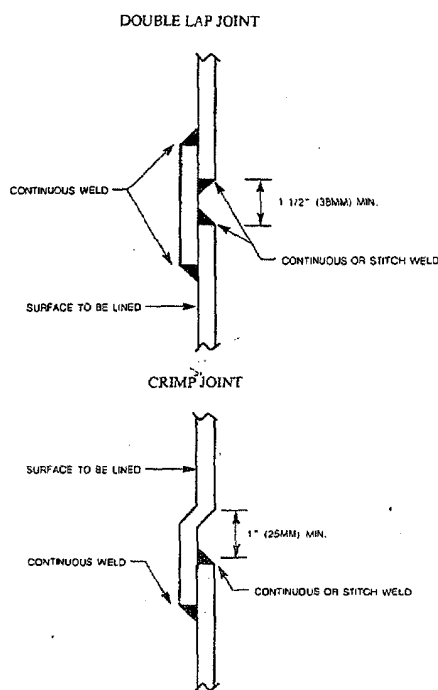


FIG. 2 (Continued) Joint Fabrication for Lining Application

4.2 The following note shall appear on appropriate detail and arrangement drawings: All surfaces to be lined shall meet the requirements of ASTM Specification D 4618.

CORNER JOINTS

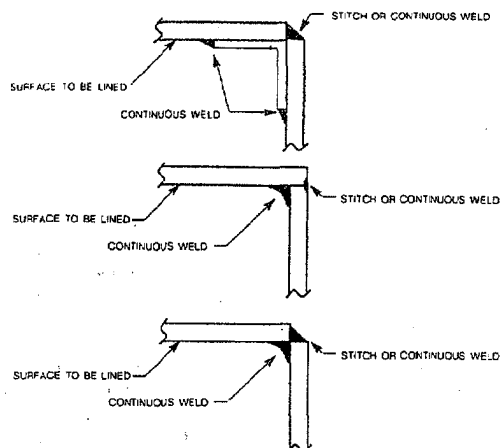


FIG. 2 (Continued) Joint Fabrication for Lining Application

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Standard Practice for Inspection of Linings in Operating Flue Gas Desulfurization Systems¹

This standard is issued under the fixed designation D 4619; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice describes procedures for conducting inspections of the conditions of various linings in operating Flue Gas Desulfurization (FGD) system components.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 7.

2. Significance and Use

2.1 Periodic inspections are essential to evaluate lining performance, to detect existing damage potential problems, and to plan scheduled maintenance. The frequency of these inspections may diminish or increase with time depending upon lining performance.

3. Recordkeeping

3.1 Lining condition will depend on the operating conditions experienced by the lining systems. Records of these conditions that are maintained by the owner/operator should be evaluated for potential effects upon the linings. These may include:

- 3.1.1 Dates of lining installation and initial operation,
- 3.1.2 Solution/gas temperatures in lined components,
- 3.1.3 Solution/gas chemistry (pH, composition),
- 3.1.4 Start up/shut down dates,
- 3.1.5 Gas velocities and particulate loading, and
- 3.1.6 Ambient conditions.

3.2 Any known change in the process criteria or modifications of the physical design shall be identified and dated.

3.3 All past history pertaining to the lining systems should be available during the inspection process. They may include:

- 3.3.1 Copies of existing lining specifications and installation procedures.
- 3.3.2 Quality control documents of the existing lining installation.
- 3.3.3 Copies of previous inspection reports.
- 3.3.4 Documentation pertaining to any maintenance of existing lining systems.

4. Inspection Team

4.1 The owner/operator should select a team of experienced personnel to conduct the inspection. Personnel representing the following may be included:

- 4.1.1 Owner's representative,
- 4.1.2 Lining manufacturer,
- 4.1.3 Lining applicator,
- 4.1.4 Equipment designer,
- 4.1.5 Architect engineer,
- 4.1.6 Third party inspectors, and
- 4.1.7 System designer.

5. Pre-Inspection Procedure

5.1 Prior to conducting an inspection of the lining, the owner/operator shall ensure that the following services and equipment are provided.

5.1.1 *Safety*—The inspection team shall verify that the equipment being inspected has been made safe for entry. This shall include lockout procedures for related equipment such as, but not limited to, the boiler, dampers, valves, fans, and pumps.

5.1.2 *Lighting*—Sufficient lighting shall be provided to assure general lighting of the overall area plus localized high intensity lights for close visual observation or taking of photographs, or both. The lighting fixtures shall be equipped with a safety guard to minimize breakage and injury.

5.1.3 *Access to Lining Surfaces*—The access equipment must meet all safety requirements of OSHA and the owner/operator. The equipment must be capable of placing the inspectors close enough to the lining surface to perform all inspection procedures.

5.1.4 *Cleaning*—Selected lining surfaces to be inspected shall be cleaned of any deposits or buildup that will obscure examination of the lining. The cleaning procedure selected must not cause damage to the lining.

5.1.5 *Ventilation*—Provisions must be made to assure that adequate fresh air is provided in all FGD components being inspected. If some components are on line, provisions must be taken to adequately isolate such components.

6. Hazards

6.1 All safety requirements of OSHA and the owner/operator, must be met when performing all inspection operations. Residues, including acids, heavy metals, or other hazardous materials, may be present in deposits, on the lining surfaces, or in the atmosphere. Precautions shall be taken to protect personnel. Confined entry safety requirements shall be adhered to where applicable.

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.09 on Protective Linings for FGD Systems.

Current edition approved Aug. 15, 1991. Published October 1991. Originally published as D 4619 - 86. Last previous edition D 4619 - 86.

Inspection Procedures

7.1 The inspection should include visual examination, photographic examination, mapping of potential problem areas, specific destructive or nondestructive testing, and removal (if required) of representative samples for analysis (see Table 1).

7.2 Temperature may influence observed or tested lining parameters, such as crack width, hardness, and adhesion. During inspection, temperature of the lining surface and the interior and exterior ambient temperatures of the component should be measured and recorded.

7.3 Other parameters important to the inspection should be discussed with the parties involved and agreed to prior to the inspection.

Report

8.1 The owner/operator shall designate who is responsible for the preparation of an inspection report.

8.2 Report the following information:

8.2.1 Pertinent background information contained in Section 3,

8.2.2 Date(s) of inspection,

8.2.3 Participants and their affiliation,

8.2.4 Documentation of inspection,

8.2.5 Photographs, as applicable,

8.2.6 Mapping of problem areas,

8.2.7 Test results, and

8.2.8 Conclusions and recommendations.

9. Repairs

9.1 If repairs of the lining are required, the owner/operator or his representative shall prepare specific repair specifications or procedures, or both, with the input of the lining manufacturer(s), the applicator(s), and others as necessary.

9.2 These specifications or procedures, or both, may be prepared in advance or as a result of the inspection.

10. Keywords

10.1 cementitious linings; chemical resistant linings; flue gas desulfurization (FGD); inorganic linings; inspection of linings; organic linings; power generation facility linings

TABLE 1 Lining Maintenance Inspection Parameters

Organic Resins	Organic Elastomers	Inorganic/Cementitious	Inorganic/Masonry
Visual Inspection: Rust Chemical Degradation Surface Effects: Abrasion/erosion Alligatoring/checking Discoloration/charring Flaking Cracking Softening Blistering/delamination: Size Density Entrapped contents for analysis Mechanical Damage Physical Testing: Lining thickness Continuity testing of suspect areas, (confirm test voltage with lining manufacturer) Adhesion testing, if required	Visual Inspection: Rust Chemical Degradation Surface Effects: Abrasion/erosion Swelling Softening Crazing Cracking Surface sloughing Blistering: Size Density Entrapped contents for analysis Mechanical Damage: Gouging Cutting Tearing Overcompression (mating surfaces) Adhesive failure Cohesive failures Reversion (Reverting to soft conditions with loss of physical properties) Physical Testing: Lining thickness Continuity testing of suspect areas, (confirm test voltage with lining manufacturer) Adhesion testing, if required Shore durometer hardness	Visual Inspection: Erosion/mechanical damage Cracking Softening Spalling Rust staining Efflorescence Anchor exposure Delaminations Substrate exposure Physical Testing: Hardness (Schmidt hammer) Measure crack widths and pattern Core samples, if required Remove cementitious layer to permit visual examination of underlying membrane/substrate	Visual Inspection: Erosion/mechanical damage Cracking: Mortar joint Brick face Spalling Softening: Brick Mortar Expansion joint Color Change: Brick Mortar Physical Testing: Hardness (Schmidt hammer) Core samples, if required Petrographic analysis, if required

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Measuring Paint Spatter Resistance to Roller Application¹

This standard is issued under the fixed designation D 4707; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the tendency of a paint to spatter when applied with a paint roller to a substrate.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials²

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²

2.2 Other Document:

Photographic Standards of Paint Roller Spatter³

3. Summary of Test Method

3.1 The test paint is applied to a black plastic panel by draw-down. The coated plastic panel is immediately mounted on an essentially vertical surface above a sheet of paper used to catch any spatter. A specially designed notched spool roller (see 5.1) is rolled through the film following a defined procedure, tending to generate spatter. Any spatter falls upon the spatter catch paper, and after drying is rated against the photographic standards.

4. Significance and Use

4.1 Paint spatter generated by roller application is dependent on the properties of both the paint being applied and the paint roller cover used for the application. To eliminate the influence of the paint roller cover as a variable, and thus restrict the spatter-inducing variable to the paint under test only, the paint roller cover is replaced by a standard notched spool roller to generate spatter by a mechanism that simulates that of a paint-applying roller cover.

4.2 Although most of the development work to establish this test method was undertaken using latex paints, sufficient

work was also done to show its applicability to solvent-reducible paints.

4.3 Tests during the development of this test method showed that the spattering properties of paints, like other physical properties, may in time change. Therefore, the results of this test are valid only for the time when the test is run.

5. Apparatus

5.1 *Notched Spool Test Roller* (Fig. 1).⁴

5.2 *Glass Plate*, at least 9 by 19 in. (230 by 485 mm) by $\frac{1}{8}$ in. (6 mm) thick.

5.3 *U-shaped Film Caster*,⁵ having a 7-mil (175- μ m) clearance by $5\frac{1}{4}$ in. (135 mm) wide.

5.4 *Mechanical Metronome*, with swinging arm.

6. Materials

6.1 *Black Plastic Panels*,⁶ $6\frac{1}{2}$ by 17 in. (165 by 430 mm).

6.2 *Flannel Cloth*.

6.3 *Masking Tape*, 1 in. (25 mm) wide.

6.4 *Spatter Catch Paper*, 18 by 18 in. (455 by 455 mm).

This paper can be any convenient paper to which the spatter droplets will adhere and of a color to contrast with the color of the paint under test.

7. Sampling and Conditioning

7.1 Sample the material in accordance with Practice D 3925.

7.2 Prior to testing, the samples shall be conditioned in accordance with the standard atmosphere described in Specification D 3924. The testing shall take place under the same conditions.

8. Procedure

8.1 Clean the top of the glass plate and both sides of the black plastic panel to ensure that they are free of specks. Place the black plastic panel on the glass plate and tape the narrow end at the top to the glass plate. Smooth the panel along the plate to ensure a close fit.

8.2 Stir the paint under test thoroughly and strain to remove all skins and particles. Place the film caster with the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved May 29, 1987. Published July 1987.

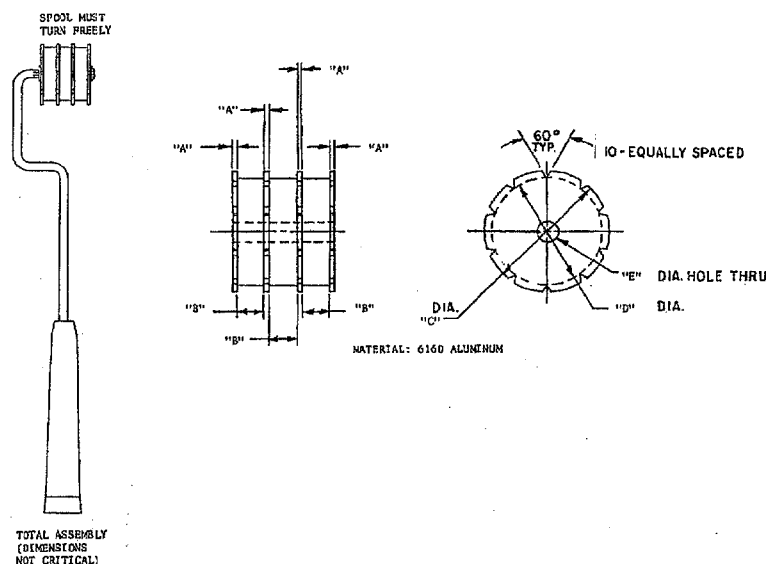
² *Annual Book of ASTM Standards*, Vol 06.01.

³ Copies of the pictorial photographic standards are contained in *Pictorial Standards of Coatings Defects* and may be obtained from the Federation of Societies For Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

⁴ The E-Z Paint Notched Spool Test Roller, obtainable from the Precision Gage and Tool Co., 28 Volkenand Ave., Dayton, OH 45410, has been found suitable for this purpose.

⁵ The Dow film caster, available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910, has been found suitable for this purpose.

⁶ Leneta P-121-10N dull black plastic panels $6\frac{1}{2}$ by 17 in. by 10 mil (production tolerance: ± 1 mil) (165 by 432 by 250 μ m) in size, obtainable from the Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ 07423, are suitable for the purpose.



Dimensions

		in.	mm
A	Flange thickness	0.063 ± 0.005	1.60 ± 0.13
B	Space between flanges	0.343 ± 0.005	8.71 ± 0.13
C	Total diameter	1.600 ± 0.015	40.16 ± 0.38
D	Diameter under notches	1.400 ± 0.015	35.56 ± 0.38
E	Hole diameter	0.281 ± 0.005	7.14 ± 0.13

FIG. 1 Notched Spool Roller

0-mil (175- μ m) side down immediately below the taped portion of the black plastic panel. Turn on the metronome, adjusted for 80 beats/min. Into the well formed by the film caster, pour sufficient paint so that the well is filled from corner to corner. Draw down the paint the full length of the black plastic panel, until the film caster is fully beyond the far edge. The rate of application should be fairly slow, 3 to 4 s from end to end, to prevent pinholes or holidays in the paint film.

8.3 Immediately upon completion of the draw-down, remove the black plastic panel from the glass plate, keeping the masking tape intact, and tape, with the long direction vertical, to a surface that is vertical or nearly vertical (within 5° of vertical, top sloping away from the operator) with the bottom of the black plastic panel about 1 in. (25 mm) above the laboratory bench or table (Fig. 2). It is desirable that the surface under the black plastic panel be firm but with a little resilience. A backing of 1/4-in. (6-mm) thick pasted fiberboard⁷ is ideal as very hard surfaces make proper performance of the test difficult. Center the spatter catch paper on the laboratory bench or table under the black plastic panel.

⁷ Upson or universal board, manufactured by Domtar Gypsum Co., P. O. Box 508, Lockport, NY 14094, and available at most lumberyards, has been found suitable for this purpose.

8.4 Using the clean, notched spool roller, start in one of the upper corners and roll downward and upward through the paint film. Always keep the notched spool in contact with the black plastic panel, not removing the roller from the film when changing directions. Make ten passes in each direction (20 passes total), $15 \pm 1/2$ in. (380 ± 15 mm) per pass, progressively moving sideways from one edge of the film to the other (Fig. 3). Each pass should coincide with a beat of the metronome and the motion should be continuous, not jerky. Try to emulate the motion of the metronome, using the metronome for accurate timing (practice the motion with the metronome to gain the necessary rhythm and timing), since the speed of the rolling of the notched spool roller through the paint film has been found to be the only variable significantly affecting the results of the test. Make sure that all four flanges of the notched spool roller are within the draw-down area at all times. Use sufficient pressure to maintain constant contact between the notched spool roller and the black plastic panel. Note that as long as constant contact is maintained, pressure variations have not been found to noticeably affect the results.

8.5 Remove the spatter catch paper and lay it in a suitable place to dry.

8.6 Repeat the test with a second plastic panel after cleaning the apparatus.

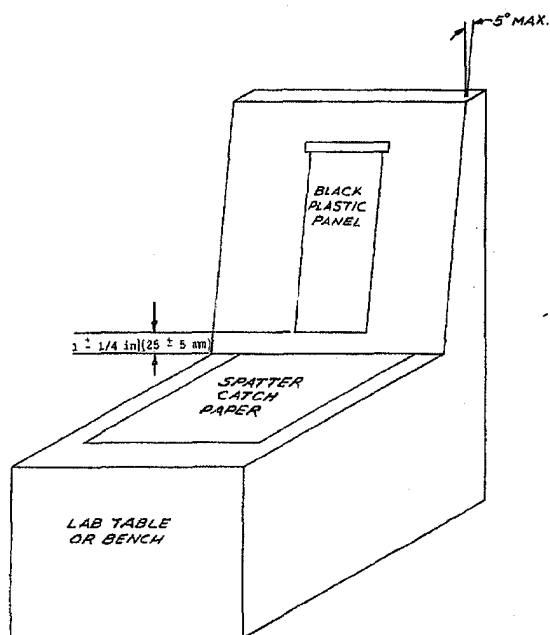


FIG. 2 Test Set-Up Schematic

9. Grading Procedure

9.1 Carefully cut an area $7\frac{1}{2}$ by $9\frac{1}{2}$ in. (190 by 240 mm) out of the front edge (edge nearest the paint) of the spatter catch paper. The $7\frac{1}{2}$ -in. side will be the front edge. Center the area with respect to the spatter so a maximum of spatter is on this sheet (Fig. 4).

9.2 Using the photographic standards numbered 1, 3, 5, 7, and 9 for comparison, rate each test with a number from 0 (low spatter resistance) to 10 (no spatter), interpolating as needed for ratings 2, 4, 6, and 8.

NOTE—Figures 5 through 9 are duplications thereof for sample purposes, not meant to be used for test grading purposes.

9.3 In rating the paints using the photographic standards, consider the population of spatter droplets of greater importance than the average size of the spatter droplets.

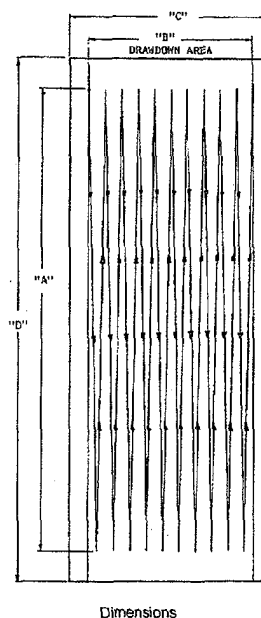
9.4 If the ratings of two tests do not agree within 1 unit, repeat until such precision is obtained.

10. Precision and Bias

10.1 A precision and bias statement is in development.

11. Keywords

11.1 paint spatter resistance; resistance—paint spatter; roller application; spattering



Dimensions

		in.	mm.
A	Notched spool pass length	$15 \pm \frac{1}{2}$	380 ± 15
B	Drawdown width (determined by blade)	$5\frac{1}{4}$	140
C	Panel width	$6\frac{1}{2}$	165
D	Panel length	17	430

FIG. 3 Test Pattern Schematic

FRONT (Edge nearest to the mounted panel during test.)

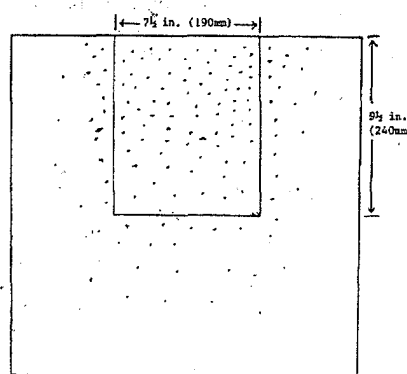
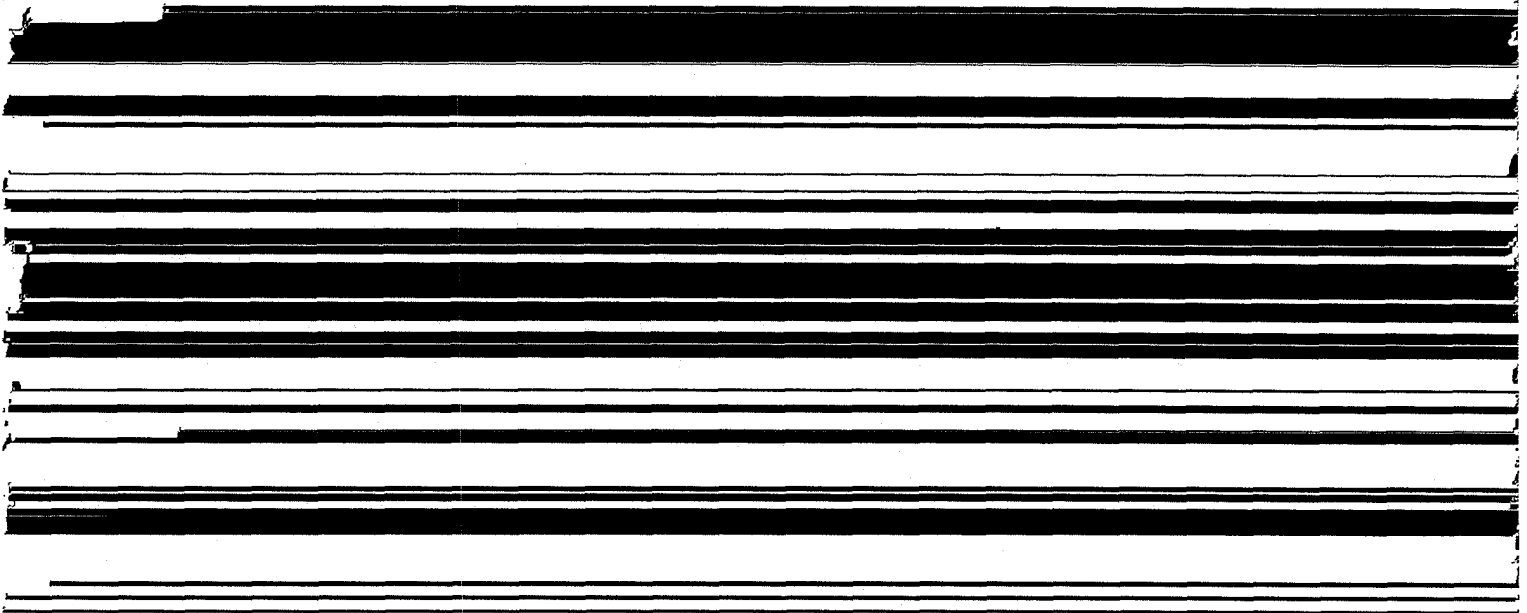
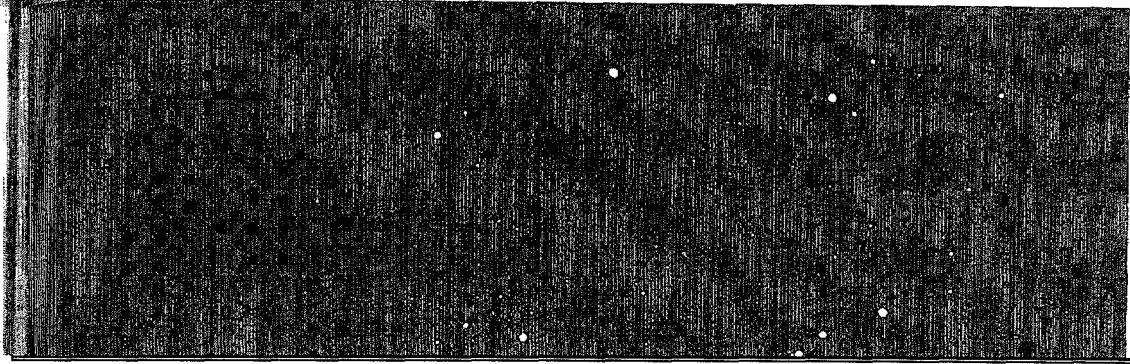
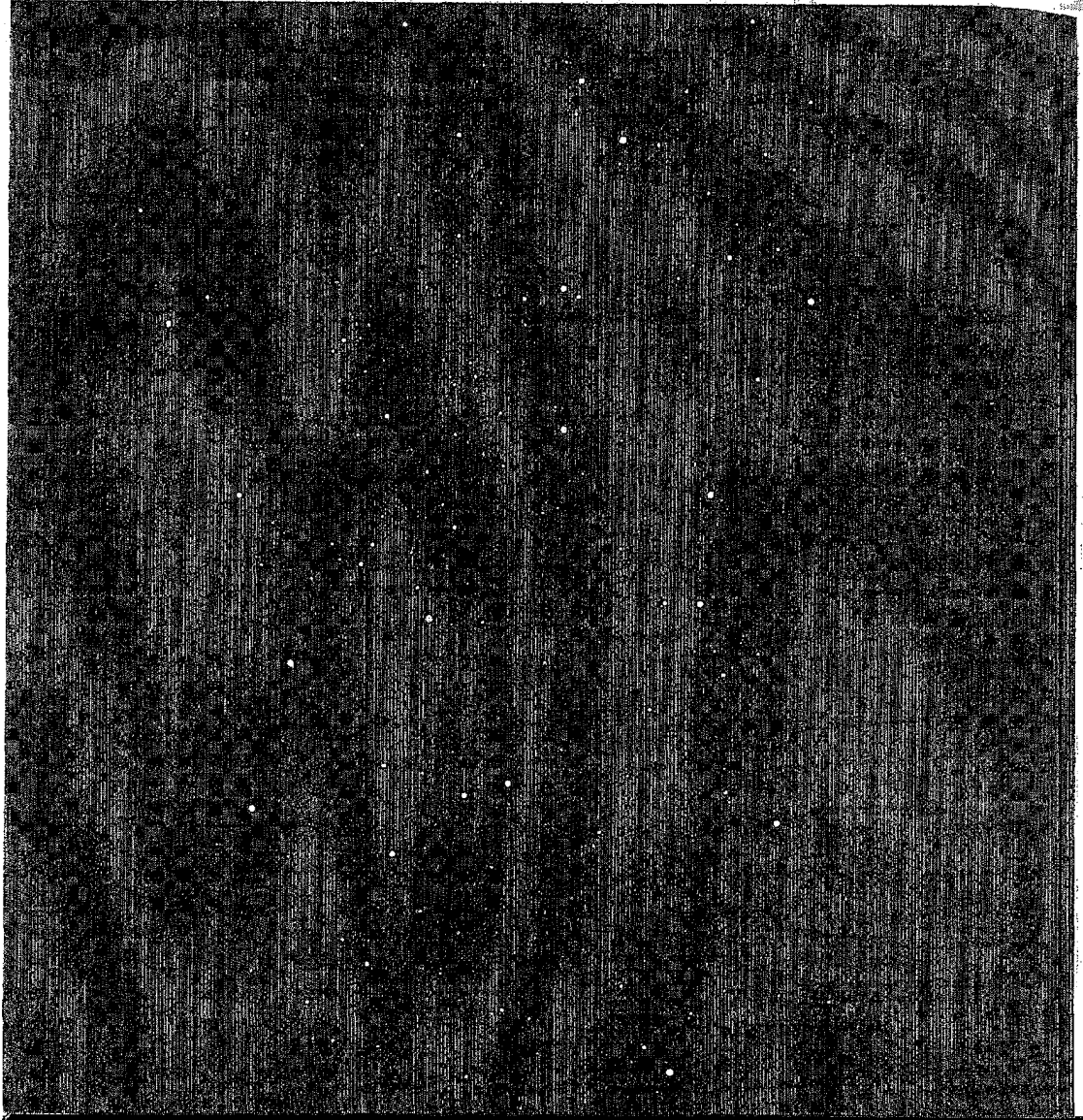


FIG. 4 Cutting Out Section of Spatter Catch paper for Evaluation

D 4707

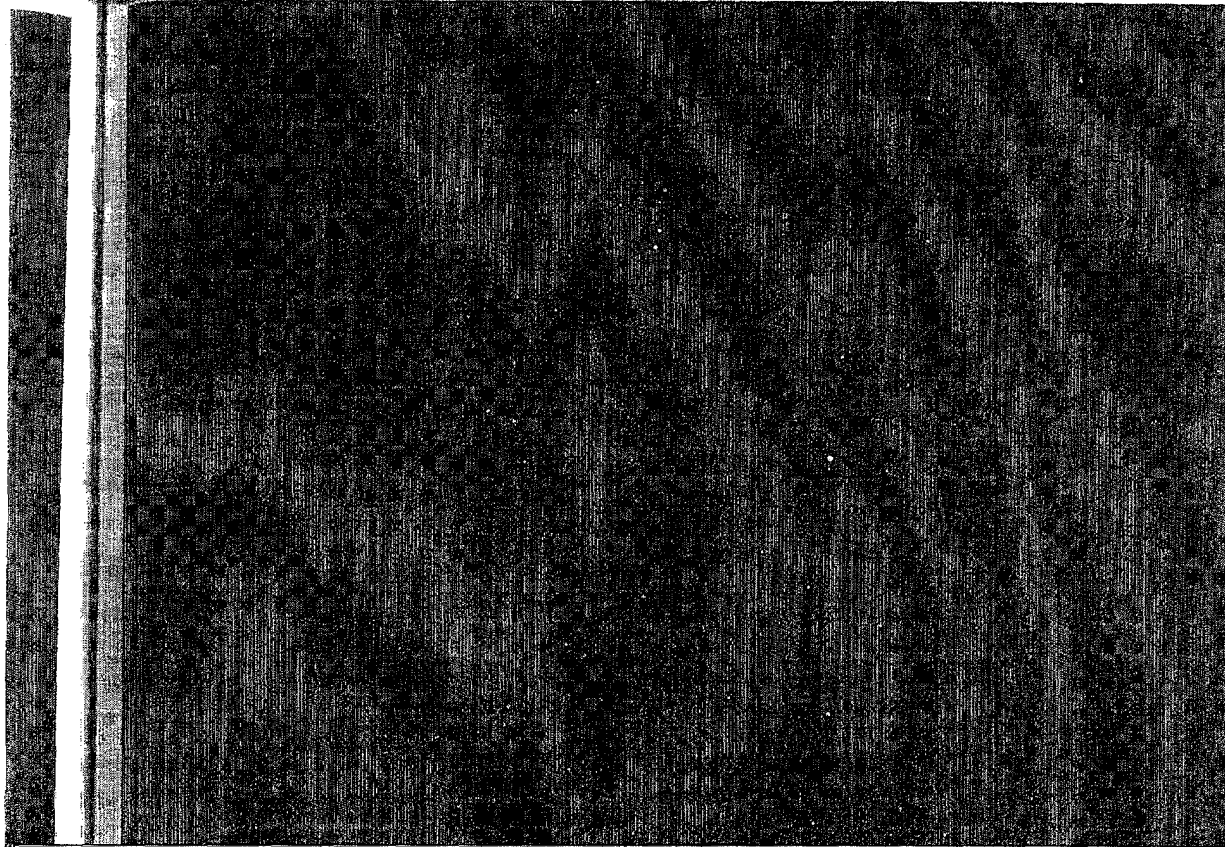


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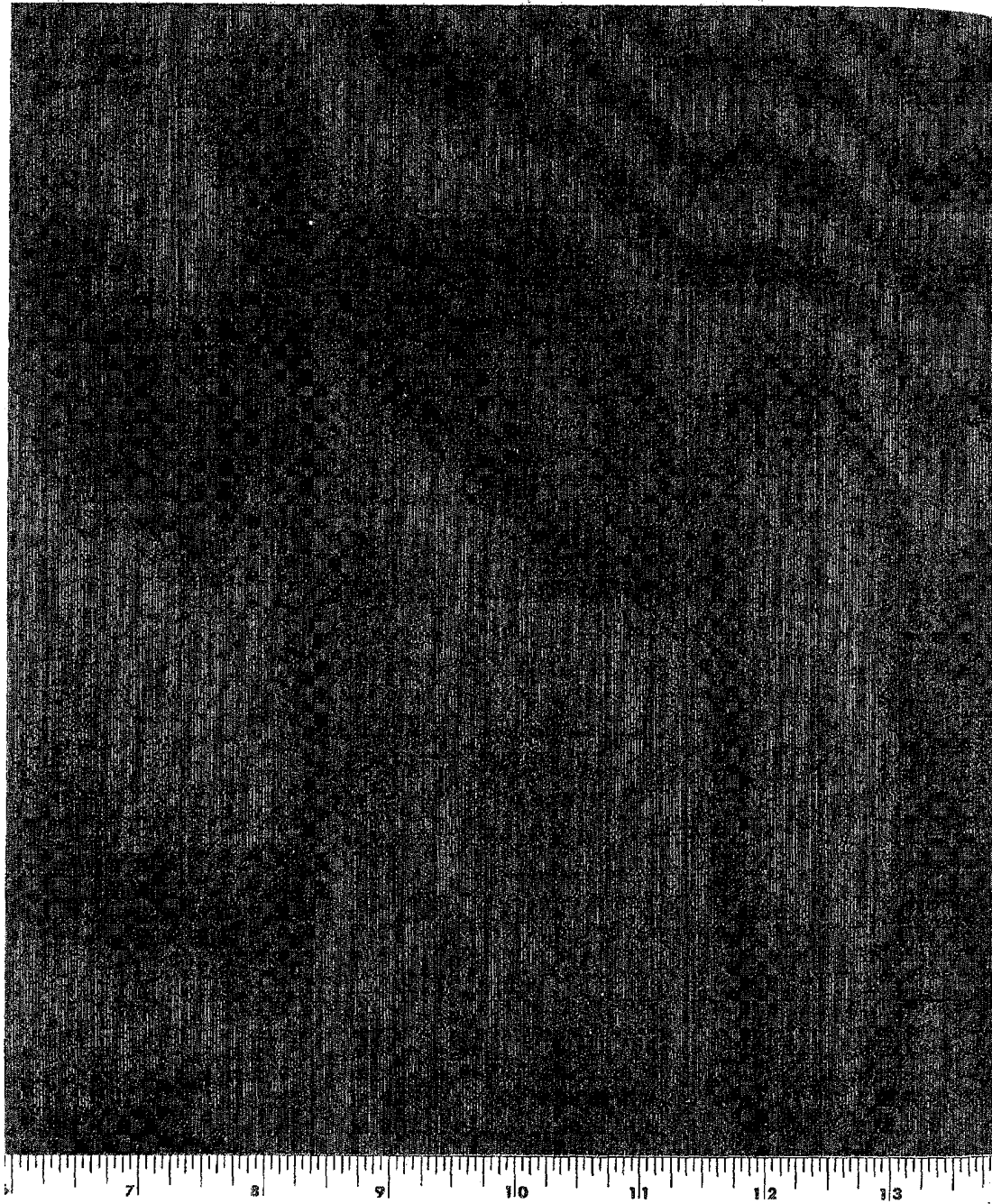


FIG. 8 Paint Spatter Resistance to Roller Application Rating Standard No. 7

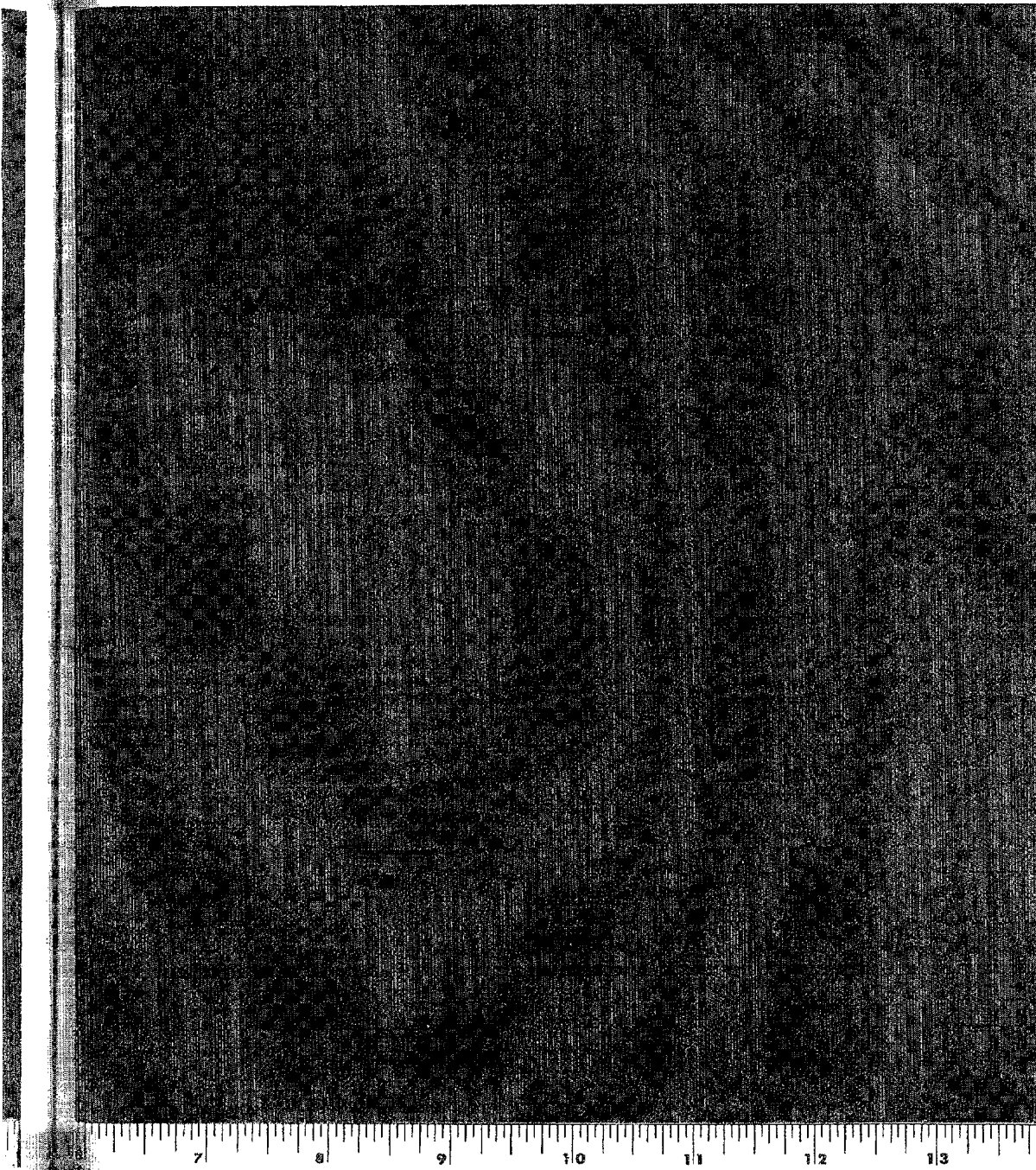


FIG. 9 Paint Spatter Resistance to Roller Application Rating Standard No. 9

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Standard Practice for Preparation of Uniform Free Films of Organic Coatings¹

This standard is issued under the fixed designation D 4708; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the preparation of free films of organic coatings for use in determining the physical properties of the coatings. Procedures are given for preparing free films on four alternative substrates. These substrates are dental foil, treated FEP (fluorinated ethylene-propylene) sheet, silicone coated paper, and halo-silane coated glass plates.

1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific hazard statement is given in 6.1.

2. Referenced Documents

2.1 ASTM Standards:

- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1653 Test Methods for Water Vapor Permeability of Organic Coating Films²
- D 2370 Test Method for Tensile Properties of Organic Coatings²

3. Summary of Test Method

3.1 Free films are prepared by depositing a uniform wet coating of the test material on a release substrate. The applied films are dried or baked, cut into appropriate size for the intended physical property test, and then stripped from the release substrate.

4. Significance and Use

4.1 Free films are required for conducting tests to evaluate the tensile properties (Test Method D 2370) and moisture vapor permeability (Test Methods D 1653) of organic coatings.

4.2 The tin foil/mercury amalgamation procedure should be used only in cases where FEP and paper substrates cannot accommodate wetting or high-temperature baking conditions.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 4708 - 87. Last previous edition D 4708 - 87.

² Annual Book of ASTM Standards, Vol. 06.01.

5. Apparatus

5.1 *Equipment*, for applying films of uniform thickness as described in Test Methods D 823.

5.2 *Micrometer Film Thickness Gage*, as described in Test Methods D 1005.

5.3 Alternative Release Substrates:

5.3.1 *Dental Tin Foil*, preferably 1-mil (25- μ m) thick.³

5.3.2 *Sheet of FEP*,⁴ preferably 2-mils (50- μ m) thick, coated with a thin film of a dry lubricant.⁵

5.3.3 *Sheet of Silicone Coated Paper*, preferably 5-mil (125- μ m) thick.⁶

5.3.4 *Glass Plates*, coated with halo-silane compound.

5.4 *Precision Specimen Cutter*, having a double blade with a foot to hold the sample.⁷

NOTE 1—Other substrates that may be suitable are 10-mil (250- μ m) thick polyethylene, photographic paper, polished steel, and fluoropolymer coated metal panels.

6. Hazards

6.1 *Mercury*—This practice involves the use of an OSHA-designated hazardous substance, mercury. For information on the potential hazards and guidance relative to use, consult the supplier's Material Safety Data Sheet. Mercury is a toxic metallic liquid. Its vapors are extremely hazardous. Small amounts of spilled mercury can vaporize sufficiently at room temperature to exceed the TVL of the vapor. Use with adequate ventilation (in a hood) or wear a respirator, and clean up spills immediately. Wear gloves when handling mercury. Containers should be kept closed. Droplets of mercury can be picked up by using a small glass pipet connected to a suction flask with a rubber hose.

7. Procedure

7.1 Prepare the free films by one of the following procedures:

7.1.1 Dental Tin Foil Substrate:

7.1.1.1 Apply each material to be tested to a strip of

³ Dental tin foil available from Yates and Bird, Chicago, IL 60610 has been found suitable for this purpose.

⁴ Teflon FEP 2-mil film thickness (Card No. 03111, Item #29499) available from E.I. du Pont de Nemours & Co., Inc., Wilmington, DE 19898, has been found suitable for this purpose.

⁵ Dry lubricant (MS-122 Fluorocarbon Release Agent) available from Miller-Stephenson Chemical Co., Danbury, CT 06810, has been found suitable for this purpose.

⁶ Silicone coated release paper Form RP-1K, size 8 3/4 by 11 3/4 in., available from the Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ, has been found suitable for this purpose.

⁷ The JDC Precision Sample Cutter manufactured by Thwing-Albert Instrument Co., 10960 Dutton Rd., Philadelphia, PA 19154, has been found suitable for this purpose.

dental tin foil. First, mount the foil on a smooth glass plate on which there may be placed a small pool of high-boiling liquid. Spread the foil out to a smooth flat surface by means of a rubber squeegee, which may be useful in holding the foil in close contact with the glass. Apply a uniform wet film of the material to the foil by one of the procedures given in Test Methods D 823.

NOTE 2—Use a liquid that is known to have little or no effect on the test coating. In many cases a light mineral oil would be suitable.

NOTE 3: Caution—Exercise care at all times to prevent the high-boiling liquids used in mounting the foil from coming in contact with the test film as these liquids may soften the film. For instance, the foil should be cleaned before amalgamation since any liquid remaining on the back of the foil and the test film will float on the bath of mercury.

7.1.1.2 Dry the applied films at $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity, or bake under conditions mutually agreeable to the producer and the user. Dry film thicknesses must not vary by more than the specified or agreed-upon tolerance of the average film thickness and must not contain visible flaws. Most films with a thickness of less than 2.0 mil (50 μm) are very difficult to handle.

7.1.1.3 When required for further treatment such as weathering, remount the coated foil on a convenient smooth, flat substrate such as a glass or steel panel. In this case, a high boiling liquid (Note 2) may be used to hold the foil in close contact with the substrate over extended periods of time.

7.1.1.4 Age or expose the coated foil to conditions mutually agreed upon. At the end of the specified or agreed upon period, remove the foil-backed film from the glass or metal substrate and cut the specimens to size using a sharp knife or a precision specimen cutter.

NOTE 4—To prepare specimens for tensile property tests a precision specimen cutter must be used to ensure nick-free edges on the specimens. Even with this instrument, it is necessary to cut each specimen independently, allowing at least $\frac{1}{2}$ in. (50 mm) of waste between specimens.

7.1.1.5 Place the foil-backed specimen film side up on a pool of mercury to remove the foil by amalgamation. After amalgamation is completed, pick up an end of the floating film with tweezers, and carefully brush the unsupported film free of mercury and amalgam with a soft camel hair brush.

7.1.2 FEP Substrate:

7.1.2.1 Apply each material to be tested to a treated sheet of FEP. First, cover a smooth, flat polished glass plate with a sheet of FEP. Coat the sheet uniformly with a light coat of a dry fluoro-carbon lubricant and allow to dry for 24 h at standard conditions. Then apply a uniform wet coating of the test material on the sheet by one of the procedures given in Test Methods D 823.

7.1.2.2 Dry the applied films in $73.5 \pm 3.5^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ humidity, or bake under conditions mutually agreeable to the producer and the user. Dry film thicknesses must not vary by more than the specified or agreed upon tolerances of the average film thickness and must be free of visible flaws. Most films with a thickness of less than 2.0 mil (50 μm) are very difficult to handle.

7.1.2.3 When required for further treatment such as weathering, remount the coated sheet on a convenient smooth, flat substrate such as a glass or steel panel.

7.1.2.4 Age or expose the coated sheet to conditions mutually agreed upon. At the end of the aging or exposure period, remove the sheet-backed film from the glass or metal substrate and cut the specimens to size using a sharp knife or the precision cutter (Note 4). Carefully strip the coating film from the sheet substrate.

7.1.3 Silicone Coated Paper:

7.1.3.1 Apply each material to a sheet of silicone coated paper. First, cover a smooth, flat substrate with a sheet of a silicone-coated release paper. Then apply a film of the material under test, dry or cure, expose or treat if required, and prepare test specimens as described in 7.1.2.

7.1.4 Halo-silane Coated Glass Plates—Apply each material to a glass plate coated with a halo-silane compound. First, coat a glass plate with a thin film of a halo-silane compound and dry. Then apply a film of the material under test, dry or cure, expose or treat if required, and prepare test specimens as described in 7.1.2.

8. Keywords

8.1 dental tin foil substrate; film; halo-silane coated glass plates; lacquer; organic coatings; paints and related coatings; polyhexafluoropylene (FEP) substrate; silicone-coated paper; substrates; surface preparation; tension (tensile) properties; tests; vapors; varnishes

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Standard Guide for Testing Industrial Water-Reducible Coatings¹

This standard is issued under the fixed designation D 4712; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the selection and use of procedures for testing water-reducible coatings, both pigmented and clear, utilizing synthetic latices, synthetic resin emulsions, or water-reducible alkyds. The methods included are listed in Table 1. Where more than one standard is listed for the same characteristic, no attempt is made to indicate superiority of one standard over another. Selection of the standards to be followed must be governed by experience and the requirements in each individual case, together with agreement between producer and user.

1.2 This guide covers the testing of liquid coatings as applied by conventional spray, airless spray, electrostatic spray, dip, fancoat, flowcoat, roller coat, and curtain coat.

1.3 This guide includes procedures relating to proper and safe packaging, shipping and receiving, and storage and handling during use and application.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- B 117 Method of Salt Spray (Fog) Testing²
- B 287 Method of Acetic Acid-Salt Spray (Fog) Testing³
- D 16 Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products⁴
- D 56 Test Method for Flash Point by Tag Closed Tester⁵
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester⁶
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints⁷
- D 344 Test Method for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts⁸
- D 522 Test Method for Mandrel Bend Test of Attached Organic Coatings⁸
- D 523 Test Method for Specular Gloss⁸

D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁸

D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁸

D 610 Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces⁸

D 658 Test Method for Abrasion Resistance of Organic Coatings by the Air Blast Abrasive⁸

D 659 Method for Evaluating Degree of Chalking of Exterior Paints⁸

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints⁸

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints⁸

D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints⁸

D 714 Test Method for Evaluating Degree of Blistering of Paints⁸

D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints⁸

D 822 Practice for Conducting Tests on Paint and Related Coatings and Materials Using Filtered Open-Flame Carbon-Arc Light- and Water-Exposure Apparatus⁸

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels⁸

D 869 Test Method for Evaluating Degree of Settling of Paint⁸

D 870 Practice for Testing Water Resistance of Coatings Using Water Immersion⁸

D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive⁸

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers⁸

D 1014 Method for Conducting Exterior Exposure Tests of Paints on Steel⁸

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water⁹

D 1150 Single and Multi-Panel Forms for Recording Results of Exposure Tests of Paints⁸

D 1186 Test Methods for Nondestructive Measurement of Dry-Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁸

D 1200 Test Method for Viscosity by Ford Viscosity Cup⁸

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁸

D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings⁸

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved June 26 and Oct. 30, 1987. Published December 1987.

² Annual Book of ASTM Standards, Vols 03.02 and 06.01.

³ Discontinued; See 1988 Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

⁵ Annual Book of ASTM Standards, Vols 05.01 and 06.03.

⁶ Annual Book of ASTM Standards, Vols 05.01.

⁷ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁸ Annual Book of ASTM Standards, Vols 06.01.

⁹ Annual Book of ASTM Standards, Vol 11.01.

- D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes⁸
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base⁸
- D 1474 Test Method for Indentation Hardness of Organic Coatings⁸
- D 1475 Test Method for Density of Paint, Varnish, Lacquers and Related Products⁸
- D 1535 Test Method for Specifying Color by the Munsell System¹⁰
- D 1540 Practice for Effect of Chemical Agents on Organic Finishes Used in the Transportation Industry⁸
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature⁸
- D 1653 Test Method for Water Vapor Permeability of Organic Coating Films⁸
- D 1654 Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments⁸
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials¹⁰
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting¹¹
- D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting¹¹
- D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting¹¹
- D 1735 Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus⁸
- D 1737 Test Method for Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus⁸
- D 1848 Classification for Reporting Paint Film Failures Characteristic of Exterior Latex Paints⁸
- D 1849 Test Method for Package Stability of Paint⁸
- D 2091 Test Method for Print Resistance of Lacquers⁸
- D 2092 Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting⁸
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁸
- D 2197 Test Methods for Adhesion of Organic Coatings by Scrape Adhesion⁸
- D 2201 Test Method for Preparation of Hot-Dipped Nonpassivated Galvanized Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products⁸
- D 2243 Test Method for Freeze-Thaw Resistance of Water-Borne Paints⁸
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁸
- D 2246 Test Method for Finishes on Primed Metallic Substrates for Humidity-Thermal Cycle Cracking⁸
- D 2247 Practice for Testing Water Resistance of Coatings in 100 % Relative Humidity⁸
- D 2248 Practice for Detergent Resistance of Organic Finishes⁸
- D 2353 Test Method for Flow Ratings of Organic Coatings Using the Shell Flow Comparator⁸
- D 2354 Test Method for Minimum Film Formation Temperature (MFT) of Emulsion Vehicles¹²
- D 2369 Test Method for Volatile Content of Coatings⁸
- D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁸
- D 2454 Practice for Determining the Effect of Overbaking on Organic Coatings⁸
- D 2574 Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms⁸
- D 2616 Test Method for Evaluation of Visual Color Difference With a Gray Scale¹⁰
- D 2691 Test Methods for Microscopical Measurement of Dry-Film Thickness of Coatings on Wood Products⁸
- D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings⁸
- D 2794 Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)⁸
- D 2803 Test Method for Filiform Corrosion Resistance of Organic Coatings on Metal⁸
- D 2805 Test Method for Hiding Power of Paints by Reflectometry⁸
- D 2933 Test Method for Corrosion Resistance of Coated Steel Specimens (Cyclic Method)⁸
- D 3002 Practice for Evaluation of Coatings for Plastics⁸
- D 3023 Practice for Determination of Resistance of Factory-Applied Coatings on Wood Products to Stains and Reagents⁸
- D 3134 Practice for Evaluating Color and Gloss Tolerances¹⁰
- D 3168 Practice for the Qualitative Identification of Polymers in Emulsion Paints⁷
- D 3170 Test Method for Chipping Resistance of Coatings⁸
- D 3278 Test Methods for Flash Point of Liquids by Setflash Closed-Cup Apparatus¹³
- D 3281 Test Method for Formability of Attached Organic Coatings with Impact-Wedge Bend Apparatus⁸
- D 3359 Test Methods for Measuring Adhesion by Tape Test⁸
- D 3361 Practice for Operating Light- and Water-Exposure Apparatus (Unfiltered Open-Flame Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products Using the Dew Cycle⁸
- D 3793 Test Method for Low-Temperature Coalescence of Latex Paint Films⁸
- D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials⁸
- D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings⁸
- D 3928 Test Method for Evaluation of Gloss or Sheen Uniformity⁸
- D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser⁸
- D 4062 Test Method for Leveling of Paints by Draw-Down Method⁸
- D 4399 Method for Measuring Electrical Conductivity of Electrocoat Baths⁸

¹⁰ Annual Book of ASTM Standards, Vol 14.02.

¹¹ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

¹² Annual Book of ASTM Standards, Vol 06.02.

¹³ Annual Book of ASTM Standards, Vol 06.03.

D4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation⁸

D4587 Practice for Conducting Tests on Paint and Related Coatings and Materials Using a Fluorescent UV-Condensation Light- and Water-Exposure Apparatus⁸

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode¹⁴

D 2 U.S. Federal Test Method Standard No. 141c.¹⁵

D131.1 Application of Sprayed Films

3011.2 Condition in Container

Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this guide, refer to definitions D 16.

Significance and Use

4.1 This compilation of standards is intended to provide assistance in selecting appropriate tests for evaluating water-reducible coatings and for determining what characteristics should be considered for a given end use. Either single-coat operations or multicoat systems may be addressed by the proper selection of tests. Results from the various tests are not all necessarily useful in evaluating the performance of different systems for various end uses. The list can be useful to those developing coatings and coating systems and to those seeking coating systems for products.

Equipment

5.1 Use the equipment as specified in each standard.

General Requirements

6.1 Tests and observations shall be at standard laboratory conditions as specified in Specification D 3924 unless otherwise specified or agreed upon by the producer and user.

Sampling and Specimen Preparation

7.1 Sample the water-reducible coatings in accordance with Practice D 3925.

7.2 Prepare specimens as required for the specific tests on the liquid coating and the dry coating.

8. Conditions Affecting Performance

8.1 Practical requirements and performance of water-reducible coatings may vary with:

8.1.1 Type of substrate.

8.1.2 Substrate condition, for example, porosity, hardness, smoothness, flexibility, etc.

8.1.3 Type, quality, and suitability of the surface treatment or primer used under the water-reducible coating and the time before coating application.

8.1.4 Application methods and techniques.

8.1.5 Contaminants on the surface of the substrate.

8.1.6 Environmental conditions such as temperature and relative humidity.

8.1.7 Damage to container, size, and type of container.

8.1.8 Storage variables, for example storage time, excessive temperature fluctuations that may cause physical or chemical change. Special needs arise due to carbon dioxide absorption, dissolved metal compatibility, and ultrafiltration treatments.

9. Liquid Coatings Properties

9.1 *Condition in Container*—Thickening, settling, and separation are undesirable and objectionable if a liquid coating cannot be reconditioned and made suitable for application with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties after storage. Determine the condition in the container in accordance with Method 3011.1 of U.S. Federal Test Method Standard No. 141c.

9.2 *Coarse Particles and Foreign Matter*—To form uniform films of good appearance, the liquid coating must be free of coarse particles as agreed upon between the producer and the user, a typical maximum being 1 % by weight of the total paint. Determine coarse particles and foreign matter in accordance with Test Methods D 185.

9.3 *Density or Weight Per Gallon*—The density as measured by weight per gallon is used to help assure product uniformity from batch to batch. In the referenced test method, the density is expressed as the weight in pounds avoirdupois of 1 U.S. gal or the weight in kilograms of 1 L of the paint at a specified temperature. A calibrated weight-per-gallon cup is used. Determine the density in accordance with Test Method D 1475.

9.4 *Fineness of Dispersion*—The more finely a pigment is dispersed, the more efficiently it is being used. One test method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the material down a calibrated, tapered groove in a hardened steel block with the groove varying in depth from 4 to 0 mils (100 to 0 μ m). The point at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the liquid is taken as the fineness reading. Lower readings in mils or μ m or higher readings in Hegman units indicate better fineness of dispersion. Determine fineness of dispersion in accordance with Test Method D 1210.

9.5 *Pigment Suspension*—The amount and type of settling is an indication of how well the pigments remain in suspension and how easily settled pigment can be remixed. Determine degree of settling in accordance with Test Method D 869.

9.6 *Viscosity*—Viscosity refers to the flow resistance of a fluid and should fall within an agreed-upon range. Viscosities of Newtonian fluids (constant viscosity regardless of shear rate) may be measured with a Ford Cup. Viscosities of non-Newtonian materials should be measured at two or more speeds with a Brookfield rotational viscometer. Determine viscosity in accordance with Test Methods D 1200 or D 2196.

9.7 *Consistency*—Consistency is a less precise term than viscosity for evaluating the flow properties of a material. In the referenced test method, consistency is defined as the load in grams required to produce a specific rate of rotation in a specimen using the Stormer Viscometer. This is a one-speed test method and is not recommended for paints that show shear thinning or thixotropy. Determine consistency in

¹⁴ Annual Book of ASTM Standards, Vol 15.05.

¹⁵ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

TABLE 1 List of Test Methods by Properties

Test Method	Section	ASTM Standard	Federal Test Method Standard 141
I. Liquid Coating Properties:			
Coarse particles and foreign matter	9.2	D 185	...
Condition in container	9.1	...	3011.1
Conductivity	9.14	D 1125 or D 4399	...
Consistency	9.7	D 562	...
Density or weight per gallon	9.3	D 1475	...
Fineness of dispersion	9.4	D 1210	...
Flash point	9.13	D 56, D 93 or D 3278	...
Freeze-thaw stability	9.9	D 2243	...
Microorganism resistance	9.11	D 2574	...
pH	9.8	E 70	...
Package stability	9.10	D 1849	...
Pigment suspension	9.5	D 869	...
Surface tension	9.12	...	...
Viscosity	9.6	D 1200 or D 2196	...
II. Application and Film Formation:			
Panel preparation	10.1	D 609, D 1730, D 1731, D 1732, D 2082, D 2201	...
Drying properties	10.2.2	D 1640	...
Leveling properties	10.3	D 4062, D 2353	...
Low temperature coalescence	10.5	D 3793, D 2354	...
Spray properties	10.2.1	...	2131.1, 4331.1
Touch-up	10.6	D 3928	...
Uniform film preparation	10.4	D 823	...
Wet film thickness	10.3	D 1212	...
III. Appearance of Dry Film:			
Color difference by visual evaluation	11.1.1	D 1729, D 2616	...
Color difference by instrumental evaluation	11.1.2	D 2244 and D 3134	...
Color description by visual evaluation	11.1.3	D 1535	...
Color description by instrumental evaluation	11.1.4	D 2244	...
Gloss	11.2	D 523	...
Hiding power	11.3	D 344, D 2605	...
IV. Properties of Dry Film:			
Abrasion resistance	12.1	D 658, D 968, D 4060	...
Adhesion	12.2	D 2197, D 3359	...
Elongation	12.3	D 522, D 1737, D 3281	...
Exterior exposure	12.4	...	...
Blistering	12.4.1	D 714	...
Chalking	12.4.2	D 659	...
Checking	12.4.3	D 660	...
Cracking	12.4.4	D 661	...
Erosion	12.4.5	D 662	...
Flaking	12.4.6	D 772	...
Rusting	12.4.7	D 610, D 2933	...
Accelerated weathering	12.4.8	D 822, D 3361, D 4587	...
Corrosive environments	12.4.9	D 1654	...
Hardness	12.5	D 1474	...
Impact resistance	12.6	D 2794, D 3170	...
Resistance to various forms of water	12.8	...	...
Water immersion	12.8.1	D 870	...
Water fog	12.8.2	D 1735	...
Humidity resistance	12.8.3	D 2247	...
Moisture vapor permeability	12.8.4	D 1653	...
Salt spray	12.8.5	B 117, B 287	...
Humidity-thermal cycling	12.8.6	D 2246	...
Filiform corrosion	12.8.7	D 2803	...
Condensation	12.8.8	D 4585	...
Detergent resistance	12.8.9	D 2248	...
Resistance to chemicals	12.7	D 1308, D 1540, D 3023	...
Overbaking	12.9	D 2454	...
Print resistance	12.10	D 2091	...
Reporting results	12.4.10	D 1848	...
Dry film thickness	12.11	D 1186, D 1400	...
V. Analysis of Paint:			
Volatile content	13.2	D 2369	...
Volume of nonvolatile	13.3	D 2697	...
Weight of nonvolatile	13.3	D 2369	...
Pigment content	13.4	D 2371	...
Identification of vehicle solids	13.6	D 3168	...

accordance with Test Method D 562.

9.8 pH—The pH of a water-reducible coating depends on the type of vehicle used and the general formulation. It may vary from about 4 to 10. A change in pH during storage may indicate poor stability or a change in properties of a water-reducible coating. Determine pH in accordance with Test Method E 70.

9.9 Freeze-Thaw Resistance—Water-reducible coatings may be subjected to freezing conditions during shipping and storage. Suitably stabilized paints will resist several cycles of freezing and thawing without showing deleterious changes. The referenced method covers the determination of the extent to which water-reducible coatings retain their original consistency and freedom from lumps when subjected to freezing and subsequent thawing. Determine freeze-thaw resistance in accordance with Test Method D 2243.

9.10 Package Stability—Since liquid coatings cannot normally be used immediately after manufacture, they must remain stable in the package for some time. The referenced test method covers the change in consistency and in certain related properties that may take place in packaged water-reducible coatings when stored at a temperature above room temperature. Determine package stability in accordance with Test Method D 1849, at a temperature and for a period of time agreed upon by the purchaser and the seller.

NOTE—Although there is no ASTM or Federal test method for determining gassing during normal storage, special containers may be necessary to vent any spontaneous pressure buildup.

9.11 Microorganism Resistance—Microorganisms in water-reducible coatings can cause gassing, putrefaction or fermentation odors, and loss of viscosity. Determine if the liquid coating contains living bacteria and if it is resistant to attack by bacteria in accordance with Test Method D 2574.

9.12 Surface Tension—Although there is no ASTM or Federal test method for determining surface tension of liquid coatings, this is an important property of a water-reducible resin or coating. If surface tension is too high, poor pigment and substrate wetting may occur, leading to cratering, low gloss, or other surface defects. The most common methods for measuring the surface tensions of coatings probably are the ring pull method and drop weight method. For a comprehensive discussion of these and other aspects of surface tension, see *Paint Flow and Pigment Dispersion*.¹⁶

9.13 Flash Point—Nearly all water-borne coatings are incapable of sustaining combustion, but many do contain volatile solvents whose vapors can ignite if near open flame. Because they do give flash points, water-borne coatings must be tested for flash point temperature to conform with many government regulations concerning transportation, labeling, packaging, etc. Determine flash point in accordance with Test Methods D 56, D 93 or D 3278.

9.14 Conductivity—Conductivity is an important factor in the application of some water-borne coatings. Test Methods available for determining conductivity are D 1125 (specifically Methods A and B) and D 4399.

10. Application and Film Formation

10.1 Panel Preparation—Select a substrate as agreed upon

by the producer and the user. Prepare panels for testing the coating in accordance with Test Methods D 609, D 1730, D 1731, D 1732, D 2201, or D 2092. The preparation of plastics for paint testing is covered in Practice D 3002.

10.2 Application Properties—Determine the ease with which the liquid coating can be applied to various surfaces with brush, spray, or other application equipment. Application properties are generally compared to a standard, or described by requirements in a product specification. Application properties are related to such characteristics as kinematic viscosity, non-Newtonian rheology, surface tension, shear sensitivity, micelle stability, electrical resistivity, erosion abrasiveness, conductivity, heat capacity, and corrosiveness.

10.2.1 Sprayed Film Application—Liquid coatings can be applied by spray. Determine the spray application properties in accordance with Method 2131.1 of Federal Test Method Standard No. 141. The method can be modified to include application by airless spray equipment.

10.2.2 Drying Properties—The drying time of water-reducible coatings is important in determining when the applied coatings can be handled or packed. Also, inadequate drying of the film may result in poor film and poor appearance and, if used on an exterior surface, rain, dew, or snow may cause a nonuniform appearance. Determine drying time in accordance with Test Method D 1640, or as agreed upon by producer or user.

10.3 Leveling Properties—Leveling is an important factor when uniform surfaces are to be produced, as it affects hiding and appearance. The referenced methods cover the laboratory determination of the relative leveling characteristics of liquid coatings. Determine the leveling characteristics in accordance with Test Method D 4062. Measure wet film thickness in accordance with Test Method D 1212.

10.4 Producing Films of Uniform Thickness—The following test method covers the preparation of various films of uniform thickness essential in conducting tests. Prepare films in accordance with Test Methods D 823.

10.5 Low Temperature Coalescence of Paints—A test method to determine how well the latex particles in coating will fuse together or coalesce to form a continuous film at low temperature is described in Test Method D 3793. A test for the minimum film formation temperature is described in Test Method D 2354.

10.6 Touch-Up—For many coating systems it is important to be able to repair damage sustained during production, delivery, or after delivery. A coating can be tested by applying it with a small nylon bristle brush or air brush to a small section of a panel previously coated with it. When the touch-up area has dried, it is examined to see if it differs significantly from the initial coating. Determine the ability to touch up the coating in accordance with Test Method D 3928. Test the adhesion of the original and touch-up areas in accordance with Test Methods D 3359 or other agreed-upon test method.

11. Appearance of Dry Film

11.1 Color—The color of a water-reducible coating may be specified independently or as the color-difference with respect to another color that is usually the standard. Visual and instrumental methods are both applicable. An opaque

¹⁶ Patton, T., *Paint Flow and Pigment Dispersion*, 2nd Ed., Wiley-Interscience, New York, 1979, pp. 205-246.

film is preferred that may be prepared by making one or more applications of water-reducible coating onto a black and white substrate until the substrate is completely obscured. Each application should be performed in a normal manner with respect to application method, drying, and film thickness.

11.1.1 *Color Differences by Visual Evaluation*—Visual comparison of color is fast and often acceptable although numerical values are not obtained. The referenced standard covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, size of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials. Determine color difference by visual evaluation in accordance with Practice D 1729 or Test Method D 2616.

11.1.2 *Color Differences of Opaque Material by Instrumental Evaluation*—Color difference between a product and the standard can be measured by an instrument. Generally, the tolerance is agreed upon by the purchaser and the seller and may also be required if a product specification is involved. Color instruments provide numerical values that can be subsequently compared to later measurements. The referenced method covers the instrumental determination of small color differences observable in daylight illumination between nonfluorescent, nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (10.1.1) should be used to verify the results. Make instrumental measurement of color difference in accordance with Method D 2244. Tolerances are discussed in Recommended Practice D 3134.

11.1.3 *Color Description by Visual Evaluation*—In some cases it is necessary to specify or identify a color instead of a color difference from some standard. Various color atlases are available, the most common being the Munsell System. Describe or identify the Munsell color in accordance with Method D 1535.

11.1.4 *Color Description by Instrumental Evaluation*—Instrumental measurements involve the determination of CIE tristimulus values, X, Y, and Z, from which other color coordinates such as L^* , a^* , b^* , or L , a , b values may be calculated or obtained directly with some instruments. Describe or identify in accordance with Method D 2244.

11.2 *Gloss*—Water-reducible coatings vary in gloss and the end use determines whether the gloss should be high, semi-gloss, eggshell, or flat. Determine the gloss in accordance with Test Method D 523 using 20, 60, or 85° geometry as appropriate.

11.3 *Hiding Power (Dry Opacity)*—Hiding power is the measure of the ability of a paint to hide the substrate. It is, however, dependent upon uniform film thickness which is influenced by flow and leveling. Test Method D 344 is a practical test in which paint is applied with a brush, film thickness is approximately measured, and opacity is evaluated visually as compared to a standard paint. Results are affected by flow and leveling application properties of the paint. Test Method D 2805 is considered to be a more precise and accurate test that does not need a material paint standard. Paint is applied with an applicator bar to minimize the effects of flow and leveling, film thickness is rigorously measured, and opacity is instrumentally evaluated. Determine

hiding power in accordance with Test Methods D 344 or D 2805.

12. Properties of Dry Film

12.1 *Abrasion Resistance*—Abrasion resistance is a measure of the ability of a dried coating to withstand wear and marring from objects rolled or pulled across the surface. Determine abrasion resistance in accordance with Test Method D 658, D 968, or D 4060 (see methods to determine the applicability for interlaboratory use).

12.2 *Adhesion*—Adhesion is the property of the coating that resists removal from the substrate when scraped. Test Methods D 2197 covers the use of a scrape adhesion tester and a parallel groove adhesion tester. In Test Methods D 3359 cuts are made in the film and pressure-sensitive tape applied and removed. Determine adhesion in accordance with Test Methods D 2197 or D 3359.

12.3 *Elongation*—Elongation is a measure of the flexibility of coating films. Determine elongation in accordance with Test Methods D 522, D 1737, or D 3281.

12.4 *Exterior Exposure*—If the paint is intended for exterior exposure, tests may be run in accordance with Method D 1014, and Standard D 1150 and evaluated by the following methods:

12.4.1 *Blistering*—Determine the degree of blistering in accordance with Test Method D 714.

12.4.2 *Chalking*—Determine the degree of chalking in accordance with Method D 659.

12.4.3 *Checking*—Determine the degree of checking in accordance with Test Method D 660.

12.4.4 *Cracking*—Determine the degree of cracking in accordance with Test Method D 661.

12.4.5 *Erosion*—Determine the degree of erosion in accordance with Test Method D 662.

12.4.6 *Flaking*—Determine the degree of flaking in accordance with Test Method D 772.

12.4.7 *Rusting*—Determine the degree of rusting in accordance with Test Methods D 610 or D 2933.

12.4.8 *Accelerated Weathering*—Determine the resistance to accelerated weathering in accordance with Test Method D 822, D 3361, or D 4587.

12.4.9 *Corrosive Environment*—Determine the resistance to corrosive environment in accordance with Test Method D 1654.

12.4.10 *Reporting of Results*—The reporting of results can often be helped by reference to the classifications prescribed in Classification D 1848.

12.5 *Hardness*—Hardness is a measure of the ability of a dried coating to resist indentation. Determine hardness in accordance with Test Method D 1474.

12.6 *Impact Resistance*—An important property of a water-reducible coating is its ability to withstand a striking blow or impingement. Determine the impact resistance in accordance with Test Methods D 2794 or D 3170.

12.7 *Resistance to Chemicals*—An important property of a water-reducible coating is its ability to resist spotting, softening, or removal when subjected to household chemicals or strong cleaners. Determine resistance to chemicals in accordance with Test Methods D 1308 or D 1540 or Practice D 3023.

12.8 *Resistance to Various Physical Forms of Water*—

The ability of a dried coating to resist water in many different forms is an especially important property of water-reducible coatings.

12.8.1 *Water Immersion*—Determine the resistance to water absorption in accordance with Practice D 870.

12.8.2 *Water Fog*—Determine the resistance to water fog in accordance with Practice D 1735.

12.8.3 *Humidity Resistance*—Determine the resistance to 100 % relative humidity in accordance with Practice D 2247.

12.8.4 *Moisture Vapor Permeability*—Determine the resistance to moisture vapor transmission in accordance with Test Method D 1653.

12.8.5 *Salt Spray*—Determine the resistance to salt spray (fog) in accordance with Methods B 117 or B 287.

12.8.6 *Humidity-Thermal Cycling*—Determine the resistance to humidity-thermal cycling in accordance with Test Method D 2246.

12.8.7 *Filiform Corrosion*—Determine the resistance to filiform corrosion in accordance with Test Method D 2803.

12.8.8 *Condensation*—Determine the resistance to condensed water such as dew in accordance with Practice D 4585.

12.8.9 *Detergent Resistance*—Determine the resistance to detergent solution in accordance with Practice D 2248.

12.9 *Overbaking*—The ability of a coating to withstand a baking temperature moderately higher than initial bake or for a longer period of time is an important property. Measure the resistance to overbaking by Practice D 2454.

12.10 *Print Resistance*—Print resistance is the ability of a dried coating to withstand pressure from a textured surface without any noticeable marking on the coating. Measure print resistance in accordance with Test Method D 2091.

12.11 *Dry Film Thickness*—There are several methods currently being used for determining dry film thickness. Depending on the substrates being used, the following test methods should be considered: D 1005, D 1186, D 1400, and D 2691.

13. Analysis of Paint

13.1 *Chemical Analysis*—If a specification requires certain raw materials or certain components in a given amount, then chemical analysis is required. Chemical analysis deter-

mines whether the specified components are present, and if they are, in what amounts. It does not necessarily establish quality, which can also be greatly affected by manufacturing techniques. Most ASTM analytical methods, such as Method D 215, apply to solvent-type coatings. However, some of these can be adapted for analysis of water-reducible coatings.

13.2 *Volatile Content*—The percent of volatile matter indicates the water and organic solvent released from the film as it dries. This quantity subtracted from 100 % gives the nonvolatile content. Determine the volatile content in accordance with Test Method D 2369.

13.3 *Nonvolatile Content*—The percent of nonvolatile matter indicates the amount of material present that can be converted to the desired film. Determine the weight percent of nonvolatile in accordance with Test Method D 2369. Determine the volume percent of nonvolatile in accordance with Test Method D 2697.

13.4 *Pigment Content*—Although the referenced method describes the procedure for quantitative separation of the vehicle from the pigment in solvent-reducible coatings, it can be adapted for water-reducible coatings to determine the weight percent pigment in the paint. Determine the pigment content in accordance with Test Method D 2371.

13.5 *Pigment Analysis*—The analysis of pigment may be required if the product is covered by a specification, or if it is agreed upon between the producer and the user. Analyze the pigment in accordance with methods appropriate for the constituents present or specified.

13.6 *Identification of Vehicle Solids*—The suggested method covers the qualitative characterization or identification of separated paint vehicle solids by infrared spectroscopy. It is useful in detecting uniformity, batch to batch, and the presence of adulterants. Characterize vehicle solids in accordance with Practice D 3168.

14. Keywords

14.1 alkyds; appearance of materials; application properties; brush application; color; curtain coat; dip application; electrostatic spray; emulsion vehicles; exterior exposure; fancoat; film formation rates; flowcoat; instrumental evaluation; opaque film; paints and related coatings; paints, water-base; roller coat; spray method; visual examination; water-reducible coatings.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Methods for Nonvolatile Content of Printing Inks, Resin Solutions, and Vehicles¹

This standard is issued under the fixed designation D 4713; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^e) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Paragraphs 3.2 and 11.1.2 were editorially corrected in May 1988.

1. Scope

1.1 These test methods cover the determination of weight content of nonvolatile matter in two types of printing inks.

1.2 Test Method A is applicable to heatset-type printing inks and resin solutions; solvents in such systems typically have initial boiling points in the range from 240 to 275°C (470 to 535°F) and vapor pressures less than 0.2 mm Hg.

1.3 Test Method B is applicable to liquid-type printing inks and vehicles based on aqueous or organic solvents that evaporate readily at ordinary room temperatures.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—Test Method A (for heatset systems) specifies a specimen film thickness that is much thinner than those produced by related test methods; one exception is Test Method B in Test Method D 1259, which is recommended as a referee test.

NOTE 2—Test Method B (for liquid ink systems) is similar to Test Method D 2369 except that a solvent is not required for spreading the test specimen.

2. Referenced Documents

2.1 ASTM Standards:

D 1259 Test Methods for Nonvolatile Content of Resin Solutions²

D 2369 Test Method for Volatile Content of Coatings³

E 1 Specification for ASTM Thermometers⁴

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens⁵

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁶

3. Summary of Test Methods

3.1 *Test Method A—Heatset Systems.* A 0.15-g specimen is mechanically spread in a 57-mm weighing dish to a nominal thickness of 80 ± 10 g/m² and heated in a forced

ventilation oven at 110°C for 3 h.

3.2 *Test Method B—Liquid Ink Systems.* A 0.5-g specimen is dispensed into a 57-mm weighing dish by means of a disposable syringe, mechanically spread out, and heated in an oven at 110°C for 1 h.

4. Significance and Use

4.1 Nonvolatile content of printing inks is useful for specification acceptance between the supplier and the purchaser.

4.2 In order to obtain accurate results for heatset systems within the specified 3-h heating time, the specimen film thickness must be less than 100 g/m², and the oven must have forced ventilation. Thickness of the specimen film is less critical for liquid ink systems.

5. Apparatus

5.1 *Balance*, accurate to 1 mg.

5.2 *Oven*, forced-ventilation type conforming to Type IIB in Specification E 145 and maintained at $110 \pm 2^\circ\text{C}$.

5.3 *Thermometer*, bulb-type, capable of reading $110 \pm 2^\circ\text{C}$, such as Thermometer 88C prescribed in Specification E 1.

5.4 *Weighing Dish*, such as an aluminum foil dish 57 mm wide, the lid of a 1-lb ink can 94 mm wide, or other flat-bottomed container. The bottom of the container must not have a trough or depression into which the test material might collect.

5.5 *Spatula*, or small ink knife.

5.6 *Spreading Device*, one per weighing dish, of heat-stable material, such as a glass stirring rod or thick L-shaped wire.

5.7 *Forceps*,

5.8 *Desiccator*,

5.9 *Syringe*⁷ (for liquid ink systems only), single-use 2 to 5-mL capacity without needle, or other weighing device listed in the Apparatus section of Test Method D 1259.

6. Reagents

6.1 *Toluene*, technical grade.

7. Preparations of Equipment and Sample

7.1 Check the levelness of shelving in the oven; adjust, if necessary. Lay the thermometer on shelf with the bulb at the place where the samples will be placed. Adjust oven controls

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved June 26, 1987. Published August 1987.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 05.03 and 14.03.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Annual Book of ASTM Standards, Vols 06.03, 08.03, and 14.02.

⁷ Available from any scientific supply house.

until thermometer reads $110 \pm 2^\circ\text{C}$. If air flow is adjustable, set control dampers at 50 %.

7.2 Wear disposable gloves prior to handling weighing dish, spreading device, or syringe in order to minimize contamination by moisture from hands.

7.3 Measure diameter of bottom of weighing dish in millimetres. For example, a catalogue listing of 57 mm refers to the top diameter, whereas the bottom diameter may be only 50 mm. The bottom diameter must be used in the calculations of weight per unit area in 9.3.

7.4 Mark weighing dishes with a suitable notation. Rinse with toluene and heat in oven at 110°C for $\frac{1}{2}$ h.

7.5 Thoroughly mix ink in container to ensure that the sample is uniform. Close can after removing specimen. Reseal when finished.

8. Procedure

8.1 Test Method A—Heatset Systems:

8.1.1 For each specimen, tare to the nearest milligram two weighing dishes each with a spreading device. Retain spreading device throughout the test.

8.1.2 Transfer a representative portion of the sample to the tip of a spatula and dab about 0.15 ± 0.02 g around the bottom of each 57-mm dish, or 0.43 ± 0.02 g if a 94-mm can lid is used. Quickly reweigh and calculate the weight per unit area in accordance with 9.3. If in excess of 100 g/m^2 , discard and weigh out a new specimen.

8.1.3 With spreader, smooth out the specimen into a reasonably uniform film covering the entire bottom of the dish. High viscosity inks may require a few drops of a suitable solvent to aid in spreading out the film.

8.1.4 Place the dishes in the forced draft oven at 110°C for 1 h. Remove dishes from oven, cool in desiccator, and reweigh.

8.2 Test Method B—Liquid Ink Systems:

8.2.1 Tare weighing dishes as in 8.1.1. Transfer 2 to 4 mL of representative sample to syringe and weigh. Dispense a 0.5 ± 0.1 g specimen from the syringe to a 57-mm dish, or 1.5 ± 0.1 g to a 94-mm can lid. Immediately spread out as in 8.1.2. Reweigh syringe.

8.2.2 Repeat 8.2.1 with second dish.

8.2.3 Place dishes in forced draft oven at 110°C for 1 h. Remove dishes from oven, cool in desiccator, and reweigh.

9. Calculation

9.1 Calculate content of nonvolatile matter as follows:

$$NVM, \% = (W/S) \times 100$$

where:

W = specimen weight after heating, g.

S = initial specimen weight, g.

9.2 Optional: The percent of volatile matter may be calculated by difference as follows:

$$VM, \% = 100 - NVM \%$$

9.3 Calculate initial weight/area of each specimen:

$$W/A = 1\,000\,000\,S/3.14\,R^2, \text{ g/m}^2$$

where:

W = weight,

A = area, and

R = radius of dish bottom, mm = diameter/2.

NOTE 3—For a dish with a 50-mm bottom diameter, weight/area = 510 S. For a can lid with a 94-mm bottom diameter, weight/area = 1450 S.

10. Report

10.1 Report *NVM* to the nearest 0.1 % as the mean of replicate determinations.

10.2 Optional: Report *VM* to the nearest 0.1 %.

10.3 Report the mean weight per unit area of the initial specimens to the nearest gram per square metre.

11. Precision and Bias

11.1 Precision:

11.1.1 *Test Method A—Heatset Systems*. An interlaboratory⁸ study was conducted in which one operator in each of five laboratories tested in duplicate on each of two days four heatset printing inks, of which two were low *NVM* (about 50 %) and two were high *NVM* (about 60 %). The round-robin data were analyzed according to Practice E 691. There were no outliers. The within-laboratory pooled standard deviation was found to be 0.44 % absolute at 12 degrees of freedom, and the between-laboratories pooled standard deviation was 2.0 % absolute at 16 degrees of freedom. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1.1 *Repeatability*—Two results, each the mean of two runs obtained by one operator, should be considered suspect if they differ by more than 1.2 % absolute.

11.1.1.2 *Reproducibility*—Two results, each the mean of two runs obtained by operators in different laboratories, should be considered suspect if they differ by more than 5.7 % absolute.

11.1.2 *Test Method B—Liquid Ink Systems*. See Precision section of Test Method D 2369.

11.2 *Bias*—In the interlaboratory study of heatset inks described in 11.1, the mean values for *NVM* agreed with the calculated values within 1 % absolute.

12. Indexing Terms

12.1 These test methods are indexed under the following terms: Heatset-type printing inks, ink, nonvolatile matter content, ovens, printing inks, resin solutions, solvents, and vehicles.

⁸ Supporting data are available from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103. Request RR D01-1053.

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Standard Test Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub¹

This standard is issued under the fixed designation D 4752; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method describes a solvent rub technique for assessing the MEK resistance of ethyl silicate (inorganic) zinc-rich primers. The MEK resistance of some two-component ethyl silicate zinc-rich primers has been shown to correlate well with the cure of the primer as determined by diffuse reflectance infrared spectroscopy.² The technique can be used in the laboratory, field, or in the fabricating shop.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 ASTM Standards:

D 740 Specification for Methyl Ethyl Ketone³

D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base⁴

3. Description of Term Specific to This Standard

3.1 *double rub*—a back-and-forth motion over the test area of approximately 2 in. (50 mm).

4. Significance and Use

4.1 Ethyl silicate zinc-rich primers cure by the reaction of the vehicle with moisture thereby providing a binder. As relative humidity and temperature vary during the day, so does the rate of cure. A certain minimum degree of cure is necessary prior to topcoating. It has been shown that the degree of cure of ethyl silicate zinc-rich primers can be measured by the chemical changes occurring using diffuse reflectance infrared spectroscopy.² This solvent rub test has been shown to correlate well with the infrared spectroscopic results of two-component ethyl silicate inorganic zinc systems.

4.2 The cure rating required for the application of specific topcoats must be agreed upon before the test method is used.

5. Reagents and Materials

5.1 *Methyl Ethyl Ketone* (MEK), in accordance with Specification D 740.

5.2 *Cotton Cheesecloth*.

5.3 *Squeeze Bottle*.

6. Precautions

6.1 Methyl ethyl ketone is a flammable liquid. Its vapors form explosive mixtures in air. Repeated or prolonged contact can cause drying of the skin. Consult supplier's Material Safety Data Sheet(s) for specific hazard information.

7. Procedure

7.1 Select areas on the primer surface at least 2 in. (50 mm) long on which the tests will be run. Measure the dry film thickness of the primer in the selected areas in accordance with Test Methods D 1186.

7.2 Clean the surface first with fresh water to remove loose material.

7.3 Immediately fold the cheesecloth into a pad containing four thicknesses of the cloth. Saturate the cloth to a dripping wet condition with the methyl ethyl ketone.

7.4 Rub the test area with the saturated cloth, exerting moderate pressure with the thumb, using a 2-in. (50-mm) long stroke that encompasses the test area.

7.5 Continue rubbing the surface with the MEK saturated pad, wetting the pad as necessary without lifting it from the surface, until either the metal substrate is exposed or 50 double rubs have been completed. Record the number of rubs when the substrate is exposed.

7.6 Select an adjacent area to be used as a control. Repeat 7.1 through 7.5, except use a dry cheesecloth to establish the effect of burnishing without the influence of MEK.

TABLE 1 Scale for Resistance Rating

Resistance Rating	Description
5	No effect on surface; no zinc on cloth after 50 double rubs
4	Burnished appearance in rubbed area; slight amount of zinc on cloth after 50 double rubs
3	Some marring and apparent depression of the film after 50 double rubs
2	Heavy marring; obvious depression in the film after 50 double rubs
1	Heavy depression in the film but no actual penetration to the substrate after 50 double rubs
0	Penetration to the substrate in 50 double rubs or less

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Material and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

Current edition approved Nov. 27, 1987. Published January 1988.

² Starr, T. L., Henton, L. E., Lewis, W. S., and Rideout, F. A., "Improved Field Reliability of High Performance Coating Systems: Phase II—Develop Procedures and Criteria in Critical Performance Areas," available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

³ Annual Book of ASTM Standards, Vol 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.01.

7 Inspect the test areas and the cheesecloths. Rate the results in accordance with Table 1.

Report

8.1 Report the following information:

- 8.1.1 Dry film thickness of the primer.
- 8.1.2 Elapsed time between the application of the primer and the running of the tests.
- 8.1.3 Number of tests conducted.
- 8.1.4 Resulting ratings.
- 8.1.5 In the case of a zero rating, number of double rubs required to expose the substrate.
- 8.1.6 *Field and Fabricating Shop Tests*—Identification of

the area or piece tested.

9. Precision and Bias

9.1 Since no standard for measuring the MEK resistance of ethyl silicate zinc-rich primers has been developed prior to this test method, precision and bias statements will be developed in round-robin testing.

10. Indexing Terms

10.1 This test method is indexed under the following terms: curing characteristics, double rub method, drying or curing, ethyl silicate (inorganic) primer, methyl ethyl ketone, MEK (methyl ethyl ketone) resistance, primer, solvent rub method, visual examination, and zinc-rich primer.

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Standard Test Method for Determination by X-ray Fluorescence Spectroscopy of Titanium Dioxide Content in Paint¹

This standard is issued under the fixed designation D 4764; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of titanium dioxide content in liquid paint. This test method is applicable to both water-reducible and solvent-reducible paints.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazards statements are given in Section 7.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
 - D 476 Specification for Titanium Dioxide Pigments²
 - D 1193 Specifications for Reagent Water³
 - D 1394 Test Methods for Chemical Analysis of White Titanium Pigments²
 - D 2369 Test Method for Volatile Content of Coatings⁴
 - D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings⁴
 - D 3980 Practice for Interlaboratory Testing of Paint and Related Materials⁴
 - D 4563 Test Method for Determination by Atomic Absorption Spectroscopy of Titanium Dioxide Content of Pigments Recovered from Whole Paint⁴
 - E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁵

3. Summary of Method

3.1 Paint containing a known amount of titanium dioxide is used as the reference standard. A solution of tetraethylammonium bromide in 2-ethoxyethanol is added to the standard paint and to each paint being analyzed. Drawdown films of the standard and test paint are made on thin plastic sheets. The intensities of key X-ray fluorescence peaks of titanium and bromine are measured. The titanium dioxide content is determined by comparing the ratio of the intensities of titanium and bromine peaks in the test paint and the standard. Differences in the nonvolatile content of the paints

are taken into account in the calculation of results.

4. Significance and Use

4.1 Titanium dioxide pigments are components with high refractive index that significantly influence the opacity, color, durability, and other properties of coatings. This test method for determining titanium dioxide content is quicker and easier to use than Test Methods D 1394, a wet chemical analysis method for pigments. It is conveniently applicable to single samples and to large numbers of samples. Only a single relatively stable reagent used to prepare standards and paints under test need be stored. Drawdown specimens used as standards, once prepared, can be stored indefinitely and used repeatedly.

5. Apparatus

5.1 *X-ray Fluorescence Spectrometer*, suitable for measurement of the baseline corrected intensity of the $K\alpha$ lines of titanium and of bromine. For spectrometers equipped with a tungsten or chromium target and a lithium fluoride (200) analyzing crystal, these lines are observed at 2θ angles of 86.09° and 29.97° , respectively.

NOTE 1—Follow the recommendations of the manufacturer of the instrument used. Use operating conditions that ensure that the count rate for bromine and titanium are within the linear response range of the detector. Although the base operating conditions and count rate may be different for different instruments, these differences will not affect the outcome of the analysis.

5.2 Paint Shaker.

5.3 *Film Applicator*, to produce a 3-mil (75- μm) wet film thickness.

5.4 *Plastic Sheet*,⁶ with no interfering X-ray fluorescence peaks and not attacked by paint solvent.

5.5 *Perforated Suction Plate*, or other flat surface.

5.6 *Vials and caps*, 20 mL.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests, unless otherwise specified. It is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society

¹ This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Feb. 26, 1988. Published April 1988.

² *Annual Book of ASTM Standards*, Vol 06.02.

³ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 15.05.

⁶ Leneta P-121-10N dull black plastic panels 6½ by 17 in. by 10 mils (165 by 432 by 0.25 mm) in size, available from Leneta Co., P.O. Box 576, Ho-Ho-Kus, NJ 07423, have been found suitable for this purpose.

where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that conforms to the requirements of Type II of Specification D 1193.

6.3 *2-ethoxyethanol*, purified grade.

6.4 *Tetraethylammonium Bromide*, internal standard solution—Into a 100-mL volumetric flask weigh approximately 10.80 g of tetraethylammonium bromide (TEAB) to 0.1 mg. Dissolve the TEAB in 2-ethoxyethanol, fill the flask to the mark with 2-ethoxyethanol, and mix thoroughly. (TEAB dissolved in 2-ethoxyethanol can be used as an internal standard for both water-reducible and solvent-reducible paint. However, water may be substituted for 2-ethoxyethanol, if only water-reducible paint is to be analyzed.)

Hazards

7.1 As exposure to excessive quantities of x-radiation is injurious to health, X-ray producing equipment can be dangerous to both the operator and persons in the immediate vicinity unless safety precautions are strictly observed. Therefore, users should avoid exposing any parts of their bodies, not only to the direct beam, but also to secondary or scattered radiation that occurs when an X-ray beam strikes or passes through any material. It is strongly recommended that users check the degree of exposure by film carried on them or by the use of dosimeters and that blood counts be made periodically. Before utilizing the equipment, all persons designated or authorized to operate X-ray instrumentation or supervise its operation, should have a full understanding of its nature and should also become familiar with established safe exposure factors by a careful study of the National Bureau of Standards Handbook "X-ray Recommendations of the International Roentgen Ray Committee on X-ray Protection,"⁸ the manufacturer's instruction manual, and other standard publications on the subject. Inquiries should be made of state agencies as to existing requirements.

Specimen Preparation

8.1 Thoroughly mix and sample each paint in accordance with Practice D 3925. Prepare duplicate specimens of the standard paint, which contains a known concentration of titanium dioxide, and each test paint using the following procedure. Into a 20-mL vial weigh approximately 7.00 g of paint to 1 mg. Pipet 2 mL of TEAB internal standard solution (see 6.4) into the paint and thoroughly mix.

8.2 Place a plastic sheet on the perforated suction plate. On the plastic sheet make a 3.0-mil (75- μ m) thick drawdown of the liquid paint. Air dry the drawdown film overnight. Cut

TABLE 1. Instrument Conditions

	Ti K α	Br K α
Analyzer crystal	LIF	LIF
Counter	Flow or Scintillation	Scintillation
Collimator	Fine	Coarse
Order	First	First
Tube KV/ma	10/5	50/24
Time, s	20	20
Peak, 2 θ , °	86.00	29.97
Background, 2 θ , °	85.00	29.00

from the drawdown on the plastic sheet a disk of a size appropriate for the specimen holder of the spectrometer.

8.3 Determine the percent nonvolatile content of each paint at 110°C in accordance with Test Method D 2369 if the information is not already available.

9. Procedure

9.1 Turn on the spectrometer and allow it to stabilize thoroughly before beginning collection of results. With the aid of the manufacturer's literature, select spectrometer settings to permit measurement of the intensity of the K α peaks of titanium and bromine. For instruments equipped with an X-ray tube with a tungsten or chromium target, the settings prescribed in Table 1 may be used as guidelines (Note 1).

9.2 Place the disk in the specimen holder in such a way that the coated side of the sheet is turned toward the incident X-rays. Before measuring the specimens, record the background spectrum in the ranges of the K α peaks of titanium and bromine, including at least 10° on each side. Check the spectra for the presence of interfering peaks and select for each peak an angle for background measurement where interference is at a minimum.

9.3 At the selected angles measure the background intensities and the intensities of the K α peaks of titanium and bromine in counts per second (c/s). Determine the net intensity, c/s, of each peak by subtracting the background count from the peak count.

10. Calculation

10.1 Use the values of the standard paint of known TiO₂ content to calculate K_r , the reference intensity ratio of titanium relative to bromine in the film, defined as:^{9,10}

$$K_r = \frac{\frac{B}{B+N}}{\frac{T}{T+N}} \times \frac{I_b}{I_t} = \frac{B}{T} \times \frac{I_t}{I_b}$$

where:

B = TEAB used in making film, g,

= TEAB used, g, $\times \frac{2 \text{ mL}}{100 \text{ mL}}$ = TEAB used, g, $\times \frac{1}{50}$

T = TiO₂ in film, g,

= paint used, g, $\times$ (% TiO₂ in dry paint/100) $\times$ % nonvolatile matter/100,

= paint used, g, $\times$ % TiO₂ in liquid paint/100,

⁷ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

⁸ Available from National Bureau of Standards, National Center for Standards, Gaithersburg, MD 20899.

⁹ Chung, F. H., "A New Approach to Quantitative Multi-element X-ray Fluorescence Analysis," *Advances in X-ray Analysis*, 19, 1976, p. 81.

¹⁰ Chung, F. H., Lentz, A. J., and Scott, R. W., "A Versatile Thin Film Method for Quantitative X-ray Emission Analysis," *X-ray Spectrometry*, 3(4), 1974, p. 172.

= paint used, g, $\times$ weight fraction of TiO_2 in liquid paint,
 N = nonvolatile matter (from paint) in film, g,
 = paint used, g, $\times$ % nonvolatile matter/100,
 = paint used, g, $\times$ % weight fraction of nonvolatile matter,
 I_b = intensity of bromine peak, c/s, and
 I_t = intensity of titanium peak, c/s.
 Therefore

$$K_t = \frac{B}{W} \times \frac{1}{50X} \times \frac{I_t}{I_b}$$

where:

W = paint used, g, and

X = weight fraction of TiO_2 in liquid paint.

Calculate the mean of the K_t values obtained from the two disks coated with the standard paint and use in the calculation in 10.2.

10.2 Calculate the percent TiO_2 in the test paint, P , as follows:

$$P = \frac{2 \times B \times I_t}{K_t \times W \times I_b}$$

where the symbols are defined as in 10.1.

10.3 Sample Calculation:

10.3.1 Reference intensity ratio, K_t ,

Standard paint—12.8 % TiO_2 in liquid paint,

Standard solution—10.347 g TEAB in 100-mL solution,

Film—2 mL of standard solution in 7.25 g of liquid standard paint, and

X-ray intensity: $I_b = 4396$ c/s, $I_t = 1804$ c/s.

$$K_t = \frac{10.347}{7.252} \times \frac{1}{(50)(0.128)} \times \frac{1804}{4396} = 0.0915$$

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The mean of duplicate analyses is $K_t = 0.0916$.

10.3.2 TiO_2 content of test paint,

Data:

Film—2 mL of standard solution in 7.186 g wet paint, and

X-ray intensity: $I_b = 4272$ c/s, $I_t = 2569$ c/s

$$P = \frac{10.347}{7.186} \times \frac{2}{0.0916} \times \frac{2569}{4272} = 18.9 \%$$

11. Precision and Bias

11.1 In an interlaboratory study of this test method, one operator in each of five laboratories analyzed on two days two samples of latex house paint and two samples of solvent-reducible house paint. The mean weight percent titanium dioxide in samples Number 1 through 4 was, respectively, 3.64, 19.1, 7.44, and 15.8 %. The pooled within-laboratory coefficient of variation was 2.39 % with 19 degrees of freedom and the pooled between-laboratories coefficient of variation 4.47 % with 15 degrees of freedom, after discarding one duplicate value from one laboratory because the range differed significantly from all other duplicate ranges for paint sample Number 2. Based on these coefficients of variation, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 7.08 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 13.5 % relative.



Standard Practice for Continuity Verification of Liquid or Sheet Linings Applied to Concrete Substrates¹

This standard is issued under the fixed designation D 4787; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This practice covers procedures that may be used to allow the detection of discontinuities in nonconductive linings with thicknesses exceeding 20 mils (0.5 mm) applied to concrete substrates.

1.2 Discontinuities may include pinholes, internal voids, holidays, cracks, and conductive inclusions.

1.3 This practice utilizes high-voltage spark testing in conjunction with a continuous conductive membrane.

NOTE—For further information on discontinuity testing refer to Nace standard RP0188-88.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see Section 6.*

Referenced Documents

2.1 ASTM Standards:

D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies²

2.2 NACE Standards:³

RP0188-88 NACE Standard Practice Discontinuity (Holiday) Testing of Protective Coatings

Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *discontinuity*—a localized lining site that has a dielectric strength less than a determined test voltage.

3.1.2 *conductive underlayment*—a continuous layer applied to the prepared concrete surface prior to the application of a nonconductive lining layer(s) that will allow high voltage spark testing for discontinuities in the lining.

3.1.3 *spark-over*—at a certain electrical voltage and distance from a grounded surface a spark from a high-voltage testing instrument will jump across the space.

3.1.4 *high voltage spark tester*—a high voltage electrical detector used to locate discontinuities in nonconductive

linings applied to conductive substrates.

3.1.5 *test voltage*—that electrical voltage established which will allow a discontinuity at the thickest lining location site to be tested, but which will not damage the lining. The test voltage must always be set well below the dielectric breakdown strength of the lining. This voltage should normally be recommended by the lining manufacturer. The dielectric breakdown voltage strength of a solid can be determined by Test Method D 149.

4. Summary of Practice

4.1 This practice allows for high voltage electrical detection of discontinuities in new linings applied to concrete substrates through the utilization of a continuous conductive underlayment applied to the prepared concrete surface prior to the application of the nonconductive lining layer(s).

5. Significance and Use

5.1 The electrical conductivity of concrete is primarily influenced by the presence of moisture. Other factors which affect the continuity of concrete include the following:

- 5.1.1 Presence of metal rebars,
- 5.1.2 Cement content and type,
- 5.1.3 Aggregate types,
- 5.1.4 Admixtures,
- 5.1.5 Porosity,
- 5.1.6 Above or below grade elevation,
- 5.1.7 Indoor or outdoor location,
- 5.1.8 Temperature and humidity, and
- 5.1.9 Age of concrete.

5.2 The electrical conductivity of concrete itself may be successfully used for high-voltage continuity testing of linings applied directly with no specific conductive underlayment installed. However, the voltage required to find a discontinuity may vary greatly from point to point on the structure. This variance may reduce the test reliability to an unacceptable level.

5.3 Although the most common conductive underlayments are liquid primers applied by trowel, roller, or spray, and which contain carbon or graphite fillers, others may take the form of the following:

- 5.3.1 Sheet-applied graphite veils,
- 5.3.2 Conductive polymers,
- 5.3.3 Conductive graphite fibers,
- 5.3.4 Conductive metallic fibers, and
- 5.3.5 Conductive metallic screening.

5.4 Liquid-applied conductive underlayments may be desirable as they can serve to address imperfections in the concrete surface and provide a better base for which to apply the lining.

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

Current edition approved Sept. 30, 1988. Published November 1988.

² Annual Book of ASTM Standards, Vol 10.01.

³ Available from National Association of Corrosion Engineers, P.O. Box 218340, Houston, TX 77218.

5.5 This practice is intended for use with new linings applied to concrete substrates. It may also be applicable for linings previously tested by this practice and subsequently placed in service. However, consideration must be given to potential changes to the electrical properties of the conductive and nonconductive layers comprising the system.

5.6 The user may consider this practice when performance requirements of the lining in a specified chemical environment requires assurance of a lining free of discontinuities.

5.7 Factors affecting the dielectric properties and test voltage shall be considered. Some factors are the curing time of liquid-applied linings; the possible presence of electrically conductive fillers or solvents, or both; the possible presence of air inclusions or voids; and the compatibility of conductive underlayments with the specified lining.

6. Apparatus

6.1 *High Voltage Spark Tester*—An electrical detector with a voltage rating in excess of 900 V. The detector is to consist of an electrical energy source, an exploring electrode, a ground connection, and ground wire. The detector shall be equipped with a visual or audible indicator, or both.

6.1.1 *Electrical Energy Source*—Either a-c, d-c, or pulsating d-c type with the appropriate test voltage.

6.1.2 *Exploring Electrode*—The full length shall be capable of maintaining continuous contact with the surface being inspected.

6.1.3 *Ground Wire*, stranded 14 to 16 gage copper wire.

6.1.4 *Visual or Audible Indicators*, or both, to signal a closed electrical circuit. Such signals shall be essential for testing the underlayment for electrical conductivity and for exposing discontinuities in the lining after it has been applied.

7. Hazards

7.1 Solvents retained in the applied conductive underlayment may create an explosive environment as well as produce erroneous results.

8. Conductive Underlayments

8.1 The conductive underlayment shall not rely on the concrete substrate's electrical properties.

8.2 The specified lining shall be compatible with the specified conductive underlayment.

8.3 Application:

8.3.1 The finished conductive underlayment surface shall be relatively smooth. The conductive underlayment shall be considered part of the lining system and must be installed in accordance with the manufacturer's latest published instructions.

8.3.2 Visually verify that the conductive underlayment covers the entire area to be lined. Breaks at expansion joints and construction joints are allowable unless otherwise specified.

8.4 Verification of Underlayment Conductivity:

8.4.1 The surface of the applied conductive underlayment shall be clean, dry, free of oil, grease, dirt, or other contaminants and be sufficiently cured in accordance with the manufacturer's latest published instructions at the time

the high-voltage spark testing is performed. (Warning—See Section 7.)

8.4.2 Verify the operation of the high-voltage test instrument in accordance with Section 9.

8.4.3 Adjust the high-voltage test instrument in accordance with Section 10.

8.4.4 Ground the test instrument to the installed underlayment or other appropriate ground. If electrical isolation across an expansion joint is encountered, the ground wire must be moved to an appropriate ground in the same section being tested.

8.4.5 Place the exploring electrode on a nonconductive spacer so that an air gap between the surface of the underlayment and the electrode is equal to the maximum thickness of the lining.

8.4.6 The underlayment is conductive if a spark-over is emitted and the visual or audible indicator, or both, on the test instrument is activated.

8.4.7 Test Sampling:

8.4.7.1 A minimum of four test points shall be used for the first 100 ft.² Test points shall be approximately equally spaced within the test area. At least one additional test point shall be used for every 500 ft.² thereafter.

8.4.7.2 Test points most distant from the ground connection shall be included in the test sampling.

8.4.8 The specified lining shall not be applied until the conductivity of the underlayment has been verified.

9. Verifying Operation of High-Voltage Spark Tester

9.1 Test electrical source for proper voltage output.

9.2 Follow the equipment manufacturer's operating instructions for verifying the operation of the high voltage tester.

9.3 If the tester fails to signal, it shall be considered defective.

10. Adjustment of High-Voltage Spark Tester for Verifying Conductivity of Underlayment

10.1 Establish the test voltage based on the maximum specified thickness of the nonconductive lining, its dielectric strength, and the lining manufacturer's recommendations.

10.2 Following the equipment manufacturer's instructions, set and check the test voltage established in 10.1.

11. Application of the Lining or Coating

11.1 Apply the specified lining in accordance with governing specifications.

12. Adjustment of High Voltage Spark Tester for Verifying Conductivity of the Applied Lining

12.1 Select the proper test voltage to provide reliable spark-over to locate a holiday under normal test conditions. The voltage selected must jump an air gap equal to the maximum specified dry film thickness of the lining being tested and not arc through the lining at the minimum specified dry film thickness.

12.2 Adjust the tester to the test voltage established in 12.1 as follows:

12.2.1 Connect a high-voltage voltmeter or a spark-gap calibrator between the electrode and the ground wire.

12.2.2 Switch the detector to the ON position.

12.2.3 Perform field checking of the test voltage with the electrode placed against the surface of the lining since the exploring electrode voltage may be reduced by the slight current flow to the lining.

12.2.4 If required, compare measured voltage with the selected test voltage. Depending on the type of tester, adjust to the selected voltage $\pm 5\%$.

12.2.5 Switch the detector to the OFF position.

12.2.6 Disconnect the voltmeter or spark-gap calibrator.

3. High Voltage Spark Testing for Verifying Continuity of Applied Lining

13.1 The surface of the applied lining shall be clean, dry, free of oil, grease, dirt, or other contaminants and be sufficiently cured in accordance with the manufacturer's latest published instructions at the time the high-voltage

spark testing is performed (**Warning**—See Section 7).

13.2 Attach the ground wire from the instrument ground terminal to the conductive underlayment or appropriate ground in the same manner as was required in 8.4.4. Make contact with the exploring electrode at a known discontinuity to verify that the instrument is properly grounded. For each ground location, make contact with a known discontinuity. Conduct this test periodically during the test.

13.3 With the exploring electrode in continuous contact with the lining surface, move it over the entire surface of the lining at a rate of 1 ft/s (0.3 m/s) maximum in a sweeping motion with overlapping passes to ensure that the entire surface has been subjected to the test.

13.4 Identify discontinuities that require repair with a compatible marker.

13.5 Completely test the lining one time only. Repair all defects found in the lining and retest only those repaired areas.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Bond Strength of Thermoplastic Traffic Marking Materials¹

This standard is issued under the fixed designation D 4796; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method provides an instrumental means for the determination of thermoplastic traffic marking material bond strengths using cement bricks and steel cubes.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 109 Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)²

C 881 Specification for Epoxy-Resin-Base Bonding Systems for Concrete³

D 883 Definition of Terms Relating to Plastics⁴

E 284 Terminology Relating to Appearance of Materials⁵

F 412 Terminology Relating to Plastic Piping Systems⁶

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Definitions D 883, E 284, and F 412, unless otherwise indicated.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *cement brick*—a brick formed by mixing cement and fine sand together and allowing to harden.

3.2.2 *thermoplastic*—traffic marking (same as 3.2.3).

3.2.3 *thermoplastic traffic marking*—a highly filled 100 % total solids highway marking system that when heated to a molten state can be extruded or sprayed onto a road surface and when cooled forms a solid durable delineator.

4. Summary of Test Method

4.1 The thermoplastic specimen is prepared for this test by first melting a sample to its application temperature under continuous agitation. The specimen is then applied to the specified cement brick by a hot drawdown blade. Two

steel cubes are then immediately placed onto the hot thermoplastic line and the excess thermoplastic trimmed away from around the two steel cubes. After the trimming is complete, the steel cubes are removed. A heated steel cube is bonded with epoxy to the square of thermoplastic and allowed to cure overnight before determining the bond strength on a Dillon dynamometer or similar device.

5. Significance and Use

5.1 The function of this test method is to provide numerical instrumental results indicating the cohesive or adhesive, or both, bond strength of thermoplastic traffic marking to a specified cement brick substrate.

5.2 The use of this test method allows the user and manufacturer to control the quality of the product and make inferences about the performance of the thermoplastic traffic marking product. Results from these tests also provide information helpful in researching and developing thermoplastic traffic marking materials.

5.3 Strict adherence to the procedures outlined is necessary for precision of the test method. Under no conditions should the bond strength be accepted unless there is conformance to 9.14. Precise results are obtained only when one steel block is epoxied to the thermoplastic traffic marking on the cement brick.

6. Types of Separation in Bond Strength Tests

6.1 *Thermoplastic to Steel Cube Separation*—This type of separation occurs where there is an insufficient bond between the thermoplastic and steel cube probably due to insufficient coverage of the epoxy adhesive.

6.2 *Thermoplastic to Thermoplastic Separation*—This type of separation is caused by internal cohesive failure of the thermoplastic. This separation is acceptable when it exceeds the specified bond strength.

6.3 *Thermoplastic to Cement Brick Separation*—This type of separation is caused by the failure of the bond between the thermoplastic specimen and the cement brick.

6.4 *Cement Brick to Cement Brick*—This type of separation is caused by the internal cohesive failure of the brick. This is due, in most cases, to a bond between the thermoplastic and cement brick that exceeds the cohesive strength of the cement brick. This separation is not acceptable when the bond strength values are lower than specified.

7. Apparatus

7.1 *Agitator Blade*, 6 in. (15 cm) long with a 1/2-in. (1-cm) steel shaft and a 1 3/4 by 1 in. by 1/8-in. (4.5 by 2.5 by 0.3-cm) straight horizontal steel blade.

7.2 *Capped Bolts*, two, 3/8 in. (16 mm) in size.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved Oct. 31, 1988. Published December 1988.

² Annual Book of ASTM Standards, Vol 04.01.

³ Annual Book of ASTM Standards, Vol 04.02.

⁴ Annual Book of ASTM Standards, Vol 08.01.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Annual Book of ASTM Standards, Vol 08.04.

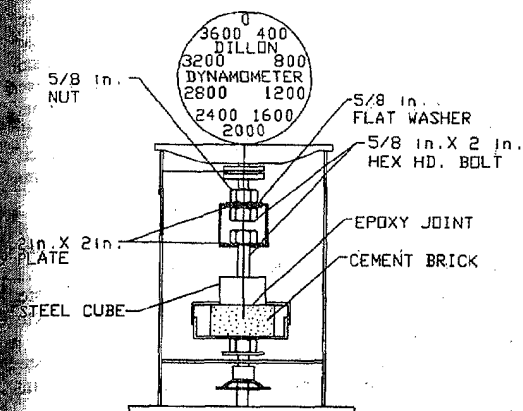


FIG. 1 Bond Strength Testing Apparatus

7.3 *Cement Bricks*, 3½ by 2 by 7½ in. (9 by 5 by 19 cm) in size with a compressive strength of 3000 to 5000 psi (Note 1).

NOTE 1—Concrete bricks conforming to Test Method C 109 have been used but proved more variable due to migration of a thin veneer of cement to the top of the brick making determinations erratic. The cement bricks may be obtained from local block plants. The term "cement" brick is common for the industry and is used in this test method extensively.

NOTE 2—The unit should be fitted with a steel frame to hold the cement brick for testing.

7.4 *Dynamometer*,⁷ with a capacity of 6000 lbs in 25-lb divisions having a pull-rate capability of ¼ in./min. (Note 2) (See Figs. 1, 2, 3, and 4).

7.5 *Draw Down Blade*, 2 by 1 by 4 in. (5 by 2.5 by 10 cm) in size capable of laying down a .125 mil (0.125 in.) wet thermoplastic film 2 in. wide.

7.6 *Drill Press*, or other apparatus capable of agitating the thermoplastic during meltdown to the application temperature at 600 to 800 r/min in the jacketed electric pots.

7.7 *Epoxy Resin and Hardener*, Type I or II, Grade 2, Class C in accordance with Specification C 881.

7.8 *Hot Plate*, capable of maintaining 537°C.

7.9 *Gravity Convection Oven*, capable of maintaining 260°C.

7.10 *Electric Pots, Jacketed*, for heating and melting the thermoplastic to 218°C.

7.11 *Spatulas*, for cutting, stirring, and shaping the thermoplastic.

7.12 *Steel Cubes*, two, 2 by 2 by 2 in. (5 by 5 by 5 cm) in size threaded in the center of one side for a ⅝-in. (16-mm) capped bolt.

8. Sampling

8.1 Samples may be obtained by an appropriate quartering or riffle sampling method where deemed necessary

⁷ A Dillon Dynamometer available from Weigh-Tranex, 1000 Armstrong Drive, P.O. Box 1000, Fairmount, MN 56031, or equivalent, has been found suitable for this purpose.

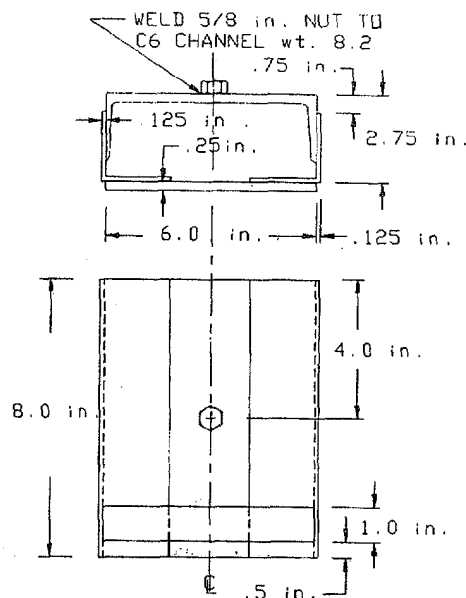


FIG. 2 Tension Bed

considering the physical form of the material.

9. Procedure

9.1 Under continuous agitation melt a specimen of the thermoplastic to be tested to a temperature of 218°C. If the specimen is a dry powder mix, allow the specimen to cool to

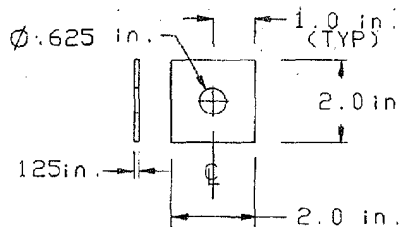
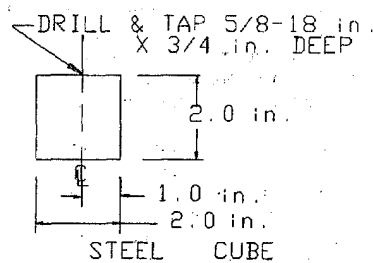


FIG. 3 Load Plate (2 Required)

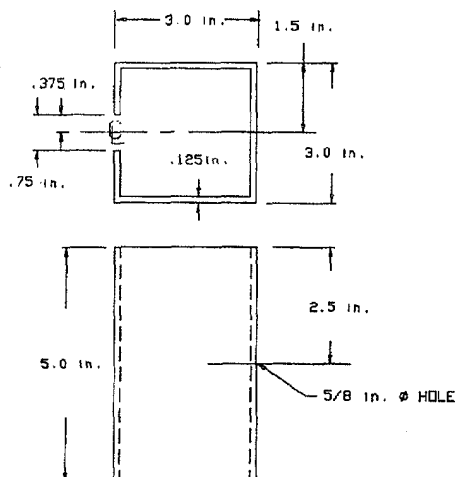


FIG. 4 Coupling Hitch

193°C under continuous agitation and reheat under agitation to 218°C.

NOTE 3—Dry powder mixed thermoplastic must be conditioned to ensure a homogeneous melt necessary for consistent results. Premelted block thermoplastic does not require this conditioning. The specimen may be melted on a hot plate set at 537°C or in a jacketed electric pot. Continuous agitation is necessary to prevent scorching and settling. If the specimen is melted in an oven set at 260°C, the specimen must be agitated every 15 min until 218°C is reached.

9.2 Heat the draw down blade to 218°C.

9.3 Obtain a dry room temperature cement brick that has been brushed or textured on the side to be coated with thermoplastic.

9.4 Heat one 2 by 2 by 2 in. (5 by 5 by 5 cm) steel cube in an oven or on a hot plate to 49°C for 2 h prior to making the thermoplastic draw down on the cement brick.

9.5 When the thermoplastic specimen is melted to 218°C under continuous agitation, remove the agitator blade from the thermoplastic.

9.6 Remove the draw down blade from the hot plate and immediately place it on the cement brick and make the draw down the full length of the brick with the melted thermoplastic on the brushed or rough textured side of the brick.

9.7 Immediately place two room temperature steel cubes on the thermoplastic approximately 1 in. from each end of the brick and trim the plastic from around the two steel cubes before the plastic cools and hardens.

9.8 Remove the steel cubes by hand and allow the thermoplastic to cool for 1 h.

9.9 Prepare the epoxy solution using the proper resin and hardener ratio and mix thoroughly.

9.10 Remove the steel cube heated to 49°C from the oven and place a small amount of epoxy on the heated cube. Place the steel cube on the thermoplastic square and rub to ensure an even coating and good adhesion. To ensure an even coating, remove the steel cube from the thermoplastic square and visually inspect the square and cube and then replace the steel cube on the square and rub. Do not allow any excess epoxy solution to flow from the steel cube and thermoplastic square onto the cement brick.

NOTE 4—Only one steel cube can be epoxied at a time to the thermoplastic on the brick because the shock of the first pull will cause a premature release of the second thermoplastic square.

9.11 Place a weight such as a cement brick on the steel cube epoxied to the thermoplastic square and allow to cure for a minimum time of 8 h.

9.12 Screw the 5/8-in. (15.9-mm) capped bolt into the steel cube epoxied to the thermoplastic and place the brick into the steel frame mounted onto the dynamometer.

9.13 Set the dynamometer to zero and pull the steel cube at 1/4 in./min.

9.14 Two tests are run on each brick. Separations involving at least 80 % of the thermoplastic area to the cement brick, thermoplastic to thermoplastic, and cement brick to cement brick are acceptable for reporting bond strengths.

10. Calculations

10.1 Calculate the bond strength as follows:

10.1.1 Average the readings obtained in 9.13 if the conditions in 9.14 are met.

10.1.2 Divide the average reading obtained by 4. The result is the bond strength in pounds per square inch.

10.1.3 If one of the two tests fails then that test is repeated until the conditions of 9.14 are met.

11. Report

11.1 Report the following information:

11.1.1 The type of separation and bond strength, the batch number, color, and type of thermoplastic.

11.1.2 The determination of the area of separation is subjective. The area not involved in the separation is usually attached to the steel cube or cement brick.

12. Precision and Bias

12.1 *Precision*—No general statement of precision can be made because of lack of sufficient data at this time.

12.2 *Bias*—No statement of bias can be prepared for this test method since there is no absolute method for use as a comparative basis.

12.3 Experience dictates that test results with ± 50 psi are acceptable.

13. Index Terms

13.1 This test method is indexed under the following terms: thermoplastic—traffic marking; bond strength; cement brick.

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Standard Test Methods for Chemical and Gravimetric Analysis of White and Yellow Thermoplastic Traffic Marking Containing Lead Chromate and Titanium Dioxide¹

This standard is issued under the fixed designation D 4797; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover procedures for the chemical and gravimetric analysis of white and yellow thermoplastic traffic marking containing lead chromate and titanium dioxide pigment.

1.2 The analytical procedures appear in the following order:

	Sections
Percent Binder	10
Percent Glass Beads (Note 1)	11
Percent Titanium Dioxide	12
Percent Lead Chromate and Analysis of Chrome Yellow and Chrome Orange Pigments (Note 2)	13

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—Test for 1.50 glass spheres only.

NOTE 2—This modified analysis of chrome yellow and chrome orange pigments must be used because the heat resistant chrome yellows in the thermoplastic cannot be analyzed by Test Method D 126.

2. Referenced Documents

2.1 ASTM Standards:

- D 126 Test Methods for Chemical Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green²
- D 883 Definitions of Terms Relating to Plastics³
- D 1193 Specification for Reagent Water⁴
- D 1394 Test Methods for Chemical Analysis of White Titanium Pigments²
- F 412 Terminology Relating to Plastic Piping Systems⁵

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Definitions D 883 and F 412, unless otherwise indicated.

3.2 Descriptions of Terms Specific to This Standard:

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Oct. 31, 1988. Published December 1988.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vols 08.01 and 08.04.

⁴ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁵ Annual Book of ASTM Standards, Vol 08.04.

3.2.1 *ash*—the inorganic components of thermoplastic traffic marking including the pigment, glass spheres, and filler.

3.2.2 *binder*—the organic components of thermoplastic traffic marking that bind the pigments, glass spheres, and filler together as a unit.

3.2.3 *filler*—the inorganic components of thermoplastic traffic marking not including the pigments or glass spheres.

3.2.4 *pigment*—titanium dioxide and lead chromate colorants.

3.2.5 *thermoplastic*—See *thermoplastic traffic marking*.

3.2.6 *thermoplastic traffic marking*—a highly filled 100 % total solids highway marking system that when heated to a molten state can be extruded or sprayed onto a road surface and when cooled forms a solid durable delineator.

4. Summary of Test Method

4.1 The thermoplastic material is prepared for the described test methods by melting a sample to its application temperature under continuous agitation. The specimen is then poured into round patties on a clean tin plate or baking pan. The patties are then broken into pieces for ignition in a muffle furnace. The percent binder is calculated from the ashed specimen and the various tests for glass spheres, titanium dioxide, and lead chromate pigment are performed on the ashed residue. The tests for pigment type or glass spheres may be run on the same ashed specimen. Specimen selection and preparation are the same for each sample tested.

5. Significance and Use

5.1 The function of these test methods is to define the percent of binder, glass, titanium dioxide, and lead chromate present in the composition of the thermoplastic traffic marking as defined by the applicable specification for the manufacture of a specific thermoplastic traffic marking.

6. Apparatus

- 6.1 Balance, analytical, accurate to 0.1 mg.
- 6.2 Buret, 10 mL, 0.1-mL divisions.
- 6.3 Buret, 50 mL, 0.1-mL divisions.
- 6.4 Crucibles, 30 mL, porcelain.
- 6.5 Desiccator.
- 6.6 Erlenmeyer flask, 500 mL.
- 6.7 Furnace (Muffle), capable of maintaining 1100°C.
- 6.8 Hot plate, capable of maintaining 537°C.
- 6.9 Jones Reductor.

6.10 *Mortar and Pestle*, glazed ceramic or other impervious type.

6.11 *Oven*, capable of maintaining 260°C.

6.12 *Sieve*, 3 in., 45- μ m (No. 325) (metal).

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

7.3 *Alcohol*—Ethyl alcohol 95 %.

7.4 *Potassium Chloride* (KCl).

7.5 *Potassium Hydroxide Solution*—Dissolve 50 g of potassium hydroxide (KOH) in 100 mL of freshly distilled water.

7.6 *Potassium Iodide* (KI).

7.7 *Potassium Permanganate Solution* (KMnO₄) (0.1N).

7.8 *Sodium Thiosulfate, Standard Solution* (Na₂S₂O₃ · 5H₂O) (0.1 N).

7.9 *Starch (Soluble) Indicator Solution*—Dissolve 10 g of soluble starch in 1 L of deionized or distilled water.

7.10 *Hydrochloric Acid Solution* (HCl) (1+2).

7.11 *Hydrochloric Acid, Concentrated* (HCl).

8. Sampling

8.1 Samples may be obtained by an appropriate quartering or riffle sampling method where deemed necessary considering the physical form of the material.

9. Preparation of Specimens

9.1 Melt a sample of approximately 1000 g of thermoplastic traffic marking to 210 to 218°C under continuous agitation on a hot plate set at 537°C or stir every 15 min in an oven set at 260°C.

9.2 Flow the sample out on a smooth clean surface and allow it to cool to room temperature. Patties 1/8 in. (3 mm) thick will facilitate breaking up specimens for the described analysis.

9.3 Break the specimen into small pieces and weigh 10 g to the nearest 0.1 mg into a 30-mL weighed crucible.

9.4 Cover the crucible and place into a muffle furnace preheated to 540°C and ash for 1 h or until no carbonaceous materials remain.

9.5 Remove the crucible with the ashed remains of the specimen and place into a desiccator and cool to room temperature.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

10. Percent Binder

10.1 Procedure:

10.1.1 Weigh the crucible and ash (see Section 9) to the nearest 0.1 mg and calculate the percent of organic binder *D* as follows:

$$D = [1 - (S/W)] \times 100$$

where:

S = ashed weight of thermoplastic specimen, g, and

W = weight of thermoplastic specimen, g.

11. Percent Glass Beads

11.1 *Interferences*—Acid-insoluble fillers will affect the glass-sphere analysis and must be removed by some physical separation method or accounted for quantitatively, or both.

11.2 Procedure:

11.2.1 Weigh the crucible and ash (see Section 9) to 0.1 mg and calculate the percent ash.

11.2.2 After the ashed material has been weighed, transfer the ash to a mortar and pestle and grind with minimal pressure to reduce the ash to a fine grained consistency without crushing the beads. Carefully transfer the ashed contents into a 400-mL beaker.

11.2.3 Add to the ash approximately 150 mL of cold 1+2 HCl and stir occasionally until the effervescence has ceased completely.

11.2.4 Place the beaker on a hot plate preheated to approximately 260°C and boil for 20 min under continuous agitation to dissolve all acid-soluble filler pigments.

11.2.5 Remove the beaker from the hot plate, and while hot, immediately dilute the contents with 150 mL of cold water. Allow the beads to settle. Decant the water carefully so as not to lose any glass beads.

11.2.6 Continue diluting with 150-mL aliquots of water and decanting until the water is nearly clear. Then transfer the residue into a weighed 3-in. 45- μ m (No. 325) sieve and wash with 500 mL of cold water.

11.2.7 Dry the sides and the bottom of the sieve with a paper towel and dry for 1 h in a gravity oven preheated to 100°C.

11.2.8 Place the sieve in a desiccator and cool to room temperature. Weigh the sieve and glass beads to 0.1 mg and calculate the percent beads *G* as follows:

$$G = (B/W) \times 100$$

where:

B = weight of the glass beads, g, and

W = weight of thermoplastic specimen, g.

12. Percent Titanium Dioxide

12.1 Procedure:

12.1.1 Weigh the crucible and ash (see Section 9) to 0.1 mg and calculate the percent of ash which is also equal to the percent filler (pigments and beads).

12.1.2 After weighing the above ashed materials, carefully transfer the ash to an agate or similar impervious-type mortar. Grind the ash to a consistent fine powder except for the glass beads. The pulverized ash must be stirred each time specimens are taken to weigh the prescribed amount for the analysis.

12.1.3 Weigh 1 ± 0.1 g of ash for the titanium analysis.

12.1.4 Proceed with the titanium analysis as described in Test Methods D 1394. The Jones Reductor or aluminum reduction method may be used.

12.1.5 At the end of the digestions described in Test Methods D 1394, filter the undigested materials through a 3-in. 45- μ m (No. 325) sieve with hot water. This will prevent clogging of the Jones Reductor. This step is not necessary if the aluminum reduction method is used.

12.1.6 Calculate the percent of titanium dioxide (TiO_2) from the total ash weighed. The result gives the percent of TiO_2 in the total thermoplastic specimen.

12.1.7 Calculate the percent ash A as follows:

$$A = (S/W) \times 100$$

where:

S = weight of ash, g, and

W = weight of thermoplastic specimen used, g.

12.1.8 Calculate the percent of total TiO_2 in the T based on percent ash:

$$T = 0.08 AMN/CS_a$$

where:

A = ash (pigment + filler + beads), %,

S_a = weight of ash used, g,

M = amount of 0.1 N potassium permanganate (KMnO_4) solution used, mL, (see the paragraph on titration with KMnO_4 solution of the Procedure section of the Jones Reductor Method of Test Methods D 1394),

N = normality of KMnO_4 used,

C = correction factor for TiO_2 pigment used (usually 1.0). This represents the purity of the pigment if specified, and

0.08 = weight of TiO_2 per 1 mL of 1 N KMnO_4 , g.

13. Percent Lead Chromate

13.1 Procedure:

13.1.1 Weigh the crucible and ash (see Section 9) to 0.1 mg and calculate the percent of ash.

13.1.2 After weighing the above ashed materials, carefully transfer the ash to an agate or similar impervious type mortar and grind the ash to a consistent fine powder except for the glass beads. The pulverized ash must be stirred when specimens are taken to weigh the prescribed amount for the analysis.

13.1.3 Weigh 1 ± 0.1 g of the ash for the lead chromate (PbCrO_4) analysis.

13.1.4 Analyze the dry ash as follows:

13.1.4.1 Transfer the ashed specimen to a 500-mL Erlenmeyer flask. Add 6 mL of the (KOH) solution (see 7.5) and 10 mL of distilled water. Heat until dissolved. Do not allow to dry. To the hot solution add 50 g of KCl. Dilute with water to 200 mL and swirl until dissolved. Add 16 mL of HCl slowly. Add 10 g of KI and titrate with the standard sodium thiosulfate solution (see 7.7) using the starch solution (see 7.8) as the indicator. Titrate to a green endpoint.

NOTE 3—A 10-mL buret may be used since the titrant required is small for most thermoplastic analysis.

13.1.5 Calculate the percent ash A as follows:

$$A = (S/W) \times 100$$

where:

S = weight of gas, g, and

W = weight of the thermoplastic specimen used, g.

13.1.6 Calculate the percent of total PbCrO_4 in the sample (binder + filler) L based on percent ash as follows:

$$L = 0.1077 AMN/CS_a$$

where:

A = ash (pigment + filler + beads), %,

C = correction factor for PbCrO_4 pigment used (for example, 90 to 100 %, normally 1.0). This represents the purity of the pigment if specified,

M = amount of 0.1 N sodium thiosulfate solution used, mL,

N = normality of sodium thiosulfate used, and

S_a = weight of ash used, g.

14. Precision and Bias

14.1 No general statement of precision can be made because of lack of sufficient data at this time.

14.2 No statement of bias can be prepared for this test method since there is no absolute method for use as a basis for comparison.

15. Index Terms

15.1 This test method is indexed under the following terms: glass spheres (in traffic paint); lead chromate; titanium dioxide; thermoplastic traffic marking material.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Practical Washability of Organic Coatings¹

This standard is issued under the fixed designation D 4828; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the relative ease of removal of common soil and stains from interior coatings by manual or mechanical washing with a sponge and a liquid or powder cleanser.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 3450 Test Method for Washability Properties of Interior Architectural Coatings³

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials³

3. Summary of Test Method

3.1 The test material is applied to a plastic panel and allowed to dry for 7 days or as mutually agreed. Soilants or stains, or both, are applied to the film. The film is washed for up to 100 cycles using a sponge and a liquid or powder cleanser. After rinsing and drying, the panel is evaluated for the degree of soil or stain removal, difference in erosion, and any change in gloss/sheen or color between the washed and unwashed area.

4. Significance and Use

4.1 Interior architectural paints are subjected in use to soiling by dirt or other stains. This test method provides a way to assess relative ease of soil or stain removal from a paint film using materials common to households. This test method includes a way to evaluate the film for washability properties and changes in appearance. Thus, a formulator may evaluate the effects of composition on the washability properties of a paint. Users may also compare the ease of soil removal from difference paints that are tested, preferably at the same time.

5. Apparatus

5.1 *Sponge and Holder.*⁴

5.2 *Soil and Stain Applicator* (see Fig. 1).

5.3 *Weight*, 100 g.

5.4 *Balance*, weighing accurately to 0.1 g.

5.5 *Doctor or Bird Film Applicator*, having a 7-mil (0.18-mm) clearance by 6-in. (150-mm) film width.

5.6 *Glass Plate*, 17½ by 6½ by ¼ in. (455 by 165 by 6.3 mm).

5.7 *Washability Machine.*⁵

5.8 *Black Plastic Panels.*⁶

5.9 *Masking Tape.*

5.10 *Straightedge*, approximately 17 in. (430 mm) in length.

5.11 *Cotton Tipped Swabs.*

5.12 *Medicine Droppers.*

5.13 *Suction Plate*, for drawdowns.

6. Reagents and Materials

6.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.2 *Soil and Staining Medium*—Examples found acceptable for use with this test method include, but are not limited to pencil, crayon, mercurochrome, ball-point pen, waterborne felt-tip markers, lipstick and mineral-oil-borne soilant as outlined in Test Method D 3450.

6.3 *Cleaning Media*—Examples found acceptable for use with this test method include, but are not limited to, commercial liquid cleansers, laboratory standardized liquid cleaners as outlined in Test Method D 3450, and powder cleansers. Powder cleansers containing chlorine may effect the color of the washed portion of the test panel.

7. Preparation of Apparatus

7.1 *Washability Machine*—Level the apparatus before use and operate at 37 ± 1 cpm. (A cycle consists of a complete forward and reverse stroke.)

7.2 *Sponge and Holder*—Add sufficient weight to the

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 4828 - 88. Last previous edition D 4828 - 88.

² *Annual Book of ASTM Standards*, Vol 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ A sponge, 3 by 3¼ by 1¼ in. (75 by 95 by 45 mm), Part No. AG-8116, and a metal holder, Part No. AG-8115, available from BYK-Gardner, Inc., 2435 Linden Lane, Silver Spring, MD 20910 or a sponge, Part No. WA 2222, and metal holder, Part No. WA 2220, available from the Paul N. Gardner Co., 316 N. E. First Street, Pompano Beach, Florida 33060-6699 have been found acceptable for this purpose. An equivalent may be used.

⁵ Washability machine, Model AG-8100, available from BYK-Gardner, Inc. or Model WA 2037D, available from the Paul N. Gardner Co., have been found suitable for this purpose. Other straight-line wash testers may be adapted to meet the requirements of this test method.

⁶ Leneta P-121-10N dull black plastic panels, 17 by 6½ in. by 10 mils (430 by 165 by 0.25 mm), available from the Leneta Co., P.O. Box 86, Ho-Ho-Kus, N.J. 07423, are suitable for this purpose. An equivalent may be used.

prior to 100 cycles, stop and record the number of cycles before proceeding to 8.8.

8.7 Mechanical Method:

8.7.1 Place the sponge and holder at one end of the panel so that its long axis is parallel to the length of the panel (see Fig. 2). Attach to the cable of the washability machine. Allow the sponge to travel a maximum of 100 cycles. If all the soils or stains are removed prior to 100 cycles, stop and record the number of cycles before proceeding to 8.8.

8.8 Remove the test panel and evaluate the condition of each soil or stain in the path of the sponge and rate as follows:

- 0—No change from original intensity (depth) of soil or stain,
- 3—Slight change from original, but readily visible,
- 5—Moderate change from original, slightly visible,
- 7—Large change from original, barely visible, and
- 10—All soil and stain removed.

If a difference exists between a pair of lines, report the lower rating of the pair. When a soil or stain is removed prior to 100 cycles, assign a rating of 10 and note the number of cycles in which each soilant or stain was removed.

8.9 Rinse the panel with running tap water and remove any clinging particles of cleaning medium by gently moving the palm of the hand over the path the sponge followed during the washing cycle. Blot the panel and allow to dry. Rate the washed area relative to the unwashed areas for gloss or color change and erosion as given below:

Gloss Change	Color Change	Erosion
N—None	N—None	N—None
I—Increase	SD—Slightly darker	S—Slight
II—Large increase	CD—Considerably darker	M—Moderate
D—Decrease	SL—Slightly lighter	
DD—Large decrease	CL—Considerably lighter	

9. Report

9.1 Report the following information:

- 9.1.1 Type of soilant(s), stain(s), washing medium(s) and washing method used and the results obtained in 8.8 and 8.9,
- 9.1.2 Any soils or stains that were removed in less than 100 cycles, and
- 9.1.3 Any deviation from the recommended procedure.

10. Precision and Bias

10.1 *Precision*—On the basis of interlaboratory tests in which operators in seven laboratories using the manual washing method, tested four coatings including solvent- and waterborne systems covering a range of pigment volume concentrations, the following criteria should be used for judging the acceptability of the results at the 95 % confidence level:

10.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 20 % of their mean rating with the powder cleaner and 40 % with the liquid cleanser.

10.1.2 *Reproducibility*—Results obtained from the four round robins indicate that the manual method is not suitable for between laboratory evaluations.

10.1.3 A precision and bias statement for the mechanical method is being developed.

10.1.4 *Bias*—Bias cannot be determined for this test method.

11. Keywords

11.1 cleansability; soil/dirt resistance; stain resistance; washability; wet abrasion resistance

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Standard Test Method for Detection of Lead in Paint by Direct Aspiration Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 4834; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

Scope

1.1 This test method is intended as a screening test to determine if the solids in a paint contain more than 0.06 % lead. The test described can differentiate between 0.05 and 0.06 %. Paints giving a result greater than 0.05 % should be analyzed quantitatively for lead using Test Method D 3335. This test method provides a more definitive and reliable screening test than Test Method D 3618.

1.2 There is no reason to believe that higher levels of lead could not be determined by this test method provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²
D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings³
D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy³
D 3618 Test Method for Detection of Lead in Paint and Dried Paint Films³

Summary of Test Method

3.1 A weighed quantity of the paint sample is diluted (solvent-reducible paints in methyl isobutyl ketone and water-reducible paints in water) and then aspirated into the burner of an atomic absorption spectrometer. Lead content of the paint is determined from a calibration plot of absorbance versus concentration, prepared from standard coatings containing known amounts of lead.

Significance and Use

4.1 Current U.S. regulations restrict the amount of lead that may be present in consumer paint products to less than 0.06 % based on the total solids. This test method provides a

rapid means for screening paints to determine compliance with those regulations. The test method may be adjusted to meet regulations with a different limit on lead content.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of an atomizer and either a single- or a three-slot burner, gas pressure regulating and metering devices for air and acetylene, lead source lamp with a regulated constant-current supply, a monochromator and associated optics, a photosensitive detector connected to an electronic amplifier, and a readout device.

5.2 *Volumetric Flasks*, 50 mL.

6. Reagent and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

6.3 *Methyl Isobutyl Ketone (MIBK)*, technical grade.

6.4 *Standard Paints*—Prepare at least 3 standard paints by adding accurately weighed quantities of an analytically assayed compatible lead compound to a solvent-reducible or water-reducible paint that has been analyzed by Test Method D 3335 and shown to contain less than 10 ppm of lead. Determine the nonvolatile content of the base paint and choose quantities of the lead compound so that the lead concentrations of the standard paints will be approximately 300, 600, and 900 $\mu\text{g/g}$ based on the nonvolatile content.

7. Calibration and Standardization

7.1 Prepare at least three solvent-reducible or water-reducible standard paint specimens (see 6.4) that bracket the expected lead concentration in the sample to be tested. Following the procedure in 8.2 dilute the standards for aspiration.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.
Current edition approved Dec. 16, 1988. Published February 1989.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ Reagent Chemicals, American Chemical Society Specifications, Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

7.2 Operational instructions for atomic absorption spectrophotometers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

7.3 Turn the instrument on and set the wavelength to the 283.3-nm lead line. Apply the current recommended by the manufacturer to the lead source lamp. Allow the instrument to warm up for about 15 min and set the slit width. Adjust the air and acetylene pressure or flow rates and ignite the burner in accordance with the manufacturer's instructions. A background corrector should be used.

7.4 Aspirate each of the diluted standard paints and record the corresponding absorbance readings. Aspirate MIBK or water, as appropriate, between standards.

NOTE 1: Caution—It is important that MIBK or water be aspirated immediately after each diluted standard paint to minimize fouling of the aspirator/nebulizer system which could change the aspiration rate and absorbance reading. Continued response for lead while aspirating MIBK or water may indicate contamination of the burner head with pigment and require cleaning of the burner head.

NOTE 2—To be certain that the diluted paint specimen remains homogeneous during aspiration, the flask containing the specimen may be stirred continuously with a small magnetic stirring bar.

7.5 Construct a calibration curve on linear graph paper by plotting the absorbance readings versus micrograms of lead present in each diluted standard paint as follows:

$$A = (B \times D \times E)$$

where:

A = lead present, μg ,

B = lead in standard paint based on nonvolatile content (see 6.4), $\mu\text{g/g}$,

D = weight of standard paint, g, and

E = nonvolatile portion of standard paint, %.

8. Procedure

8.1 Mix the paint to be tested until homogeneous, preferably on a mechanical shaker. Determine the nonvolatile content in accordance with the appropriate test method selected by consulting Guide D 2832.

8.2 Prepare at least two replicate specimens by weighing by difference from a weighing bottle or disposable syringe to 0.1 mg, $0.5 \pm 0.02\text{-g}$ quantities of the paint being tested, into 10 mL volumetric flasks. Dilute to the mark with MIBK, if the paint is solvent reducible, or with water, if it is water reducible. Mix thoroughly.

NOTE 3—Adjustment of specimen size and dilution volume may be made as required for linear calibration and reasonable response for the instrument used as well as for differing lead content limits.

8.3 Aspirate the diluted paint specimens *immediately* (Caution—See Note 1) after mixing and measure the absorbance (see Note 2). Aspirate MIBK or water, as appropriate between specimen aspirations. Determine the micrograms of lead present from the calibration curve.

NOTE 4—For maximum accuracy, calibration and standardization (See Section 7) should be completed just prior to aspirating the diluted paint specimen.

9. Calculation

9.1 Calculate the mean concentration of lead in the nonvolatile portion of the paint specimen as follows:

$$L = (C \times 100)/(NV \times S)$$

where:

L = lead in nonvolatile portion of the paint specimen, ppm ($\mu\text{g/g}$),

C = lead in the aspirated specimen solution (8.3), μg ,
100 = factor derived from multiplying the numerator by 100 (to convert NV used to a whole number) and 10⁶ (to obtain ppm) and multiplying the denominator by 10⁶ (to convert grams of specimen to micrograms),

NV = nonvolatile portion of paint specimen, %, and

S = weight of specimen, g.

10. Report

10.1 Report the lead concentration expressed on the nonvolatile content of the paint sample.

11. Precision and Bias⁵

11.1 *Precision*—The precision estimates are based on an interlaboratory study in which five different laboratories analyzed in duplicate on 2 days, three samples of solvent reducible paints and three samples of water-reducible paints containing from 464 to 742 ppm lead. The within-laboratory coefficient of variation was found to be 3.34 % relative at 24 df and the between-laboratories coefficient of variation was 6.72 % relative at 18 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 9.76 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 19.96 % relative.

11.2 *Bias*—Bias is under investigation.

12. Index Terms

12.1 This test method is indexed under the following terms: spectroscopy—atomic adsorption; lead content—paint; lead screening test.

⁵ Supporting data is available from ASTM Headquarters. Request RR: D01-1060.

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Standard Test Method for Determining the Relative Tinting Strength of Chromatic Paints¹

This standard is issued under the fixed designation D 4838; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method describes the determination of the absorption tinting strength of a chromatic test paint relative to that of a standard or reference paint of the same chemical type. The procedures are based on dilution of the paints with a standard mixing white paint, followed by instrumental measurement and calculation. Provision is made for correcting the results for small differences in hue or chroma, or both, between the test and reference chromatic paints.

1.2 This test method is intended for the comparison of paints containing the same type of vehicle (acrylic, alkyd, or oil) and single-pigment colorants of the same Colour Index² name and number. The amounts of the pigment and of the other components of the paint need not be known.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 ASTM Standards:

- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature³
- D 4303 Test Methods for Lightfastness of Pigments Used in Artists' Paints³
- E 284 Definitions of Terms Relating to Appearance of Materials⁴
- E 308 Method for Computing the Colors of Objects by Using the CIE System⁴
- E 1164 Practice for Obtaining Spectrophotometric Data for Object-Color Evaluation⁴

Terminology

3.1 Definitions:

3.1.1 *tinting strength*—measure of the effectiveness with which a unit quantity of a colorant alters the color of a material. For scattering and absorbing colorants (pigments),

both absorption and scattering tinting strength must be specified.

3.1.2 *tinting strength, absorption*—relative change in the absorption properties of a standard white material when a specified amount of an absorbing pigment, black or chromatic, is added to it.

DISCUSSION—This is the common definition of tinting strength; however, this definition of the term can be misleading. For example, the tinting strength of a yellow colorant depends on its scattering as well as its absorption. Its tinting strength as determined from a mixture with white provides no information about its behavior when mixed with low-scattering colorants, such as a black.

3.1.3 *tinting strength, scattering*—relative change in the scattering properties of a standard black material (with no white pigment present) when a specified amount of a white or chromatic scattering pigment is added to it.

3.1.4 For other definitions, see Definitions E 284.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *drawdown*—a layer of paint deposited on a substrate by use of a drawdown bar to evaluate the characteristics of the paint.

3.2.2 *drawdown bar*—a bar designed to deposit a specified thickness of wet paint film uniformly on a specified test panel or other substrate.

4. Summary of Test Method

4.1 Chromatic paints are diluted with white paint to obtain mixtures that will produce a drawdown having 35 to 45 % reflectance factor at the wavelength of maximum absorption.

4.2 Drawdowns of these mixture paints are produced at complete hiding.

4.3 The drawdowns are measured to obtain tristimulus filter readings R , G , B either directly or by computation from CIE tristimulus values X , Y , Z .

4.4 One of the samples is designated the standard, and the percents of tinting strength, % TS, of the others are calculated relative to that of the standard. Provision is made for correcting this tinting strength for small differences in hue, chroma, or both, between the standard and the test specimen, and for obtaining an average tinting strength and a range.

5. Significance and Use

5.1 Tinting strength may be one factor in judging the relative economic value of paints, since pigment concentration contributes to strength in a major way; other factors are formulation and color development in grinding. The user may also select products for other properties, such as

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

Current edition approved June 24, 1988. Published November 1988.

² *Colour Index*, The Society of Dyers and Colourists, London, 1987. Available from the American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

transparency, that are accompanied by different tinting strengths. The results of this test method may be used for production control or quality comparisons.

5.2 The product with the greatest or the least tinting strength may not be the most desirable for a given artistic use. For example, low tinting strength may lead to the need to use an excessively high pigment concentration to obtain a desired color effect, and this may lead to defects in the dry paint film.

5.3 This test method applies only to single-pigment paints. The tinting strength of paints that contain two or more chromatic pigments with different optical properties cannot be evaluated by this test method.

5.4 The term "similar chemical type" used in 1.1 does not limit the ingredients in the paints to identity, but refers to compatibility in the case of vehicles and to similarity in the case of pigment types.

5.5 While the instrumental evaluation of tinting strength is described, visual comparisons can also be used, with lower precision, and should be made to provide confirmation of the instrumental and computational results.

5.6 If the sample and standard are widely different in appearance when prepared at the same ratio of chromatic to white paint, another sample should be prepared to bring the two closer in appearance, to obtain the most accurate results.

5.7 The quantities of chromatic and white paints mixed must be accurately known, on either a weight or a volume basis, but the concentration of pigment in the chromatic paint need not be known.

5.8 When the paints being compared have the same vehicle and pigment (same Colour Index name and number) the values of uncorrected tinting strength from 9.1 and corrected tinting strength from 9.2 should be nearly the same. If they are not, an average of the two tinting strengths is recommended as the best estimate of the true value, and a range provides a measure of the magnitude of the uncertainty, which is due to differences in hue or chroma, or both, between the paints.

5.9 Strictly speaking, the Kubelka-Munk-type analysis of this test method should not be applied to the tristimulus filter readings used, but only to spectral data. For the purposes of the relative comparisons of this test method, however, the errors introduced by the calculations used cancel to an adequate degree.

6. Apparatus and Materials

6.1 *Laboratory Balance*, top-loading, having a sensitivity of 0.01 g.

6.2 *Drawdown bars*, capable of producing smooth paint films with wet-film thicknesses between 0.003 and 0.010 in. (0.075 and 0.25 mm).⁵

6.3 *Opacity charts*, sealed-paper type with black and white areas.⁶

6.4 *Color-Measuring Instrument*, either a spectrophotometer providing 1931 CIE tristimulus values X , Y , Z for CIE

standard illuminant C , or a tristimulus colorimeter providing either such tristimulus values or colorimeter readings R , G , B .

6.5 *Mixing White Paint*, prepared as described in the Specimen Preparation, Mixing Whites for Dilution of Colors section of Test Method D 4303. Alternatively, a commercial titanium-dioxide white artists' paint may be used. The mixing white paint must be made with the same vehicle type (acrylic, alkyd, or oil) as the paints to be tested.

7. Specimen Preparation

7.1 Obtain representative samples of the chromatic paints to be tested. For tube paints, expel the entire contents of the tube and mix thoroughly before sampling.

7.2 Determine the approximate amount of chromatic paint to be added to 20 g of mixing white paint to obtain a drawdown with 35 to 45 % reflectance factor at the wavelength of maximum absorption. If the amount of chromatic paint is not known in advance, consult the tables in Appendix X1. For pigments other than those listed, use as the general guideline the addition of 5 g of chromatic paint containing an inorganic pigment or 1 g of chromatic paint containing an organic pigment to the 20 g of mixing white paint.

NOTE 1—Appendix X1 of Test Method D 4303 describes a method for computing the necessary adjustments in quantities required. Additional trials are needed to obtain the desired level of reflectance factor.

7.3 Weigh out the chromatic and mixing white paints to the nearest 0.01 g, and mix thoroughly.

7.4 Prepare drawdowns by placing the paint mixture at one end of an opacity chart and pulling the drawdown bar smoothly through the paint and across the chart. Paste paints should be spread with a spatula or palette knife over the entire chart area to be covered before pulling the bar down the chart.

7.5 Allow the drawdowns to reach the dry-to-touch time as described in the Procedure section of Test Method D 1640. Acrylic paints should dry in air overnight. Alkyd paints may require 5 days to dry. Oil paints may require 20 months to dry.

7.6 Determine whether each drawdown is at complete hiding by measuring the portions of it over the black and over the white areas of the chart to determine tristimulus value Y or colorimeter reading G . If the quotient Y_B/Y_W or G_B/G_W , where the subscripts refer to measurements over black and white, respectively, is greater than 0.98, the drawdown can be considered at complete hiding. If the drawdown is not at complete hiding, prepare a thicker drawdown or a drawdown made with multiple coats of paint, one over another.

NOTE 2—At the required dilution with white, a drawdown bar with an aperture of 0.006 in. (0.15 mm) will usually make a drawdown at complete hiding. In the cases of some acrylic paints and a few oil paints it may be necessary to increase the aperture to 0.010 in. (0.25 mm) to obtain complete hiding. If this leads to a slow-drying film or a film that is wrinkled when dry, multiple coats can be applied by depositing a second 0.006 in. (0.15 mm) coat, drawing down at a right angle to the first coat. After this coat dries, a third coat can be applied if necessary by using a shorter bar that rides over the previous coats.

⁵ Suitable drawdown bars can be obtained from the Paul N. Gardner Company, Inc., P.O. Box 10688, Pompano Beach, FL 33061-6688, or BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

⁶ Leneta 2A charts, available from the Leneta Co., Box 576, Ho-Ho-Kus, NJ 07423, and the Morest Co., 211 Center St., New York, NY 10013, have been found satisfactory for this purpose.

Procedure

8.1 Obtain values of R , G , B for each sample by either of the two following procedures.

8.1.1 Measure the drawdown with a spectrophotometer or tristimulus colorimeter to determine 1931 CIE tristimulus values X , Y , Z for CIE standard illuminant C . Follow Practice E 1164 and Method E 308. If hemispherical (integrating-sphere) geometry is used, measure with the specular component excluded.

8.1.2 If a colorimeter that is direct reading in R , G , B is used, measure these quantities.

8.2 If X , Y , Z are measured, calculate R , G , B by use of the following equations:

$$R = (X/0.98 - 0.2Z/1.18)/0.8 \quad (1)$$

$$G = Y \quad (2)$$

$$B = Z/1.18 \quad (3)$$

NOTE 3—The use of the 1931 CIE system (and standard observer) and standard illuminant C is specified because all known tristimulus colorimeters that are direct reading in R , G , B measure for these conditions. If values of X , Y , Z are obtained by spectrophotometry, the 1964 CIE system and other CIE standard illuminants may be used. Equations 1 to 3 are specific to the conditions, and must be replaced by the appropriate equations if other conditions are specified.⁷ The article referenced also discusses correcting the measured values for surface reflections.

Calculation

9.1 Calculate uncorrected relative tinting strength, % TS_{UC} as follows:

9.1.1 Using decimal-fraction values of R , G , B , calculate Kubelka-Munk-type ratios of absorption coefficient, K , to scattering coefficient, S :

$$(K/S)_R = (1 - R)^2/2R \quad (4)$$

$$(K/S)_G = (1 - G)^2/2G \quad (5)$$

$$(K/S)_B = (1 - B)^2/2B \quad (6)$$

9.1.2 Calculate the pigment concentration term C_P :

$$C_P = Q_c/(Q_c + Q_w) \quad (7)$$

where:

Q_c = quantity of chromatic paint, g,

Q_w = quantity of white paint, g.

NOTE 4—If it is desirable to use volume rather than weight as the basis for comparison of tinting strengths, determine the densities of the paints and compute the volumes of the weighed samples. Calculate C_P by use of Eq 7 using volumes instead of weights.

9.1.3 Calculate normalized values of (K/S) , denoted N , as follows:

$$N_R = (K/S)_R/C_P \quad (8)$$

$$N_G = (K/S)_G/C_P \quad (9)$$

$$N_B = (K/S)_B/C_P \quad (10)$$

9.1.4 Select one of the specimens to be denoted the standard and assigned the value of 100 % tinting strength. The tinting strength of the remaining specimens will be

determined relative to that of the standard.

9.1.5 Select the value of N to be used in the calculation of % TS_{UC} by one of the following three methods:

9.1.5.1 Select N based on the visually determined color of the specimen: For blue and green specimens, select N_R ; for purple and red specimens, select N_G ; and for yellow and orange specimens, select N_B . Relabel the selected value N_{1SPEC} and N_{STD} for the specimens and the standard, respectively. Relabel the remaining two values of N as N_{2SPEC} and N_{3SPEC} for the specimens and N_{2STD} and N_{3STD} for the standard.

9.1.5.2 If the specimen color cannot be classified accurately in 9.1.5.1, select the lowest value of N as N_1 . The same selection must be made for the standard and all specimens to be compared. Relabel the values of N as in 9.1.5.1.

9.1.5.3 If the values of N_R and N_B for the specimen are both low and approximately equal, follow the procedure in Annex A1 to select N and calculate the tinting strength.

9.1.6 Calculate % TS_{UC} as follows:

$$\% TS_{UC} = 100 (N_{1SPEC}/N_{1STD}) \quad (11)$$

9.2 Calculate tinting strength corrected for differences in hue and chroma, % TS_C , by use of the following equations:

$$d_{SPEC} = N_{2SPEC} + N_{3SPEC} \quad (12)$$

$$d_{STD} = N_{2STD} + N_{3STD} \quad (13)$$

$$D = (d_{SPEC} - d_{STD})/2 \quad (14)$$

$$\% TS_C = 100 (N_{1SPEC} - D)/N_{1STD} \quad (15)$$

9.3 Calculate average tinting strength, % TS_{AV} , and range E as follows:

$$\% TS_{AV} = (\% TS_{UC} + \% TS_C)/2 \quad (16)$$

$$E = \pm (\% TS_{UC} - \% TS_C)/2 \quad (17)$$

10. Report

10.1 Report the following information:

10.1.1 Complete identification of the specimens, including brand and color name, date of manufacture, and lot number if available.

10.1.2 Name of color-measuring instrument used, method of standardization, and other information required in the Report section of Practice E 1164 and Methods E 308.

10.1.3 Date of test.

10.1.4 Test results for % TS_{UC} , % TS_C , or % TS_{AV} , and range.

11. Precision and Bias⁸

11.1 Based on interlaboratory intercomparisons, the results of this test method agree to within ± 6 % on an absolute basis.

12. Index Terms

12.1 This test method is indexed under the following terms: artists' paints; chromate coatings; tinting strength.

⁷ Johnston-Feller, R. M., and Bailie, C. W., "Determination of the Tinting Strength of Chromatic Pigments," *Journal of Coatings Technology*, Vol 54, No. 892, 1982, pp. 43-56.

⁸ Supporting data are available from ASTM Headquarters. Request RR:D01-1057.

ANNEX

(Mandatory Information)

A1. Procedure for Pigments with Two Separated Absorption Maxima

A1. The tinting strength of pigments, such as chromium oxide green, for which both N_R and N_B are low and approximately equal, must be calculated by the following equations:

$$\text{NUM} = (N_{R,\text{SPEC}} + N_{B,\text{SPEC}})/2 \quad (\text{A1.1})$$

$$\text{DENOM} = (N_{R,\text{STD}} + N_{B,\text{STD}})/2 \quad (\text{A1.2})$$

$$D = (N_{G,\text{SPEC}} + N_{G,\text{STD}})/2 \quad (\text{A1.3})$$

$$\% \text{TS}_{\text{UC}} = 100 \text{ NUM/DENOM} \quad (\text{A1.4})$$

$$\% \text{TS}_{\text{C}} = (\text{NUM} - D)/\text{DENOM} \quad (\text{A1.5})$$

APPENDIX

(Nonmandatory Information)

TABLE X1.1 Approximate Weight of Acrylic Paint to Mix With 20-g of Mixing White Paint

Pigment Name	Colour Index Name	Chromatic Paint, g
Alizarin crimson	PR 83	2.5
Azo yellow medium	PY 74	2.5
Burnt umber	PBr 7	3.0
Cadmium-barium orange	PO 20:1	2.5
Cadmium-barium red medium	PR 108:1	4.0
Cadmium-barium yellow light	PY 35:1	4.0
Cadmium-barium yellow medium	PY 37:1	3.3
Cerulean blue Co-Cr	PB 36	9.0
Cerulean blue Co-Sn	PB 35	10.0
Chromium oxide green	PG 17	4.0
Cobalt blue	PB 28	5.0
Dioxazine purple	PV 23 RS	1.0
Hansa yellow light	PY 3	3.0
Naphthol AS-OL red	PR 9	2.5
Naphthol red light AS-D	PR 14	2.0
Phthalocyanine blue	PB 15	0.4
Phthalocyanine green	PG 7, PG 36	0.5
Raw sienna	PBr 7	4.0
Raw umber	PBr 7	6.0
Red oxide	PR 101	1.0
Ultramarine blue	PB 29	4.0
Yellow oxide	PY 42	4.0

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Total Carbon and Organic Carbon in Water by Ultraviolet, or Persulfate Oxidation, or Both, and Infrared Detection¹

This standard is issued under the fixed designation D 4839; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method covers the determination of total carbon (TC), inorganic carbon (IC), and total organic carbon (TOC) in water, wastewater, and seawater in the range from 1 mg/L to 4000 mg/L of carbon.

1.2 This test method was used successfully with reagent water spiked with sodium carbonate, acetic acid, and pyridine. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 This test method is applicable only to carbonaceous matter in the sample that can be introduced into the reaction zone. The syringe needle or injector opening size generally limit the maximum size of particles that can be so introduced.

1.4 In addition to laboratory analyses, this test method may be applied to stream monitoring.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water²

D 1192 Specification for Equipment for Sampling Water and Steam²

D 1193 Specification for Reagent Water²

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water²

D 3370 Practices for Sampling Water²

D 4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data²

Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Specification D 1129.

3.2 Description of Terms Specific to This Standard:

3.2.1 *inorganic carbon (IC)*—carbon in the form of carbon dioxide, carbonate ion, or bicarbonate ion.

3.2.2 *total organic carbon (TOC)*—carbon in the form of organic compounds.

3.2.3 *total carbon (TC)*—the sum of IC and OC.

3.2.4 *refractory material*—that which cannot be oxidized completely under the test method conditions.

4. Summary of Test Method

4.1 *Fundamentals*—Carbon can occur in water as an inorganic and organic compound. This test method can be used to make independent measurements of IC, TOC, and TC, and can also determine IC by the difference of TC and TOC, and TOC as the difference of TC and IC.

4.2 The essentials of this test method are: (a) removal of IC, if desired, by acidification of the sample and sparging by carbon-free gas; (b) conversion of remaining carbon to CO₂ by action of persulfate, aided either by elevated temperature or ultraviolet (UV) radiation; (c) detection of CO₂ that is swept out of the reactor by a gas stream; and (d) conversion of detector signal to a display of carbon concentration in mg/L. A diagram of suitable apparatus is given in Fig. 1.

5. Significance and Use

5.1 This test method is used for determination of the carbon content of water from a variety of natural, domestic, and industrial sources. In its most common form, this test method is used to measure organic carbon as a means of monitoring organic pollutants in industrial wastewater. These measurements are also used in monitoring waste treatment processes.

5.2 The relationship of TOC to other water quality parameters such as chemical oxygen demand (COD) and total oxygen demand (TOD) is described in the literature.³

6. Interferences and Limitations

6.1 The oxidation of dissolved carbon to CO₂ is brought about at relatively low temperatures by the chemical action of reactive species produced by hot or UV-irradiated persulfate ions. Even if oxygen is used as the sparging gas, it makes a much lower contribution to oxidation than in high-temperature (combustive) systems. Not all suspended or refractory material may be oxidized under these conditions; analysts should take steps to determine what recovery is being obtained. This may be done by several methods: (a) by monitoring reaction progress to verify that oxidation has been completed; (b) by rerunning the sample under more vigorous reaction conditions; (c) by analyzing the sample by

¹ This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

Current edition approved June 24, 1988. Published September 1988.

² Annual Book of ASTM Standards, Vol 11.01.

³ Handbook for Monitoring Industrial Wastewater, Section 5.3, U.S. Environmental Protection Agency, August 1973, pp. 5-12.

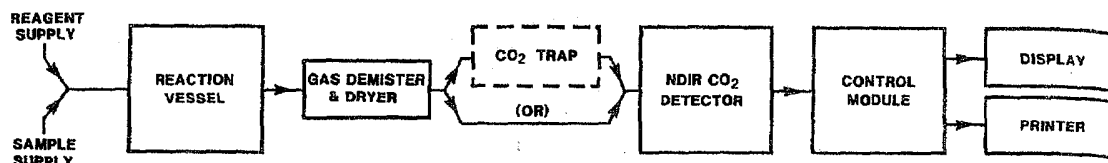


FIG. 1 Diagram of Apparatus

an alternative method known to result in full recovery; or (d) by spiking samples with known refractories and determining recovery.

6.2 Chloride ion tends to interfere with oxidative reaction mechanisms in this test method, prolonging oxidation times and sometimes preventing full recovery. Follow manufacturer's instructions for dealing with this problem. See Appendix XI for supporting data.

6.3 Homogenizing or sparging of a sample, or both, may cause loss of purgeable organic compounds, thus yielding a value lower than the true TOC level. (For this reason, such measurements are sometimes known as nonpurgeable organic carbon (NPOC)). The extent and significance of such losses must be evaluated on an individual basis. This may be done by comparing the TOC by difference (TC-IC) with the direct TOC figure, that is, that obtained from a sparged sample. The difference, if any, between these TOC figures represents purgeable organic carbon (POC) lost during sparging. Alternatively, direct measurement of POC can be made during sparging, using optional capabilities of the analyzer.

6.4 Note that error will be introduced when the method of difference is used to derive a relatively small level from two large levels. For example, a ground water high in IC and low in TOC will give a poorer TOC value as (TC-IC) than by direct measurement.

7. Apparatus

7.1 *Homogenizing Apparatus*—A household blender is generally satisfactory for homogenizing immiscible phases in water.

7.2 *Sampling Devices*—Microlitre-to-millilitre syringes are typically required for this test method. Alternatives include manually operated or automatically operated sampling valves. Sampling devices with inside diameters as small as 0.15 mm may be used with samples containing little or no particulate matter. Larger inside dimensions such as 0.4 mm will be required for samples with particulate matter.

NOTE 1—See 6.1 concerning oxidation of particulate matter.

7.3 *Apparatus for Carbon Determination*—This instrument consists of reagent and sample introduction mechanism, a gas-sparged reaction vessel, a gas demister or dryer, or both, an optional CO₂ trap, a CO₂-specific infrared detector, a control system, and a display. Fig. 1 shows a diagram of such an arrangement.

7.3.1 Sparging requires an inert vessel with a capacity of at least double the sample size with provision for sparging with 50 to 100 mL/min of carbon free gas. This procedure will remove essentially all IC in 2 to 10 min, depending on design.

7.3.2 *Oxidation*—The reaction assembly contains reagent and sample introduction devices, and a reactor vessel with sparging flow of carbon-free gas. The vessel may be heated by an external source, and may contain a UV lamp. The reaction vessel and sparging vessel (see 6.3.1) may be combined.

7.3.3 *Gas Conditioning*—The gas passing from the reactor is dried, and the CO₂ produced is either trapped and later released to the detector, or routed directly to the detector through a chlorine-removing scrubber.

7.3.4 *Detector*—The CO₂ in the gas stream is detected by a CO₂-specific nondispersive infrared (NDIR) detector.

7.3.5 *Presentation of Results*—The NDIR detector output is related to stored calibration data and then displayed as milligrams of carbon per litre.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,⁴ where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type I or Type II. The indicated specification does not actually specify inorganic carbon or organic carbon levels. These levels can affect the results of this test method, especially at progressively lower levels of the carbon content in the samples to be measured. Where inorganic carbon in reagent water is significant, CO₂-free water may be prepared from reagent water by acidifying to pH 2, then sparging with fritted-glass sparger using CO₂-free gas (time will depend on volume and gas flow rate, and should be determined by test). Alternatively, if the carbon contribution of the reagent water is known accurately, its effect may be allowed for in preparation of standards and other solutions. CO₂-free water should be protected from atmospheric contamination. Glass containers are required for storage of water and standard solutions.

8.3 *Acid*—Various concentrated acids may be used for acidification of samples and of the oxidizing reagent. Acids such as phosphoric (sp gr 1.69), nitric (sp gr 1.42), or sulfuric (sp gr 1.84) are suitable for most applications. Sulfuric acid

⁴ "Reagent Chemicals, American Chemical Society Specifications," American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the A.C.S., see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY and the "United States Pharmacopeia."

should be used in the form of a 1 + 1 dilution, for safety reasons. Hydrochloric acid is not recommended.

8.4 Organic Carbon, Standard Solution (2000 mg/L)—Choose a water-soluble, stable reagent grade compound, such as benzoic acid or anhydrous potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$). Calculate the weight of compound required to make 1 L of organic carbon standard solution; for example, $\text{KHC}_8\text{H}_4\text{O}_4 = 0.471$ g of carbon per gram, so one litre of 2 g/L of standard requires 2/0.471, or 4.25, grams of KHP. Dissolve the required amount of standard in some CO_2 -free water in a 1-L volumetric flask, add 1 mL of acid, and dilute to volume. This stock solution, or dilutions of it, may be used to calibrate and test performance of the carbon analyzer.

8.5 Persulfate Solution—Prepare by dissolving the appropriate weight of potassium or sodium persulfate in 1 L of water, to produce the concentration specified by the instrument manufacturer. If specified, add 1 mL of phosphoric acid (sp gr 1.69) and mix well. Store in a cool, dark place. Recipes for this reagent solution may be modified by manufacturers to meet the needs of specific applications, for example, high chloride samples.

8.6 Gas Supply—A gas free of CO_2 and of organic matter is required. Use a purity as specified by the equipment manufacturer. The use of oxygen is preferred for the UV-persulfate method, and nitrogen or helium is preferred if a CO_2 trap is used between reactor and detector.

9. Sampling and Sample Preservation

9.1 Collect the sample in accordance with Specification D 1192 and Practice D 3370.

9.2 To preserve samples for this analysis, store samples in glass at 4°C. To aid preservation, acidify the samples to a pH of 2. It should be noted that acidification will enhance loss of inorganic carbon. If the purgeable organic fraction is important, fill the sample bottles to overflowing with a minimum of turbulence and cap them using a fluoropolymer-lined cap, without headspace.

9.3 For monitoring of waters containing solids or immiscible liquids that are to be injected into the reaction zone, use a mechanical homogenizer or ultrasonic disintegrator. Filtering or screening may be necessary after homogenization to reject particle sizes that are too large for injection. Volatile organics may be lost. See 6.3.

9.4 For wastewater streams where carbon concentrations are greater than the desired range of instrument operation, dilute the samples as necessary.

10. Instrument Operation

10.1 Follow the manufacturer's instructions for instrument warmup, gas flows, and liquid flows.

11. Calibration

11.1 Use the stock solution of 2000 mg/L of carbon, and various dilutions of it, for calibration.

NOTE 2—Dilutions should be made with CO_2 -free water (see 8.2).

11.2 Calibration protocols may vary with equipment manufacturers. However, in general, calibrate the instrument in accordance with the manufacturer's instructions, and use standards to verify such calibration in the specific range of

interest for actual measurements. Plots of standard concentration versus instrument reading may be used for calibration or to verify linearity of response.

11.3 Establish instrument blank according to the manufacturer's instructions.

12. Procedure

12.1 Mix or blend each sample thoroughly and carry out any necessary dilution to bring the carbon content within range of the instrument.

12.2 If inorganic carbon is to be measured directly, inject the sample into the analyzer under appropriate conditions.

12.3 If inorganic carbon is to be removed by sparging prior to sample introduction, acidify to approximately pH 2 with concentrated acid (if not already done) and sparge with an appropriate flow of gas. Samples with high alkali content or buffer capacity may require larger amounts of acid. In such cases, incorporate this dilution into the calculation. If incomplete sparging of CO_2 from IC is suspected, sparge and analyze the sample and then repeat the procedure until appropriate conditions are established. In difficult conditions, use of a fritted-glass sparger may help.

12.4 To measure TOC, inject an appropriate volume of the sample into the analyzer. If external sparging is required to remove IC, inject a sparged sample for the TOC measurement. See 6.3.

12.5 To measure TC, inject an appropriate volume of unsparged sample.

13. Calculation

13.1 Read carbon values directly from a digital display or printer, or both.

14. Precision and Bias⁵

14.1 Collaborative Test—This test method was evaluated by sending seven identical sample to each of ten laboratories and asking them to measure TOC and TC exactly in accordance with this test method. Three of the ten laboratories did not make the TC measurement. One of the samples consisted of laboratory reagent water. The other six were of that water spiked to various levels with acetic acid, pyridine, and sodium carbonate. TC levels ranged from 0.6 to 2 000 mg/L, and TOC levels from 0.3 to 1 700 mg/L. An *F* test at 95 % confidence level showed no significant difference between the results of the five laboratories using UV-persulfate oxidation and those of the five laboratories using hot persulfate. Consequently, results were pooled for further analysis.

14.2 Removal of Outliers—Application of outlier tests specified in Practice D 2777 – 85 resulted in the elimination of one laboratory's TC and TOC results. In addition, three laboratories did not perform the TC analysis, so the effective number of laboratories was six for the TC measurement. Five of their individual results were later eliminated by outlier test. In the TOC determination, one additional laboratory failed the outlier test, leaving a total of eight. Three individual results were later eliminated.

⁵ Supporting data are available from ASTM Headquarters. Request RR: D-19-1130.

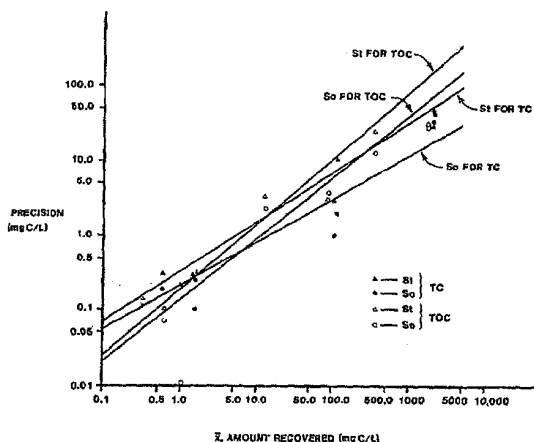


FIG. 2 Precision Versus Amount Recovered

14.3 *Precision*—Separate determinations of precision were made for TC and TOC measurements:

$$\text{For TC: } S_r = 0.03x + 0.3$$

$$S_o = 0.01x + 0.2$$

$$\text{For TOC: } S_r = 0.08x + 0.1$$

$$S_o = 0.04x + 0.1$$

where:

x = the recovered C concentration, mg/L,

S_r = overall precision, and

S_o = single-operator precision.

Fig. 2 shows a log-log plot of the overall and single-operator precision of all TC and TOC measurements not eliminated by outlier tests.

14.4 *Bias*—Fig. 3 plots “amount added” against “amount found,” with overall precision shown as an error bar. Bias significant at the 95 % level (student’s t -test) is flagged. Water that was used as one of the samples is omitted, since no value equivalent to “amount added” is available. The contribution of the carbon in the water to the spiked samples was allowed for before analysis of bias. In general, bias is positive, with the values running from 1 % to 25 % of the amount added, with no particular pattern evident. Of the twelve bias measurements, ten were below 10 %. Users of this test method should make their own determination of bias.

14.5 *Matrix Effects*—Participants were asked to measure the TC and TOC levels in a water sample of their choice, and

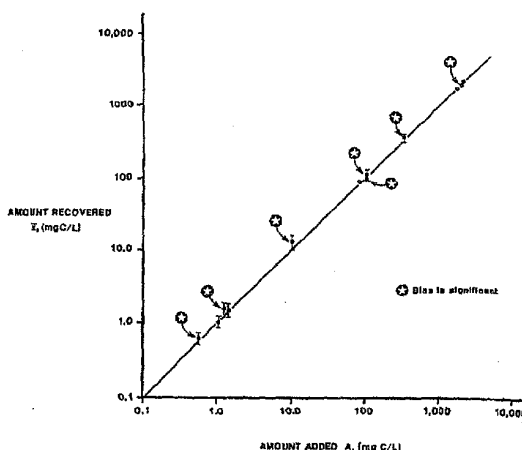


FIG. 3 Bias: Amount Added Versus Amount Recovered

then to spike the sample with one of the study samples and to measure the sample again. The chosen samples were: sink waste; DI water with KHP; soil solution; tap water with added IC; plant waste stream; synthetic sewage, and anion resin brine wash. TC recoveries averaged 86 % (range from 74 % to 92 %), and TOC, 82 % (from 47 % to 92 %). The negative bias, versus the positive bias noted in 14.4, can reflect incomplete oxidation of spiking compounds in the presence of other organics, errors introduced by sample handling, or other effects. In any event, no one matrix was studied in sufficient depth to provide an answer. Users of this test method should conduct their own experiments to determine recovery in their particular circumstances.

14.6 The quality assurance (QA)/quality control (QC) portion of this test method has not been completely established at this time. It is the intent of Subcommittee D19.06 that procedures be incorporated into this test method that require a minimum level of QC. These procedures will require, at minimum, a method startup check and ongoing performance checks. The analysts performing this test method will be required to measure their performance against the performance level achieved by the laboratories that participated in the ASTM round-robin study done on this test method. These formal QC procedures will be incorporated at such time as they have been officially accepted by the Society. See Practice D 4210.

APPENDIX

(Nonmandatory Information)

X1. RECOVERIES OF VARIOUS COMPOUNDS FROM CHLORIDE-CONTAINING SOLUTIONS WITH UV-PERSULFATE OXIDATION

TABLE X1.1 Percent Recovery

Analyte	No Mercuric Reagent	With Mercuric Reagent
Potassium hydrogen phthalate	90.5	101.2
Urea	101.1	...
Methanol	96.6	99.3
Nicotinic acid	91.0	97.9
Pyridine	88.6	98.0
Proline	86.0	92.2
n-Butanol	74.3	96.2
Acetic acid	66.5	101.3
Laudine	64.7	96.6
Acetonitrile	5.0	88.0

TABLE X1.2 Recoveries of Potassium Hydrogen Phthalate from Chloride-Containing Solutions Using Hot Persulfate Oxidation

ppm of Carbon	Recovery
200	99.4 %
800	92.0 %

X1.1 *Conditions*—Inject into the instrument 200 μ L of solution, containing 100 ppm of carbon in the form of the compound indicated plus 1.8 % of chloride ion. Take results at the completion of oxidation or after 8 min, whichever occurs first.

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Standard Test Method for Erosion Testing of Antifouling Paints Using High Velocity Water¹

This standard is issued under the fixed designation D 4938; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of erosion rates for marine antifouling paint systems immersed in flowing natural seawater.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

A 569/A 569M Specification for Steel, Sheet and Strip, Carbon (0.15 Maximum Percent), Hot-Rolled, Commercial Quality²

D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

D 1889 Test Methods for Turbidity of Water⁴

D 2200 Pictorial Surface Preparation Standards for Painting Steel Surfaces³

2.2 U.S. Military Specifications:⁵

MIL-P-24441 Paint, Epoxy-Polyamide

DOD-P-24647 Paint, Antifouling, Ship Hull (Metric)

DOD-P-24655 Paint, Underwater Hull, Anticorrosion (Metric)

3. Summary of Test Method

3.1 Steel panels coated with the antifouling paint system under evaluation are positioned in a high velocity water channel, similar to the type shown in Figs. 1 to 3, parallel to the path of the flowing water.

3.2 Exposure conditions shall include at least one series of test panels evaluated at the standard water velocity of 12 m/s and shall specify the length of time, temperature, salinity, and pH. Additional velocities may be conducted at the discretion of the customer.

3.3 Color photographs and coating thickness measure-

ments shall be taken prior to exposure, at specified time intervals, and repeated at the end of the test for comparison purposes.

4. Significance and Use

4.1 This test method is intended to measure the erosion rates of ablative antifouling paint systems exposed to flowing water at velocities designed to subject the paint system to shear stresses experienced in service.

4.2 Measurement of erosion rates are necessary to help in the assessment of ablative antifouling paint film thicknesses required for fouling control between scheduled drydockings of ships, in the selection of materials, in producing quality assurance, and in understanding the performance mechanism.

4.3 The test data is intended to serve as a guide for predicting the service life of ablative antifouling paints in order to calculate the necessary paint thickness to fit specified deployment cycles. Erosion rates of antifouling paints in service will vary depending on such factors as: berthing location, geographic area of operation, salinity, pH, and temperature of seawater. It should also be recognized that some areas of the ship are subject to different erosion rates.

4.4 The degree of correlation between results obtained from this test method and shipboard service has not been determined.

5. Apparatus

5.1 Water Channel:

5.1.1 High velocity flowing water in a contained channel, similar to the type shown in Figs. 1 to 3, is used to induce hydrodynamic shear stresses on painted panels to determine erosion rates of ablative antifouling paints.

5.1.2 The basic apparatus consists of a four-walled channel, rectangular in cross section, through which natural seawater flows at varying linear velocities to simulate ships' speeds.

5.1.3 All wetted materials supplying seawater to and within the channel shall be nonmetallic with the following exceptions:

5.1.3.1 Channel circulating pump impellers.

5.1.3.2 Thermowells.

5.1.3.3 Channel flow orifice plate.

5.1.4 One section of the channel shall permit testing of the panels at a standard test velocity of 12 m/s. All sections of the channel shall provide flow with fully formed turbulent characteristics. A minimum Reynolds number of 1 000 000 shall be achieved in each velocity test section. The Reynolds number, R , is calculated as follows:

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.45 on Marine Coatings.

Current edition approved April 28, 1989. Published June 1989.

² Annual Book of ASTM Standards, Vol 01.03.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 11.01.

⁵ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

$$R = (D \times V \times P)/U$$

where:

- D = equivalent diameter = $4 \times (\text{area of flowing liquid/wetted perimeter})$, m,
- V = velocity, in m/s,
- P = density of medium, kg/m^3 , and
- U = viscosity, P/s.

For a channel with a rectangular cross section and a test panel placed in the middle of the channel, the equivalent diameter would be calculated as follows:

$$4 \times [(A \times B)/(2A + 2B)]$$

where:

- A = width of channel from side wall to panel face, m, and
- B = height of channel, m.

5.2 *Erosion Rate Determination*—Erosion rates of ablative antifouling paints are determined by using noneroding reference points and measured in accordance with Section 8 with the following equipment:

- 5.2.1 Microtome or encapsulating/polishing equipment.
- 5.2.2 Microscope with photomicrographic capability.

5.3 *Seawater Requirements:*

5.3.1 Seawater will be circulated through the channel at a constant rate permitting testing at different flow velocities as shown in 9.3.

5.3.2 Natural seawater shall be continuously supplied to the channel during operation to eliminate stagnation or concentration effects. During operation of the channel the following data shall be obtained and recorded on a daily basis:

- 5.3.2.1 Seawater circulating rate.
- 5.3.2.2 Seawater salinity and pH.
- 5.3.2.3 Incoming seawater temperature and the channel seawater temperature.

5.3.3 Provisions shall also be made for supplying filtered seawater to the channel. A suitable filter is one which can reduce turbidity to $\frac{1}{2}$ Jackson Turbidity Unit in accordance with Test Method D 1889.

5.3.4 As a minimum, the seawater chemistry in the channel, averaged over the course of the test run, shall be within the limits specified below:

	Maximum	Minimum
Salinity, ppm	35 000	27 000
pH	8.3	7.6

5.4 *Test Panels:*

5.4.1 Steel test panels conforming to Specification A 569/569M shall be used in the channel. The panels shall measure 18.75 cm high, 15 cm long, and 1.25 cm thick. The painted test panels shall be subjected to a double sided exposure with both sides exposed to similar hydrodynamic conditions.

5.4.2 Test panels painted in accordance with Test Methods D 823 are positioned vertically and parallel to the flowing water to simulate shear stresses experienced by paints on the ship's underwater hull.

5.4.3 The coating systems shall be applied in accordance with the manufacturer's instructions for both the anti-corrosion and the antifouling paints.

5.5 *Operation*—The channel shall be operated on a continuous basis during the test period except for downtime for panel inspection and seawater filtering system backwashes.

The total running time of the test is defined as the total hours of actual channel operation under fully flowing conditions.

6. Application of Paints

6.1 Antifouling paints under evaluation may be applied by spray over a primer from the same manufacturer in accordance with the manufacturer's directions or over 9 mils of epoxy polyamide paint conforming to Military Specification MIL-P-24441 with an appropriate tie-coat if necessary. Application shall be in accordance with Test Methods D 823.

7. Hazards

7.1 Antifouling paints contain toxic materials that could cause skin and eye irritation on contact and adverse physiological effects if ingested or inhaled. In the preparation of panels and the application of various types of antifouling paints, the use of appropriate protective clothing and equipment is required consistent with local, state, and federal government regulations and recognized industrial and technical standards. Spills, overspray, and unused material shall not be flushed down the drain but shall be disposed of as hazardous waste.

8. Procedure

8.1 Abrasive blast a minimum of three test panels for each system being evaluated to near-white metal, Sa 2½ in accordance with Pictorial Standard D 2200, to obtain a 1.0 to 3.0 mils (25 to 75 μm) surface profile.

8.2 Apply an epoxy anticorrosion primer in accordance with Military Specifications DOD-P-24655 and MIL-P-24441, or the manufacturer's recommendations, whichever applies.

8.3 Apply an antifouling topcoat in accordance with Military Specification DOD-P-24647 or the manufacturer's recommendations, whichever applies.

8.4 Apply additional coats of antifouling paint in accordance with Military Specification DOD-P-24647 or the manufacturer's instructions, whichever applies. The last coat shall dry for a minimum of 7 days before any erosion testing.

8.5 Both sides of the painted test surfaces are provided with noneroding reference (NER) points before immersion in the test environment. The NER is an insoluble, tightly adherent vinyl or other suitable, compatible coating 2 ± 0.5 cm/diameter applied in the center of the panel which will blanket a portion of the eroding surface. The NER preserves the original outer surface of the antifouling and thus offers a reference for comparison with the eroded surface during later microscopic examination.

8.6 Take film thickness measurements before and after testing (see 5.2).

8.7 Panels are to be photographed prior to starting the test for comparison with photographs taken at the conclusion of the test.

8.8 A specimen of exposed antifouling paint is carefully removed for examination in a single flake which includes a fragment of the noneroding reference. This specimen is mounted for microscopic analysis in a suitable medium such as paraffin wax or epoxy resin. Care is required to ensure that the specimen is not damaged by solvent attack or heat evolution during this encapsulation.

8.9 Specimens are prepared for examination by micro-

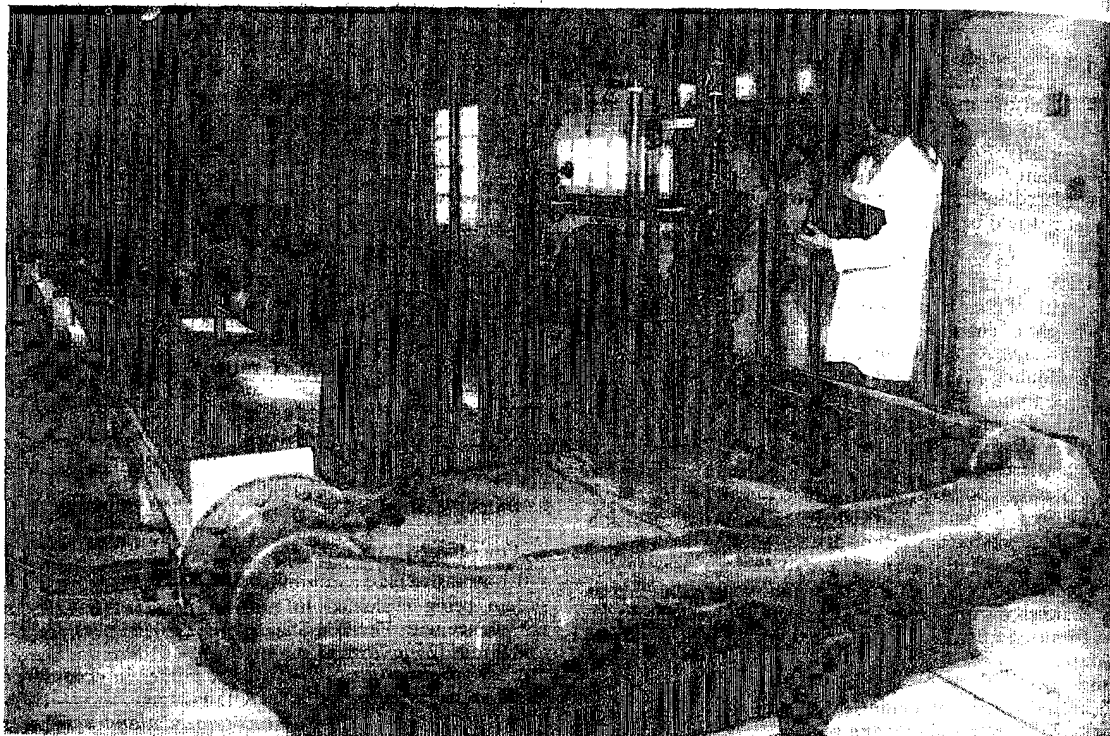


FIG. 1 High Velocity Flow Channel

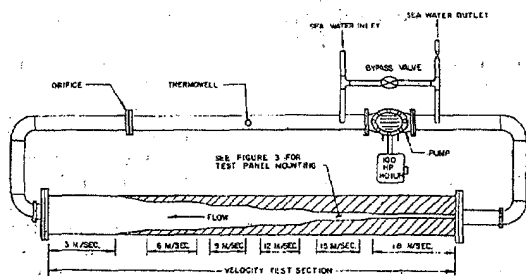


FIG. 2 Simplified Schematic of Flow Channel

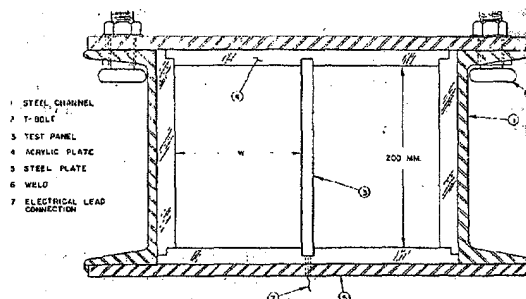
tome sectioning or by abrasive polishing to a plane surface.

8.10 Subsequent microscopic examination yields the typical image shown in Figs. 4 and 5. The material lost during the duration of the test is clearly shown as measurement d in Fig. 4. An actual photomicrograph is shown in Fig. 5.

9. Calculation

9.1 The material loss is expressed as the erosion rate of the ablative antifouling paint.

9.2 The erosion rate is calculated by dividing the micrometers of surface erosion by the duration of the test expressed in months at a specified speed.



W, mm	Velocity, m/s
50	18
60	15
75	12
100	9
150	6
300	3

FIG. 3 Cross-Section View of Test Panel Mounted in Flow Channel (Low Velocity Section)

9.2.1 Example—A 20- μ m erosion in 3 months equals 6.7 μ m/month.

9.3 The speed of the water in the channel expressed in metres per second is correlated to a ship's speed expressed in

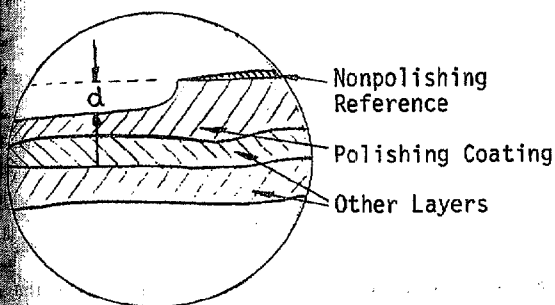


FIG. 4 Illustration of Material Lost During Testing

Tests in accordance with the following table:

Speed in Channel, m/s	Speed, knots
3	5.8
6	11.7
9	17.5
12	23.3
15	29.1
18	35.0

9.3.1 Example—A typical erosion rate is reported as 6.7 mm/month at 17.5 knots, etc.

10. Report

10.1 The final test report shall include the following data:

10.1.1 List of the paints tested,

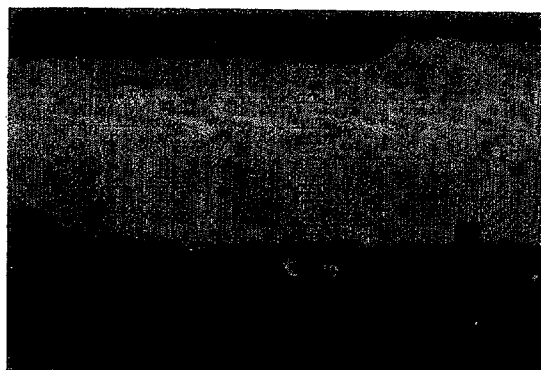


FIG. 5 Actual Microphotograph

- 10.1.2 Test duration—date started and date ended,
- 10.1.3 Thickness readings before and after exposure. Report total paint film loss at the standard test velocity and any auxiliary test velocities,
- 10.1.4 Daily measurements of the seawater temperature, salinity, and the pH,
- 10.1.5 Speed of test water correlated to knots,
- 10.1.6 Observation of the overall condition of painted panel, and
- 10.1.7 Initial and final photographs of the test panels.

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Standard Test Method for Conductimetric Analysis of Water Soluble Ionic Contamination of Blasting Abrasives¹

This standard is issued under the fixed designation D 4940; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for rapid evaluation of abrasives for the presence of ionic contamination by determining the total concentration of water soluble ionic contaminants by means of a conductivity test.

1.2 This test method does not identify the ionic species present nor provide quantitative results on each species.

1.3 This test method is based on a volume comparison among abrasives of similar sizes. A volume comparison is more closely related to surface area of the abrasives than is a weight comparison.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²
E 832 Specification for Laboratory Filter Papers³

3. Summary of Test Method

3.1 Abrasive and pure water are combined into a slurry that is stirred to leach the soluble salts from the abrasive. This slurry is filtered and conductance of the filtrate is measured. The conductivity, which is related to the concentration of soluble ionic materials contaminating the abrasive surface, is calculated from the conductance and the cell constant.

4. Significance and Use

4.1 By-product abrasives manufactured from slags that are air cooled or quenched with pure water, normally contain low concentrations of ionic materials as do mined mineral abrasives. However, slags quenched with seawater or other contaminated water, contain high amounts of ionic material as does seashore sand. This contamination of the abrasive can transfer to the steel surfaces being blasted, where it may accelerate corrosion. This test is useful in establishing the cleanliness of the abrasive at the jobsite.

4.2 This test method provides a value that indicates the concentration of total water soluble ions in accordance with their electrolytic mobility. Thus, it provides an indication of ionic corrosion potential.

NOTE 1—A typical value of conductivity for a high level of contamination is 500 $\mu\text{mho/cm}$. A typical value for a low level of contamination is 50 $\mu\text{mho/cm}$.

5. Apparatus

5.1 *Conductivity Bridge and Cell*—Any commercial conductivity bridge and conductivity cell having a range of at least 5 $\mu\text{mho/cm}$ to 1 000 000 $\mu\text{mho/cm}$ and temperature compensation capability is satisfactory. Either a dip-type, pipet-type, or cup-type cell may be used. A means of adjusting for temperature or controlling the temperature is essential. While some instruments have an adjustment to compensate for temperature, one means is to use a 25°C constant temperature bath. Another method is to stir the solution with a clean thermometer while the vessel is warmed or cooled by an external source.

NOTE 2—If temperature compensation or control is not followed correctly, an error of approximately 2 % per degree of temperature deviation from 25°C can be introduced. The deviation of conductivity with temperature in the range of 18°C is reported to be 0.0216 % per degree for chloride ions and 0.0227 % per degree for sulfate ions.

5.2 *Filter Paper*, conforming to Specification E 832, Type 1, Class C, to keep silt from fouling the surfaces of the conductivity cell.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.3 *Potassium Chloride* (KCl or 0.02 N KCl solution).

¹ This specification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

Current edition approved April 28, 1989. Published June 1989.

² *Annual Book of ASTM Standards*, Vol 06.03 and 11.01

³ *Annual Book of ASTM Standards*, Vol 14.02

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

7. Sampling

7.1 Sampling shall be as follows unless otherwise agreed upon between the purchaser and the seller. Take two 1-L samples of abrasive at random from different packages of each lot, batch, day's pack, or other unit of production in the shipment. When no markings distinguishing between units of production appear, take samples from the different packages in the ratio of two samples for each 10 000 lb (5000 kg), except that for shipments of less than 10 000 lb, take two samples. Test the samples separately.

8. Calibration and Standardization

8.1 Determination of Cell Constant:

8.1.1 The conductivity cell will come with a predetermined constant. This constant should be checked periodically, one method being as follows:

8.1.1.1 Prepare a standard solution such as a 0.0005 *N* solution of KCl by diluting a 0.02 *N* KCl solution with water or by dissolving 0.0372 g of KCl (heated before weighing for 1 h at 105°C) in water, followed by dilution to 1 L. Cool and measure the conductance at 25°C as described in Section 9. Calculate the cell constant, K_{25} , as follows:

$$K_{25} = (C_s/C_m)$$

where:

C_m = conductance, measured at 25°C (see 10.1), μmho , and
 C_s = conductivity, 72 $\mu\text{mho/cm}$ (from Table 1).

NOTE 3—In general the cell constant is not greatly affected by variations in the strength of the KCl solution, but, for greater accuracy, measurements should be made at or near the specific conductivity of the solution to be measured and at values that utilize the middle range of the scale of the conductivity bridge, using the same multiplier tap.

8.1.2 Table 1 gives values of specific conductivities for corresponding KCl solution concentrations which are useful for abrasive testing.

9. Procedure

9.1 Preparation of a Slurry Filtrate:

9.1.1 Rinse beakers, stirring rods, and funnels with reagent water until tests show the rinse water has a conductivity of 5.0 $\mu\text{mho/cm}$ or less.

9.1.2 Add 300 mL of water to 300 mL of abrasive and stir for 1 min with a stirring rod. Let stand for 8 min and then stir again for 1 min.

9.1.3 Filter sufficient supernatant liquid for tests, discarding the first 10 mL of the filtrate. The amount of supernatant liquid filtered shall be sufficient to cover the cell.

9.1.4 Rinse the conductivity cell in reagent water until the rinse water is a cleanliness of 5.0 $\mu\text{mho/cm}$ or less.

9.1.5 Rinse the conductivity cell two or three times with the filtrate then determine conductance at 25°C in accordance with the operating instructions of the instrument. Use

successive portions of the sample until a constant value is obtained.

10. Calculation

10.1 Calculate the specific conductivity of the abrasive as follows:

$$C_s = C_m \times K_{25}$$

11. Report

11.1 Report the following information:

11.1.1 The calibration value of the cell constant (both as measured and as predetermined and supplied with the conductivity cell), the date, and the name of the person checking the calibration.

11.1.2 The material, date, readings and mean in $\mu\text{mho/cm}$ along with name of person conducting the tests and identification of the apparatus.

12. Precision and Bias⁵

12.1 *Precision*—On the basis of five replicate interlaboratory tests of this test method in which three operators in three laboratories analyzed, in duplicate, six blasting abrasives containing ionogenic contamination, the within-laboratory coefficient of variation after rejecting results from one set of replicate tests as outliers, was found to be 1.7 % with 20 degrees of freedom (df) and the between-laboratory standard deviation coefficient of variation was found to be 7.4 % with 15 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of two runs obtained by the same operator should be considered suspect if they differ by more than 5 % relative.

12.1.2 *Reproducibility*—Two results, each the mean of two runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 22 % relative.

12.2 Bias:

12.2.1 Bias can be present because of the mobility of various ions. The hydrogen ion has a much greater mobility than the hydroxyl ion or other ions so that at low pH's the conductivity will be relatively higher than at high pH's for the same ionic concentration. However, the bias introduced by this factor is in the proper direction. That is, high conductivity due to a lower pH of the contamination would normally indicate greater corrosion potential.

12.2.2 A bias may be introduced by extraneous contamination or from reduced sensitivity of instruments for low levels of contamination in the range of conductivity between 0 and 30 $\mu\text{mho/cm}$.

13. Keywords

13.1 ionogenic; contamination; steel surfaces; abrasive; blasting; conductimetric; analysis; interlaboratory testing; precision; chloride; conductivity; salts.

⁵ Supporting data available from ASTM Headquarters. Request RR: D01-1061.

TABLE 1 Specific Conductivities for Potassium Chloride (KCl) Concentrations at 25°C

Normality	Heated, Dry KCl/Reagent Water Solution, g/L	KCl Conductivity, $\mu\text{mho/cm}$
0.0005	0.0373	72
0.001	0.0746	147
0.005	0.3728	716
0.01	0.7455	1414

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Standard Practice for Preparing Drawdowns of Artists' Paste Paints¹

This standard is issued under the fixed designation D 4941; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the production of uniform films of artists' tube paints and other nonflowing pigmented paints using paint applicators designed for less viscous paints.

1.2 Information on how to achieve opaque specimens from these paints is included.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²

D 4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints³

E 1164 Practice for Obtaining Spectrophotometric Data for Object-Color Evaluation⁴

3. Terminology

3.1 **Definitions**—See Definitions D 16 for definitions of terms used in this practice.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 **drawdown bar**—a metal applicator with a specified gap designed to deposit a wet paint film uniformly on a specified test panel (for example, an opacity chart) or other substrate.

3.2.2 **drawdown**—a layer of paint deposited on a substrate by use of a drawdown bar for the evaluation of paint characteristics.

4. Summary of Practice

4.1 The paint is spread over the area of the test panel to be covered by the drawdown and the bar is pulled down with pressure just sufficient to avoid lifting of the bar from the chart surface.

4.2 Test panels are allowed to dry in a dust-free environment.

4.3 If complete hiding (opacity) is needed and not produced by a single paint film, a second film is applied at a 90°

angle to the first. If required, additional layers may be applied using a narrower drawdown bar.

5. Significance and Use

5.1 Quality standards for artists' paints require the evaluation of various appearance characteristics of paint films. Tinting strength determination (Test Method D 4838) specifically requires the preparation of drawdowns for colorimetric measurement. Other evaluations such as color designation, transparency, gloss, and color difference measurements also require drawdown samples.

5.2 Artists' tube paints have a paste consistency that makes the use of traditional film application methods difficult, especially for drying oil paints.

5.3 Artists' paints vary in two properties important to the preparation of films, that is, transparency and drying time. Colorimetric determination and some other types of evaluation require paint specimens that completely hide the substrate. Very transparent paints require such a thick film to produce complete hiding that drying times are excessively long or the specimen surface is blemished. When complete hiding is necessary, this practice is designed to provide opaque films without these defects through application of a series of thin film.

6. Apparatus

6.1 **Drawdown Bars**, two, of different widths with a clearance of 0.006 in. (0.152 mm). Recommended widths are 3 in. (7.6 cm) and 6 in. (15.2 cm). The second bar is required to prepare drawdowns with more than two layers. Wire wound drawdown bars have been found to be unsuitable.⁵

6.2 **Drawdown Charts**, sealed paper type, half black and half white if transparency is being evaluated or opacity is necessary.⁶

7. Procedure

7.1 Attach chart to a firm, smooth, level plane surface using tape, a vacuum plate, or a clamp. Label the chart with the identity of the specimen and other data as required.

7.2 Mix paint sample thoroughly using two spatulas. Tube paints that have separated should be expelled completely and mixed.

NOTE 1—The use of coated freezer wrapping paper as a mixing surface simplifies clean up. The same paper can be used for weighing

¹ This specification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

Current edition approved April 28, 1989. Published June 1989.

² Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Suitable drawdown bars can be obtained from the Paul N. Gardner Co., P.O. Box 10688, Pompano Beach, FL 33061-6688; or BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

⁶ Suitable charts can be obtained from The Leneta Co., Box 576, Ho-Ho-Kus, NJ 07423.

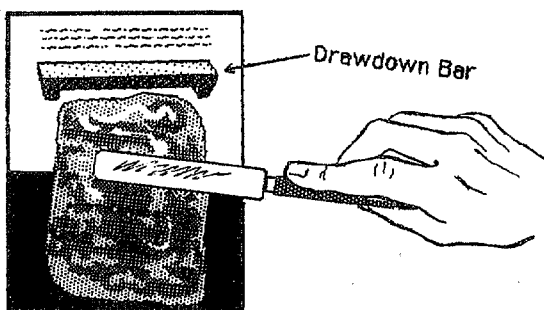


FIG. 1 Preparation for Drawdown

specimens when required as in the tinting strength determination (Test Method D 4838).

7.3 Using a spatula, spread the paint, in a thickness exceeding the bar's gap clearance, over the entire area to be covered by the drawdown. Do not cover the area to be contacted by the supporting feet of the wider of the two bars. Starting at the top of the chart and using the wider bar, draw the bar down over the paint in an even motion applying sufficient pressure to ensure that the resistance of the viscous paint does not raise the feet of the bar from the surface. When using oil paints the motion should be slow enough to allow for the high viscosity. Acrylic paints, which have short drying times, should be applied immediately after mixing to avoid premature film formation.

7.4 After drying in a horizontal position for 15 min, specimens may be hung in a vertical position in a dust-free area to complete the drying. Allow 24 h for acrylic and alkyd paints and 48 h or longer for oil paints until dry to touch.

7.5 Examine the specimen for surface defects and discard if unacceptable.

7.6 If complete hiding is required, compare, either visually or instrumentally, the lightness of the paint on the black portion of the chart with that on the white. For instrumental evaluation, follow the procedures given in Practice E 1164 to determine CIE Y for each portion, then calculate the contrast ratio of the paint film on the two portions of the chart ($CIE Y_{black}/CIE Y_{white}$). A contrast ratio of 0.98 or higher is considered opaque.

7.6.1 If opacity is not sufficient, rotate the chart through a

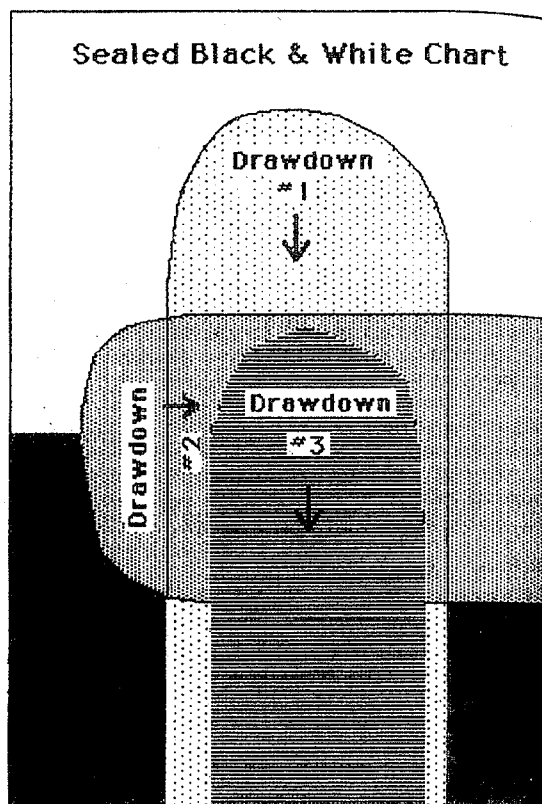


FIG. 2 Three Overlapping Drawdowns

90° angle and deposit a second film using the wider bar to repeat the procedure in 7.3 through 7.5. If additional layers are required, repeat this sequence using the narrower bar. Be careful each time that the feet of the drawdown bar ride on the surface of the combined lower layers. See Figs. 1 and 2.

NOTE 2—The most transparent paints studied were found to require three coats; however, in some instances, such as with alizarin crimson oil paint, it may not be practical to obtain complete hiding.

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Standard Test Methods for Water Pickup of Lithographic Printing Inks and Vehicles in a Laboratory Mixer¹

This standard is issued under the fixed designation D 4942; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 These test methods cover two procedures for determining the amount of water picked up by lithographic printing inks in a laboratory mixer.

1.2 Test Method A covers single-point water pickup; Test Method B covers the rate of water pickup. Both test methods are applicable to any printing ink and vehicle intended for the lithographic printing process.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Summary of Test Method

2.1 These test methods utilize a laboratory mixer for beating water or other agreed upon fluid into the test ink.

2.2 For single-point water pickup (Test Method A), 50 mL of water is normally added to 50 g of ink and mixed in for 5 min. The water picked up is determined from volumetric measurements of free water.

2.3 For rate of water pickup (Test Method B), water is added to 50 g of ink in increments of 20 mL and mixed in for 1 min or more over a cumulative time period totaling 10 min. The water taken up by the ink after each mixing interval is determined gravimetrically.

Significance and Use

3.1 The lithographic printing process requires that some dampening solution be emulsified into the ink. These test methods provide a rapid means for determining water pickup under laboratory conditions. Test results may be useful for specification acceptance between the supplier and the customer.

3.2 In order that results be comparable, the tests must be run at the same temperature and with the same type and quantity of liquid added prior to mixing.

3.3 The emulsions obtained in these test methods are of larger particle size than those typically produced in printing shops. Because of these and other variables in the printing process, water pickup results do not by themselves predict lithographic printing performance.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved April 28, 1989. Published June 1989.

4. Apparatus

4.1 *Laboratory Mixer*,² such as a Duke Ink-Water Emulsification Tester² equipped with a stainless steel specimen bowl 83 mm wide and 88 mm high, mixer blades that rotate at 90 r/min, and a timing device.

4.2 *Balance*, accurate to 0.1 g, 600-g capacity.

4.3 *Palette knives*, two.

4.4 *Thermometer*, quick response.

4.5 *pH Meter* (optional).

4.6 *Conductivity Meter* (optional).

4.7 *Graduated Cylinder*, 50 or 100-mL.

5. Reagents and Materials

5.1 *Water*—Deionized or distilled water, preferably having a pH of 5.0 to 7.0 (100 to 200 mL per sample); alternatively, fountain solution or other aqueous medium as agreed upon between the supplier and the customer may be used.

5.2 *Cleanup Materials*—Naphtha and rags or tissues.

6. Test Specimen

6.1 A minimum of 100 g is sufficient for two determinations. Before removing ink from the can, stir or otherwise ensure that the ink specimen is representative. Close the can and replace sealing tape immediately after each ink removal.

7. Conditioning

7.1 Condition the instrument, water, and ink samples in a constant temperature room or bath, preferably at $23 \pm 1^\circ\text{C}$.

7.2 Prior to use, check the alignment of the mixer blades. With the power switch of the mixer in the off position, set the clean bowl into the turntable and engage the locking pin firmly into the slot in the side of the turntable. Tilt the mixer head back and insert the blades, marked left and right, into their respective holders. Lower the mixer head. If the blades hit the side or bottom of the bowl, return the instrument to the manufacturer for realignment.

8. Test Method A—Single Point Water Pickup (by Volumetry)

8.1 Program the counter of the mixer for 5 min mixing time (450 revolutions).

8.2 *Optional*—If the first run of the day, pour test water into a beaker. Measure pH, conductivity, and temperature at the beginning of testing.

8.3 Weigh or tare the clean dry mixing bowl. Add $50 \pm$

² Available from Duke Custom Systems, 8371 Highway 49, Pleasant View, TN 37146.

0.1 g of the ink to the center of the bowl.

8.4 Pour 50 mL of water (from 8.2) into a graduated cylinder. If the ink is expected to pick up more than 100 % water, use 100 mL of water. Adjust the volume to ± 0.5 mL. Add the entire contents to the bowl.

8.5 With the mixer head tilted back insert the clean blades, marked left and right, into their respective holders. Lock the bowl on the turntable. Lower the mixer head. Press the counter reset button, making sure that 450 is displayed on the face of the counter.

8.6 Turn the mixer on. Examine contents of the bowl as mixing progresses. If 50 mL of water had been added and all of it disappears into the ink, stop, discard the ink in the bowl, clean up, and start over from 8.3, adding 100 mL of water in 8.4. The latter quantity must also be used for all other inks in the series under study.

NOTE 1—With some inks, water pickup is affected by the amount of water added prior to mixing. When 50 mL is insufficient, do not simply add another 50 mL during the run, as test results may differ significantly from those obtained by adding 100 mL at the outset.

8.7 When the mixer stops, turn the power switch off. Tilt the head out of the ink, detach the mixing blades, and add to the bowl.

8.8 Remove the bowl from the turntable and, holding the blades at the side of the bowl, decant the free water into a graduated cylinder. Run the blades *very slowly* through the ink in the bowl. Decant additional free water into the cylinder.

NOTE 2—Do not knock the bowl to force free water from the surface. Always handle the bowl gently to avoid breaking the emulsion.

8.9 Record the returned water level to 0.5 mL.

8.10 *Optional*—Measure the temperature, pH, and conductivity of the returned water. Note the appearance of the water and the consistency of the ink and the appearance of the returned water.

8.11 Discard ink left in the bowl. Clean the bowl and the mixer blades with tissue wetted with naphtha. Discard the returned water and rinse the cylinder clean.

8.12 Repeat 8.3 through 8.10 with a second specimen of the same ink.

9. Test Method B—Rate of Water Pickup (by Gravimetry)

9.1 Program the counter for the first interval of the mixing cycle.

NOTE 3—A commonly used cycle is 1-min intervals (90 revolutions) times ten determinations. Intervals need not be uniform, for example, 1, 2, 3, 5, and 10 min (90 times 3 plus 180 plus 450 revolutions).

9.2 *Optional*—Measure water properties in accordance with 8.2.

9.3 Weigh or tare the clean dry mixing bowl and blades on the balance. Add 50 ± 0.1 g of ink to the center of the bowl.

9.4 Lock the bowl on the platform of the mixer. With the mixer head raised, carefully insert the blades into their respective holders. If ink on one blade touches the upper parts of the other blade or the side of the bowl, carefully remove the ink with two palette knives and transfer to the bottom of the bowl. Lower the mixer head.

9.5 Pour 100 mL of water (from 8.2) into a beaker. Meter out 20 mL and add to the bowl.

9.6 Press the counter reset button, making sure that the

desired number of revolutions is displayed on the face of the counter. Turn the mixer on. Examine the contents of the bowl as mixing progresses. If all liquid disappears into the ink, add more as needed to maintain a layer of excess water on the surface of the ink.

NOTE 4—Few specimens will take up more than 20 mL of water within a 1-min mixing interval. If a high-water pickup specimen is being run and the mixing interval is longer than 1 min, another 20 mL should be added prior to each subsequent minute of mixing time.

9.7 When the mixer stops, turn the power switch off. Detach the mixing blades and add to the bowl.

9.8 Remove the bowl from the turntable and, holding the blades at the side of the bowl, decant the free water into the beaker containing the unused water. Run the blades *very slowly* through the ink in the bowl. Decant additional free water into the beaker (see Note 2).

9.9 Weigh the mixing bowl and contents, including the blades.

9.10 Using a palette knife, transfer the ink from the walls to the center of the bowl. Return the bowl to the mixer. Replace the blades as in 8.4.

9.11 For the next mixing interval, swirl the beaker in order to mix the returned and unused water. Meter out 20 mL and add to the bowl. Press the counter reset (or change the counter) and turn the power on. Add more water if needed to maintain an excess layer (see Note 4).

9.12 When the mixer stops, repeat 9.7 through 9.11 until the cumulative mixing time totals at least 10 min.

9.13 *Optional*—At the end of the run, make measurements in accordance with 8.10.

9.14 Discard the ink left in the bowl. Clean the bowl and the mixer blades with tissue wetted with naphtha. Discard returned water and rinse the beaker clean.

9.15 Repeat 9.3 through 9.14 with another specimen of the same ink.

10. Calculation

10.1 Calculate water pickup, P , as follows:

10.1.1 *Test Method A—Volumetric:*

$$P = (V_1 - V_2) \times 2$$

where:

P = water pickup, % or mL water/100 g ink,

V_1 = volume of water added, mL, and

V_2 = volume of returned water, mL.

10.1.2 *Test Method B—Gravimetric:*

$$P = (W - S) \times 2$$

where:

P = water pickup, % or g water/100 g ink,

W = weight of the specimen plus water picked up after each mixing interval, g, and

S = weight of initial specimen, g.

NOTE 5—The conversion from water pickup of the ink to water content, C , of the emulsion is $C = P/(100 + P)$. Units are percent or grams of water per 100 grams of emulsion.

11. Report

11.1 Report the following information:

11.1.1 The percent water pickup to the nearest whole number as the mean of the two determinations, the cumulative mixing time, and a description of the water used for

testing (for example, tap water, deionized water, or type of fountain solution).

11.1.2 If rate of water pickup was determined, plot the percent of water pickup versus the cumulative mixing time.

11.1.3 *Optional*—The mean temperature, changes in pH, conductivity, appearance of the water, and the change in consistency of the ink.

12. Precision and Bias

12.1 Precision:

12.1.1 *Test Method A*—An interlaboratory study of single-point water pickup by Test Method A was conducted in which one operator in each of eleven laboratories tested in duplicate on each of two days three lithographic printing inks ranging in 5-min water pickup from 50 to 65 %. One company was found to be an outlier and was deleted from the analysis. The within laboratory pooled standard deviation was found to be 1.58 % absolute (millilitre of water per 100 grams of ink) at 9 degrees of freedom (df), and the between laboratories pooled standard deviation was 7.1 % absolute at 30 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1.1 *Repeatability*—Two results, each the mean of two runs obtained by one operator, should be considered suspect if they differ by more than 4.5 % absolute.

12.1.1.2 *Reproducibility*—Two results, each the mean of two runs obtained by operators in different laboratories, should be considered suspect if they differ by more than 20 % absolute.

12.1.2 *Test Method B*—In an interlaboratory study of rate

of water pickup by Test Method B, water pickup values at 2½, 5, 7½ and 10 min were determined twice on one day by one operator in each of nine laboratories on six inks. The inks ranged in water pickup from 40 to 52 % at 2½ min and from 65 to 100 % at 10 min. After rejecting 12 out of 156 replicated test values as outliers, the within laboratory pooled standard deviation was found to be 1.58 % absolute (grams of water per 100 grams ink) with 97 df and the between laboratory standard deviation 3.73 % absolute with 86 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.2.1 *Repeatability*—Repeatability cannot be determined as both runs were conducted on the same day.

12.1.2.2 *Reproducibility*—Two water pickup curves, each the mean of two runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 10.5 % absolute.

12.2 *Bias*—Bias cannot be determined because there are no standard materials. The poorer interlaboratory precision of Test Method A compared to Test Method B is believed to be caused by the fact that the gross quantity of water added at one time is picked up as large globules which make it difficult for different operators to release free water in the same manner.

13. Keywords

13.1 lithographic printing inks; printing inks; inks; vehicles; water pickup; water content; emulsification; fountain solution; mixers

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Standard Test Method for Blocking Resistance of Architectural Paints¹

This standard is issued under the fixed designation D 4946; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

¹ NOTE—Paragraphs 6.5, 7.2, and 7.5 were editorially corrected in October 1990.

1. Scope

1.1 This test method describes an accelerated procedure for evaluating the face-to-face blocking resistance of trades sales paints. This is not to be confused with blocking resistance Test Method D 3003 which is concerned with blocking of industrial coatings on metal substrates, nor with Test Method D 2793 which is concerned specifically with wood product finishes and reports results on a satisfactory or not satisfactory basis, rather than by the degree of blocking tendency as in this test method.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 2793 Test Method for Blocking Resistance of Organic Coatings on Wood Substrates²

D 3003 Test Method for Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates²

3. Terminology

3.1 Definition:

3.1.1 *blocking*—The undesirable sticking together of two painted surfaces when pressed together or placed in contact with each other for an extended period of time.

4. Summary of Test Method

4.1 Dried paint films are placed face-to-face and a pressure of about 1.8 psi (127 g/cm²) is applied. These paint films are put into an oven for 30 min to make the test more stringent. After cooling, the blocked panels are peeled apart. The degree of blocking is rated subjectively for tack or seal using a series of standard descriptive terms corresponding to numerical ASTM values of 10 to 0.

5. Significance and Use

5.1 Dry paint often comes in contact with itself especially in window and door areas and, depending on its hardness,

the pressure, temperature, humidity, and duration of time the surfaces are in contact, sometimes sticks to itself (blocks). This stringent test method can be used to compare and rate subjectively the resistance of paints to blocking.

6. Apparatus

6.1 *Conditioned Room*, at 65 to 85°F (18 to 29.5°C) and 40 to 60 % relative humidity.

6.2 *Sealed Paper Test Charts*, approximately 7.5 by 11 in. (190 by 280 mm).³

6.3 *Applicator Blade*, 5 to 6 in. (13 to 15 cm) wide, 6 mil clearance.

6.4 *Oven*, 115 to 125°F (48 to 52°C).

6.5 *Rubber Stoppers*, No. 8, 1.25 in. (3.2 cm) smaller diameter.

6.6 *Weights*, 1000 g.

6.7 *Scissors*.

6.8 *Aluminum Tray or Pan*, flat.

7. Procedure

7.1 Cast the paint to be tested on a sealed test chart using the applicator blade. Condition coated panels in the conditioned room for seven days. All painted panels should be kept free of grease, oil, or fingerprints since these will affect block resistance.

7.2 After the panels have been conditioned, cut out six 1½ by 1½-in. (3.8 by 3.8-cm) sections from the painted chart. Start the cut at least ½ in. (1.3 cm) away from the edge of the drawdown.

7.3 Place the cut sections with the paint surfaces face-to-face for each paint to be tested.

7.4 The weights, stoppers, and tray should be temperature equilibrated in the oven prior to running the test.

7.5 Place the face-to-face specimens in the oven on a flat aluminum tray. Place a No. 8 stopper on top, with the small diameter in contact with the specimens, then place a 1000-g weight on top of the stopper. This results in a pressure of 1.8 psi (127 g/cm²) on the specimens. One weight and stopper is to be used for each specimen to be tested. It is recommended that "pass" and "fail" paint controls be used in each test run and that the tests be run in triplicate.

7.6 After exactly 30 min, take the stoppers and weights off the test specimens and remove them from the oven. Allow them to cool for ½ h in the conditioned room before determining the block resistance.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved May 26, 1989. Published July 1989.

² Annual Book of ASTM Standards, Vol 06.01.

³ Form WB available from The Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ 07423 has been found satisfactory for this purpose.

7.7 After cooling, separate the specimens by peeling them apart with a slow and steady force at about 180° from each other forming a "T" pattern during beginning of the separation. It is necessary to put the specimen next to the ear while separating to actually hear the degree of tack. Rate for blocking resistance on a scale of 0 to 10 (see 8.2).

8. Interpretation of Results

8.1 Blocking resistance is rated on a scale of 10 to 0, which corresponds to a subjective tack (sound of separation when peeled) or seal (the complete sticking together) rating determined by the operator. This rating system is defined in 8.2 in the appropriate descriptive terms. The degree of seal is the estimated area on the specimens where the paint surfaces adhere and some of the paper tears away from the chart when peeled.

8.2 Blocking Resistance Ratings:

Blocking Resistance Numerical Ratings	Type of Separation	Performance
10	no tack	perfect
9	trace tack	excellent
8	very slight tack	very good

Blocking Resistance Numerical Ratings	Type of Separation	Performance
7	very slight to slight tack	good to very good
6	slight tack	good
5	moderate tack	fair
4	very tacky; no seal	poor to fair
3	5 to 25 % seal	poor
2	25 to 50 % seal	poor
1	50 to 75 % seal	very poor
0	75 to 100 % seal	very poor

9. Report

9.1 Report the blocking resistance rating determined in accordance with 8.2.

10. Precision

10.1 Data are unavailable for a conventional precision statement. However, based on actual laboratory experience, with experienced operators, the repeatability is estimated to be plus or minus one blocking resistance unit. Numerical values may differ from operator to operator but relative ranking should be about the same. As in many tests the precision improves with practice.

11. Keywords

11.1 blocking; blocking resistance; sticking; tack

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Standard Test Method for Determination of the Upper Layer Separated from a Viscous Liquid¹

This standard is issued under the fixed designation D 4948; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of the amount of liquid separated as an upper layer in a 24-h period from viscous solutions or dispersions that contain dispersed solids such as paints, enamels, pigmented lacquers, adhesives, polishes, and other similar materials.

NOTE—The amount of clear liquid that separates during this test is one of the criteria in the United Nations Recommendations on the Transportation of Dangerous Goods² for the placement of flammable viscous liquids into packing groups related to flash points (See 4.1).

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 *ASTM Standard:*
D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings³

3. Summary of Test Method

3.1 A measured amount of the material to be tested is placed in a graduated cylinder and allowed to stand undisturbed for 24 h. The volume percent of the separated top layer is then determined.

4. Significance and Use

4.1 The United Nations Committee of Experts on the Transport of Dangerous Goods in their recommended regulations place materials having a flash point below 73.5°F (23°C) in Packing Group II. However, if viscous substances such as paint and related coatings, adhesives, polishes, etc., meet certain requirements, they can be placed in Group III along with materials having a flash point between 73.5 and 140°F (23 and 60.5°C). One of the requirements is that less than 3 % of clear liquid separates from the bulk of the material when subjected to this test method.

4.2 At the present time most international regulatory bodies such as the International Civil Aviation Organization

(ICAO) and the International Maritime Organization (IMO) use the U.N. Recommendations. It is anticipated that most national transportation regulatory bodies will adopt the U.N. Recommendations as their regulations for control of transportation of hazardous materials. At present the United States permits the transshipment of hazardous materials through the United States to other countries under regulations of the IMO and ICAO.

5. Apparatus

5.1 *Graduated Cylinder*,⁴ 100-mL, glass-stoppered, 250-mm total height and 30-mm inside diameter.

5.2 *Constant Temperature Cabinet or Room*, at 73.5 ± 3.5°F (23 ± 2°C).

6. Sampling

6.1 Obtain a uniform quart sample of the material using standard methods for sampling in accordance with Practice D 3925.

7. Conditioning

7.1 Store the sample or a representative portion at a constant temperature of 73.5 ± 3.5°F (23 ± 2°C) until the sample reaches a uniform temperature of 73.5°F (23°C).

8. Procedure

8.1 Thoroughly stir the sample to obtain uniform consistency and, in the case of materials containing particles, uniform dispersion of these particles.

8.2 Immediately pour 100 mL of the sample into the graduated cylinder. Insert a stopper and leave the cylinder undisturbed at 73.5 ± 3.5°F (23°C) for 24 h.

8.3 After 24 h, measure the volume of any upper separated layer and determine the percent of the separated layer compared to the total volume of the specimen.

9. Calculation

9.1 Calculate the volume percent of the clear upper liquid layer P_c as follows:

$$P_c = (V_c/V_m) \times 100$$

where:

V_c = volume of the clear upper liquid layer, mL, and
 V_m = total volume of the 100-mL specimen, mL.

⁴ A graduated cylinder, Catalog No. 2982-100, available from Corning Glass Works, Corning, NY 14831, has been found satisfactory for this purpose.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.22 on Health and Safety.

Current edition approved May 26, 1989. Published July 1989.

² *Transport of Dangerous Goods: Recommendations of the Committee of Experts on the Transport of Dangerous Goods*, United Nations, 3rd ed., New York, 1984.

³ *Annual Book of ASTM Standards*, Vol 06.01.

10. Report

10.1 Report the volume percent of the separated clear liquid to within 0.5 %.

11. Precision and Bias

11.1 The precision of this test method will be determined.

11.2 Bias cannot be determined since no standards are available.

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Standard Test Method for Comparison of the Brush Drag of Latex Paints¹

This standard is issued under the fixed designation D 4958; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is a standardized brushout procedure for comparing the brush drag of architectural type latex paints.

1.2 With slight modifications this test method is also applicable to solvent paints.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1475 Test Method for Density of Paint, Varnish, Lacquer and Related Materials²
- D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer and Related Materials²
- D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²
- D 4287 Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer²

3. Terminology

3.1 **Definitions**—See *Paint/Coatings Dictionary*³ for definition of terms used in this test method.

3.1.1 *brush-drag*—resistance encountered when applying a coating by brush.

4. Summary of Test Method

4.1 A 2-in. (50-mm) polyester brush is used to apply the test paint on a 1.076-ft² (1000-cm²) test area. The application is made at a spreading rate of 400 ft²/gal (9.82 m²/L) and is completed in 30 to 35 s. The degree of brush drag is rated subjectively using a series of standard descriptive terms corresponding to numerical values of 1 to 10. The rank order of a set of samples is thereby established.

5. Significance and Use

5.1 As the brush drag of a paint increases, any natural tendency on the part of the painter to overspread the paint is

reduced. When all other factors are held constant, increased brush drag will result in greater film thickness with consequent improvement in durability and hiding. Conversely, sometimes it might be preferred to have a lesser degree of brush drag for easier application (that is, the amount of time and effort in applying a paint to a specific area is reduced with a lesser degree of brush drag).

5.2 This test method provides a standardized brushout procedure for the evaluation of brush drag as an alternative to customary informal ad hoc procedures. Its objective is to maximize the reliability and precision with which this characteristic may be determined.

NOTE 1—The brush drag of paints is directly related to their high-shear viscosity. There is generally good rank order agreement between results obtained by this method and Test Method D 4287. The sensitivity of this brushout method has been found sufficient to distinguish between brushabilities corresponding to high-shear viscosity differences not lower than 0.3 poise (0.03 Pa.s). Round robin data show that rank order agreement between the brushout and viscometric methods is poor when latex and solvent paints are part of the same comparison group. This is the result of these two paint types having markedly different rheological properties that affect the relative perception of brush drag.⁴

6. Apparatus

6.1 *Brush*, 2-in. (50-mm) polyester filament, 2¼-in. (70-mm) length-out, ⅞ in. (14 mm) thick, with a chiseled tip.

NOTE 2—All tests of a given series of paints, within or between laboratories, should be carried out with commercially identical brushes.

6.2 *Stopwatch*.

6.3 *Balance*, capable of weighing accurately to 0.1 g.

6.4 *Test charts*, with a sealed surface, having 1.076 ft² (1000 cm²) of test area.⁵

7. Sampling and Conditioning

7.1 Sample in accordance with Practice D 3925.

7.2 Condition the samples in accordance with the Conditioning and Testing section of Specification D 3924.

7.3 All testing should be performed under the same conditions.

8. Procedure

8.1 Do not change operators during the running of a series of specimens, since this will invalidate any conclusions as to rank order.

8.2 Determine the density in pounds per gallon of the paint sample in accordance with Test Method D 1475.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 4958 - 89. Last previous edition D 4958 - 89.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Available from Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

⁴ Supporting data are available from ASTM Headquarters. Request RR: D01-1072.

⁵ Leneta Form 8H-BW, obtainable from The Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ 07423, has been found satisfactory for this purpose.

8.3 Multiply the density by 1.221 to obtain the weight of paint in grams to apply on the specified test chart to obtain a spreading rate of 400 ft²/gal (9.82 m²/L).

8.4 *Conditioning of the Brush:*

8.4.1 Soak the brush in clean water, then spin rapidly to remove the water from the bristles as completely as possible.

8.4.2 In order to equalize the amount of paint contained on the brush, dip the brush into the can to take up a normal brush load and paint out approximately 1/2 ft² (0.05 m²) of a reasonably well sealed surface (for example, any previously painted test chart).

8.4.3 Repeat 8.4.2 utilizing another dry surface.

8.5 Place the test chart on the balance and weigh the correct amount of paint as calculated in 8.3 directly onto the center of the card.

8.6 Immediately tape the card onto a hard, flat surface, start the stopwatch, and proceed to spread the paint using the previously conditioned 2-in. (50-mm) brush. Using long, steady brush strokes, alternately parallel and perpendicular to the edge of the chart, cover the test area uniformly and completely in 30 to 35 s.

8.7 Immediately assign and record a brush drag rating according to the following series of qualitative descriptive terms, first characterizing it by a verbal description, and then by the corresponding number.

- 1—Very slight
- 2—Slight
- 3—Slight to moderate
- 4—Moderate
- 5—Moderate to considerable
- 6—Considerable
- 7—Considerable to pronounced
- 8—Pronounced
- 9—Very pronounced
- 10—Extreme

8.8 Thoroughly clean the brush with warm water and spin to remove excess water between tests.

8.9 Repeat 8.2 through 8.8 for each specimen in the set

and rate the specimen as the mean of the two results.

8.10 If more than one specimen has the same rating, brush out the similarly rated specimens again, in close comparison. If small differences are perceived, then indicate these by assigning intermediate decimal values. If no difference is found then the original ratings stand.

9. Interpretation of Results

9.1 Tabulate the paints in order of their brush drag ratings, showing verbal descriptions and numerical ratings in separate columns.

9.2 In a fourth rank order column, rank the paints from 1 to *n* (least to most brush drag), where *n* is the total number of paints in the series.

9.3 Paints with the same qualitative ratings should be assigned multiple rank numbers, with the mean of those numbers shown in parentheses, for example, 3 to 4 (3.5), 5 to 7 (6). The mean ranking value (in parentheses) is used to calculate an average ranking value when the same series of paints is ranked by more than one operator.

10. Report

10.1 Report the brush drag ranking as determined in Section 9.

11. Precision and Bias

11.1 In an interlaboratory study of this test method in which five coatings varying widely in brush drag were ranked by one operator in each of nine laboratories, two operators in one laboratory, and three operators in another laboratory, the coefficient of concordance (agreement in ranking) was found to be 0.84, reflecting the fact that seven of the fourteen operators agreed perfectly and four others reversed one of two adjacent pairs. The coefficient is statistically significant at the 99.9 % confidence level.

11.2 Bias has not been determined for this test method.

12. Keywords

12.1 brush drag; high shear viscosity; brushability; ease of brushing; drag

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Evaluation of Color for Thermoplastic Traffic Marking Materials¹

This standard is issued under the fixed designation D 4960; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the instrumental determination of color of thermoplastic traffic marking materials in the CIE 1931 system.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 883 Definitions of Terms Relating to Plastics²
- E 97 Test Method for Directional Reflectance Factor, 45-Deg 0-Deg, of Opaque Specimens by Broad-Band Filter Reflectometry³
- E 179 Practice for Selection of Geometric Conditions for Measurement of Reflectance and Transmission Properties of Materials⁴
- E 284 Terminology Relating to Appearance of Materials⁴
- E 308 Test Method for Computing the Colors of Objects by Using the CIE System⁴
- E 1164 Practice for Obtaining Spectrophotometric Data for Object-Color Evaluation⁴
- F 412 Definitions of Terms Relating to Plastic Piping Systems⁵

3. Terminology

3.1 **Definitions**—Definitions are in accordance with Definitions D 883, E 284 and F 412, unless otherwise indicated.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 **thermochromism**—a color hue change that takes place in the thermoplastic material due to temperature changes.

3.2.2 **thermoplastic traffic marking material**—a highly filled 100 % total solids highway marking material that when heated to a molten state can be extruded or sprayed onto a road surface and when cooled forms a solid, durable delineator.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is under the direct responsibility of Subcommittee D01.44 on Traffic Coatings.

Current edition approved June 30, 1989. Published August 1989.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Annual Book of ASTM Standards, Vol 08.04.

4. Summary of Test Method

4.1 The thermoplastic specimen is prepared for this test by melting a sample to its application temperature under continuous agitation and then pouring it into a TFE-fluorocarbon coated pan, to form a patty of approximately 3 in. (7.6 cm) in diameter. The patty is allowed to cool to room temperature before measuring the color. Color measurements are made on the flat side or the top side of the thermoplastic patty.

NOTE 1—No significant color differences are encountered in reading the top or bottom of the patty.

5. Significance and Use

5.1 This test method provides a standard procedure for the determination of color of thermoplastic traffic marking materials. This test method can be used in conjunction with specifications to determine the uniformity of thermoplastic traffic marking materials from batch to batch and that produced by various suppliers.

5.2 There is a slight variation in color standards and colorimeters. This test method is only applicable when results are reported with the instrument model designation and white color calibration standard identification information.

6. Sampling

6.1 Samples may be obtained by an appropriate quartering or riffle sampling method where deemed necessary considering the physical form of the material.

7. Apparatus

7.1 **Agitator Blade**, 6 in. (15 cm) long with a 1/2-in. (1-cm) steel shaft and a 1 3/4 by 1 by 1/8-in. (4.5 by 2.5 by 0.3-cm) straight horizontal steel blade.

7.2 **Drill Press**, or other apparatus capable of agitating the thermoplastic marking material in the electric pots at 600 to 700 r/min during meltdown to the application temperature.

7.3 Heating Equipment:

7.3.1 **Gravity Convection Oven**, capable of maintaining 260°C, for melting the thermoplastic traffic marking.

7.3.2 **Hot Plate**, capable of maintaining 537°C.

7.3.3 **Insulated Electric Pots**, for heating and melting the thermoplastic traffic marking materials.

7.4 **Color Measuring Instrument**, conforming to all requirements of Test Method E 97, Practices E 179 and E 1164, and Method E 308 (geometry 45°/0°, illuminant C, 2° observer).

7.5 **Spatulas**, for stirring the thermoplastic traffic marking

during meltdown on the hot plate or in the gravity convection oven.

7.6 TFE-fluorocarbon Baking Pans or Uncoated Pint Can Lids, for forming 3-in. (7.6-cm) diameter patties.

8. Procedure

8.1 Taking care to prevent scorching of the material, melt a 1000 ± 50 -g sample of the thermoplastic marking material to a temperature of 218°C under continuous agitation, by one of the following means:

8.1.1 On a hot plate set at 537°C and using a spatula as the means of agitation.

8.1.2 In an insulated electric pot with a heat setting sufficient to reach the test temperature and with agitation of 600 to 700 r/min from an electric drill press or other suitable means.

8.1.3 In an oven set at 260°C with agitation by stirring with a spatula after the first 15 min and thereafter at 15-min intervals. The first stirring at 15 min is critical to prevent scorching of the thermoplastic marking material. The sample must be completely wet in on the first stir to ensure even melting and complete blending of the components of the thermoplastic material.

8.2 Pour the thermoplastic sample into a clean, TFE-fluorocarbon-lined pan, to form a 3-in. (7.6-cm) diameter patty. If a TFE-fluorocarbon pan is not available, pour the sample into an uncoated pint tin lid to form a 3-in. diameter patty. Before pouring the patty, the sample must be agitated well to prevent settling of the components and to provide a smooth homogeneous surface for color measurement.

8.3 Allow the patty to cool to room temperature for a minimum of 30 min and not to exceed 45 min.

NOTE 2—A 30 ± 5 -min conditioning of the patty negates the initial effects of thermochromism.

8.4 Calibrate the color measuring instrument with a white calibration color standard according to the instructions supplied by the manufacturer.

8.5 Remove the patty from the TFE-fluorocarbon pan and read the color measurement values from the flat smooth side. If a pint tin lid is used then read the top of the patty. Without removing the patty from the sample port immediately take three readings. Record only the third reading for each Y, x, and y value to further compensate for any thermochromism of the thermoplastic marking material.

8.6 A small port adapter, if available, for the color measuring instrument of approximately $\frac{3}{4}$ in. (19 mm) may be used to negate the effects of geometry and texture when reading the patties. This will measure in between small surface imperfections characteristic of thermoplastic traffic marking.

9. Report

9.1 Report the following information:

9.1.1 The formula code, batch number, formula type, and color for each patty read.

9.1.2 The type of color measuring instrument used and the identification of the white color calibration standard, and

9.1.3 The exact cooling period and values Y, x and y for each sample.

10. Precision and Bias

10.1 No general statement of precision can be made because of lack of sufficient data at this time.

10.2 No statement of bias can be prepared for this test method since there is no absolute test method for use as a comparative basis.

11. Keywords

11.1 thermoplastic traffic marking; color measurement.

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Standard Test Method for Wet-to-Dry Hiding Change¹

This standard is issued under the fixed designation D 5007; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of the change in hiding power of an architectural coating during drying, by visual evaluation of the wet and dry film.

1.2 This test method is not recommended for colors other than white and tints.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 2805 Test Method for Hiding Power of Paints by Reflectometry²

D 3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials²

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *hiding index, h_s* —the Spreading Index at a standard film opacity. In this test method the latter is a visual contrast standard prepared by applying a semi-opaque white coating on a black and white hiding-power chart to give a contrast ratio of 0.98 which is just short of complete hiding, and is the conventional so-called *full hiding* end point in photometric hiding-power methods such as Test Method D 2805. Refer to the hiding indices of the wet and dry films as h_{sw} and h_{sd} respectively.

3.1.1.1 *Discussion*—Since this test method is intended to measure hiding-power difference rather than hiding power itself, a 98 % contrast ratio standard is not required. It is necessary only that the same standard be used for measuring both wet and dry films. See 3.1.8.1. For this purpose the standard needs to lie within the contrast range of both the wet and dry stripes, which will be true of the 98 % contrast-ratio standard for most commercial paints. With paints of unusually low hiding, a lower contrast standard may be required, which can be simply one of the stripes taken from a drawdown of the test paint.

3.1.2 *hiding power*—the spreading rate of a paint applied uniformly on a standard black and white hiding power chart

to give a standard degree of contrast just short of complete hiding.

3.1.2.1 *Discussion*—In reflectometry the standard contrast for hiding power measurements is generally accepted as the contrast ratio $C = 0.98$, which with white and light tinted coatings is equivalent to a visual color difference of about 0.75 CIELAB units. That amount of color difference can reasonably be described as “just-short-of complete-hiding.” Since this is a visual method it employs a visual comparator as a standard, which is a hiding power chart with a white coating applied at a contrast ratio of 0.98.

3.1.3 *logicator*—a multi-notch applicator with clearances, and corresponding wet film thicknesses and spreading rates, in equal percentage steps.

3.1.4 *logicator scale*—a scale whose values are in direct, linear relationship with the logarithms of corresponding spreading rates. A specified change in scale value represents the same percentage change in spreading rate over any part of such a scale.

3.1.5 *spreading index, h* —the spreading rate expressed in logicator scale units (LU) as described in 3.1.4 and 4.1.7.

3.1.6 *spreading rate, H* —the area covered per unit quantity of coating. (In this test method the quantity is volumetric).

3.1.7 *TG19 logicator*—a logicator designed for this test method with eight notches numbered at four-unit intervals on a scale from 20 to 48, the notch clearances ranging from 2.65 to 10.4 mils (67 to 264 μm) corresponding to wet film thicknesses from 1.46 to 5.7 mils (37 to 145 μm) and spreading rates from 280 to 1100 ft^2/gal (6.9 to 27 m^2/L), with one scale unit representing a change of 5 % and the four-unit interval between notches a cumulative change of 21.55 % in the clearance and corresponding film thicknesses and spreading rates. Refer to this scale unit as a logicator unit (LU). (See Fig. 1.)

3.1.7.1 *Discussion*—The percentage difference between notches is calculated as $(1.05^4 - 1) \times 100 = 21.55$. This percentage is applicable precisely to the notch clearances and approximately to their related wet-film thicknesses and spreading rates. The detailed relationships between scalar value and the notch clearance, wet film thickness, and spreading rate are given in Tables 1 and 2.

3.1.8 *wet-to-dry hiding change (WDHC)*—the difference in the Hiding Index of a paint between the wet and the dry state, expressed in logicator units (LU) as follows:

$$\text{WDHC} = \Delta h_s = h_{sd} - h_{sw}$$

3.1.8.1 *Discussion*—The WDHC is unchanged if the contrast level of the hiding standard is varied, because the resultant changes in the two hiding power values will be proportional and their ratio therefore constant.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.42 on Architectural Finishes.

Current edition approved October 27, 1989. Published December 1989.

² Annual Book of ASTM Standards, Vol 06.01.

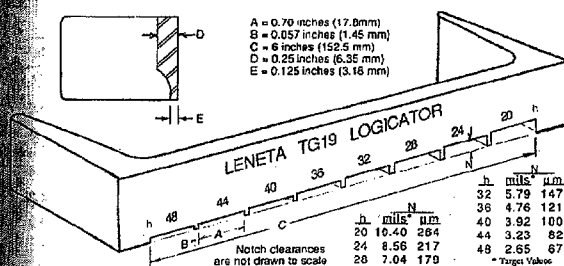


FIG. 1 Diagram of the Leneta TG19 Logicator

3.1.8.2 Discussion—The WDHC is unaffected by deviations from the estimated film thickness/clearance ratio T/N (see Note, Table 1), because the ratio of the two recalculated hiding-power values is unchanged.

Summary of Test Method

4.1 The test paint is applied with a TG19 Logicator on a logicator test chart (see 6.2 and Fig. 2) and the drawdown examined in comparison with an agreed hiding standard immediately after application and again after drying.

4.2 The scale numbers of the wet and dry stripes that match the standard in contrast are recorded as the Wet Hiding Index, h_{sw} , and the Dry Hiding Index h_{sd} .

4.3 The difference between the wet and dry hiding indices is recorded as the wet-to-dry hiding change WDHC.

4.4 If desired, the percent change in hiding power corresponding to the WDHC value is calculated and reported.

Significance and Use

5.1 Many architectural paints, particularly white and light tints, change significantly in film opacity as they dry. An increase in hiding is sometimes associated with porosity and poor film integrity with conventionally formulated coatings. A decrease can result in a disappointing paint job. The wet-to-dry hiding change is therefore a property of great practical importance.

5.2 This test method can be used in paint specifications and as a short, simple procedure for evaluation and quality control.

Apparatus

6.1 TG19 Logicator³—A multi-notch, varied-clearance applicator designed specifically for this test method (see 3.1.7 and Fig. 1).

6.2 Logicator Test Charts³—Black and white hiding charts with a chevron-stripe pattern and scale numbers printed at the top corresponding to the paint stripes applied by the Logicator (see Fig. 2).

6.3 Vacuum Plate, for holding the chart flat while the drawdown is made.

6.4 Visual Hiding Standard⁴—A narrow stripe cut from a logicator test chart on which a durable glossy white coating had been applied to obtain a contrast ratio of 0.98. The stripe

TABLE 1 Equations Relating the Logicator Scale Value or Spreading Index, h , to the Notch Clearance, N , Wet Film Thickness, T , and Spreading Rate, H

NOTE—The relationships involving T and H are based on a presumed T/N ratio of 0.55, which ratio is approximate and somewhat variable, depending on coating rheology, drawdown technique, and clearance.

Inch-Pound Units (mils, ft ² /gal):	Metric Units (μ m, m ² /L):
$N = 1.05^{68-h} = 27.6 + 1.05^h$	$N = 25.4 \times 1.05^{68-h} = 701 + 1.05^h$
$h = 68 - 47.2 \log N$	$h = 134.3 - 47.2 \log N$
$T = 0.55 N = 15.18 + 1.05^h$	$T = 0.55 N = 385.5 + 1.05^h$
$h = 55.75 - 47.2 \log T$	$h = 122.05 - 47.2 \log T$
$H = 105.7 \times 1.05^h$	$H = 2.594 \times 1.05^h$
$h = 47.2 \log H - 95.52$	$h = 47.2 \log H - 19.54$

is mounted on a blank card for convenient handling, as illustrated in Fig. 3.

Procedure

7.1 Prepare a hiding standard as described in 6.4, or use the standard supplied with the logicator test charts.

NOTE 1—Strong colors were measured according to this test method using standards prepared with the self-same coatings. Some measurements were also made using the regular white standard. Due to poor correlation in these tests it was decided to exclude strong colors from the scope of the test method. However, should measurements with such colors be attempted anyway, it is of interest to note that the white standard apparently served as well for that purpose as a standard made with the identical color.

7.2 Place a test chart on the vacuum plate. Then place the logicator at the top of the chart with its arms extending toward the operator.

7.3 Spread about 6 mL of the test paint evenly in front of the logicator, then draw down the full length of the chart (about 10 in. (254 mm)) at a uniform speed such that the total drawdown time is about 2.5 to 3 s.

7.4 Promptly after film application, determine the Wet Hiding Index (h_{sw}) in accordance with 7.5.

7.5 Observation of the Hiding Index (h_s):

7.5.1 Place the chart vertically in a well-illuminated glare-free location.

7.5.2 Hold the hiding standard next to the chart and identify by number the stripe that the standard matches in contrast. This number is the Hiding Index.

7.5.3 If there is no exact match and it is difficult to decide which of two adjacent stripes is matched more closely, record their mean as the Hiding Index. However, if the standard is perceived as closer to one or the other of the two stripes, then report the Hiding Index as one more than the lower or one less than the higher, as the case may be.

7.6 Immediately after determining the Wet Hiding Index (h_{sw}), place the drawdown horizontally in a well-ventilated, dust-free location, as specified in Specification D 3924, and allow to dry for 40 to 48 h. Then repeat 7.5 to determine the Dry Hiding Index (h_{sd}).

7.7 Make drawdowns in triplicate and calculate the mean index value to 0.1 units. Individual values that deviate from the mean by more than 1.5 units should be discarded and the test repeated.

Calculation

8.1 Subtract the Wet Hiding Index, h_{sw} from the Dry

³ Available from The Leneta Company, P.O. Box 86, Ho-Ho-Kus, NJ 07423.

⁴ A white standard of this description is supplied with the TG19 Logicator Test Charts.

TABLE 2 Logicator Scale Relationships Calculated from Equations in Table 1

Index ^A		Clearance		Film Thickness ^C		Spreading Rate ^C		Index ^A		Clearance		Film Thickness ^C		Spreading Rate ^C						
<i>h</i>	mils ^B	μm	mils	μm	ft ² /gal	m ² /L	<i>h</i>	mils ^B	μm	mils	μm	ft ² /gal	m ² /L	<i>h</i>	mils ^B	μm	mils	μm	ft ² /gal	m ² /L
20	10.40	264	5.72	145	280	6.88	36	4.76	121	2.62	67	612	15.0							
21	9.90	252	5.44	138	294	7.23	37	4.54	115	2.50	63	643	15.8							
22	9.43	240	5.19	132	309	7.59	38	4.32	110	2.38	60	675	16.6							
23	8.98	228	4.94	126	325	7.97	39	4.12	105	2.26	58	709	17.4							
24	8.56	217	4.71	120	341	8.37	40	3.92	100	2.16	55	744	18.3							
25	8.15	207	4.48	114	358	8.78	41	3.73	95	2.05	52	781	19.2							
26	7.76	197	4.27	108	376	9.22	42	3.56	90	1.96	50	820	20.1							
27	7.39	188	4.07	103	395	9.68	43	3.38	86	1.86	47	861	21.1							
28	7.04	179	3.87	98	414	10.2	44	3.23	82	1.77	45	904	22.2							
29	6.70	170	3.69	94	435	10.7	45	3.07	78	1.69	43	950	23.3							
30	6.38	162	3.51	89	457	11.2	46	2.93	74	1.61	41	997	24.5							
31	6.08	154	3.34	85	480	11.8	47	2.79	71	1.53	39	1047	25.7							
32	5.79	147	3.19	81	504	12.4	48	2.65	67	1.46	37	1100	27.0							
33	5.52	140	3.03	77	529	13.0														
34	5.25	133	2.89	73	555	13.6														
35	5.00	127	2.75	70	583	14.3														

^A Boldface values refer to notches in TG19 Logicator.

^B Target values are significant to one decimal place.

^C Based on wet film thickness estimated at 55 % of clearance.

Hiding Index, h_{SD} , to obtain the wet-to-dry hiding change (WDHC), Δh_s , as follows:

$$WDHC = \Delta h_s = h_{SD} - h_{SW}$$

The value for WDHC is positive for an increase and negative for a decrease in hiding. To avoid possible misunderstanding write the sign of the change (+ or -) in every case.

8.2 If desired, calculate the corresponding percent change in wet-to-dry hiding power as follows:

$$WDHC\% = (1.05^{WDHC} - 1) \times 100$$

9. Report

9.1 Report the following information:

9.1.1 The Wet-to-Dry Hiding Change (WDHC) in logicator units (LU) to one decimal place, as described in 8.1, and

9.2 If desired, the percent change in conventional hiding power (WDHC%), calculated as shown in 8.2.

10. Precision and Bias

10.1 In an interlaboratory study of this test method, two operators in each of two laboratories and one operator in each of six laboratories, tested in triplicate five coatings with

a wide range in wet-to-dry hiding change. Since the test was not repeated, the repeatability data is only for replicates. On this basis the intralaboratory standard deviation was 0.77 LU with 85 df and the interlaboratory standard deviation was 1.97 LU with 39 df. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

10.1.1 *Repeatability (Replicate)*—Three replicates by the same operator should be considered suspect if they differ by more than 2.6 LU.

10.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by different operators should be considered suspect if they differ by more than 5.7 LU.

10.2 *Bias*—This test method has no bias because there is no criterion for the property it measures more valid than the test method itself.

NOTE 2—In the statistical analysis results were discarded on the following basis: (1) all results from one operator who appeared to be less sensitive than the other operators in detecting differences among triplicates, (2) one replicate result from one laboratory because the triplicate range was significantly higher than for other laboratories, and (3) all three results from the same laboratory for another paint, because the mean differed significantly from other means for that paint.

11. Keywords

11.1 hiding power; wet-to-dry hiding change

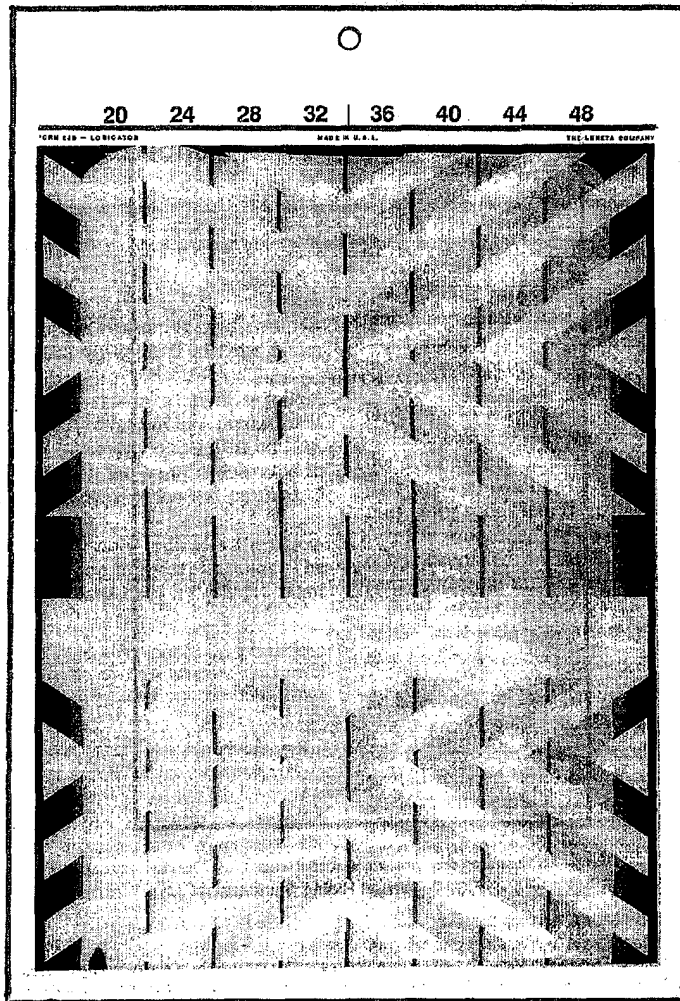
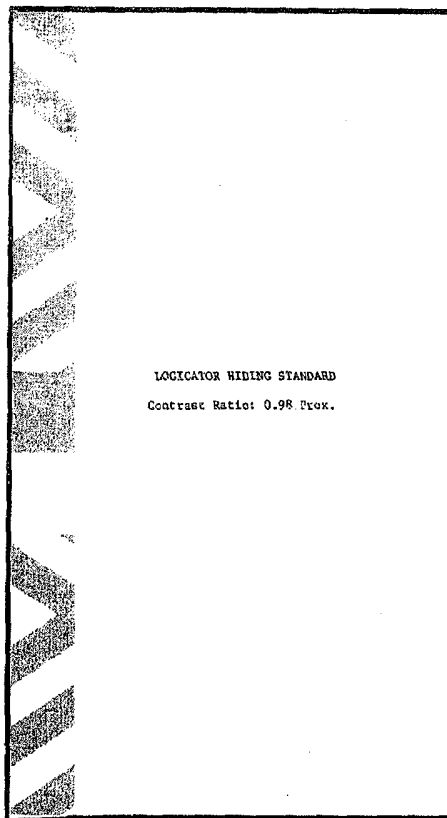


FIG. 2 Logicator Test Application



NOTE—For illustration only. Shading is not accurate.

FIG. 3 Visual Hiding Standard

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Evaluating and Comparing Transfer Efficiency Under Laboratory Conditions¹

This standard is issued under the fixed designation D 5009; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method covers the evaluation and comparison of the transfer efficiency of spray-applied coatings under controlled laboratory conditions.

1.2 This test method has been shown to yield excellent intralaboratory reproducibility. Interlaboratory precision is poorer and is highly dependent on closely controlled air flow in the spray booth, the rate at which the paint is delivered to the part, and other variables suggested in the test method.

1.3 Limitations:

1.3.1 This laboratory procedure only indicates the direction of the effect of spray variables on transfer efficiency. The magnitude of the effect is determined only by specific plant experience.

NOTE 1—This laboratory procedure requires specific equipment and procedures. For those laboratories that do not have access to the type of equipment required a more general laboratory procedure is being prepared as Procedure B.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 7 and Notes 2 and 3.*

2. Referenced Documents

2.1 ASTM Standards:

D 1200 Test Method for Viscosity by Ford Viscosity Cup²

D 2369 Test Method for Volatile Content of Coatings²

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²

2.2 National Fire Protection Association Documents³

NFPA 33 Spray Application Using Flammable and Combustible Materials

NFPA 86 Standard for Ovens and Furnaces

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *conveyor speed*—the speed of the conveyor in centimeters per minute during the test.

3.1.2 *fluid mass flow rate*—the mass flow rate of paint in grams per minute during the test.

3.1.3 *mass of foil*—the weight of each target foil in grams before being painted.

3.1.4 *mass of foil plus paint solids*—the weight of each target foil in grams after being painted and baked.

3.1.5 *mass of paint solids*—the difference in the mass of the foils before painting and the mass of the foils after painting and baking. This is the sum of the mass of the foil plus paint solids less the sum of the mass of the foil.

3.1.6 *transfer efficiency*—the ratio of the mass of the paint solids deposited on the foil to the mass of the paint solids sprayed during the test expressed as a percent.

3.1.7 *weight percent solids*—the solids content in percent of the total weight of a sample of the paint used during the test.

4. Summary of Test Method

4.1 Metal panels covered with preweighed aluminum foil are conveyed in a spraybooth past a fixed spraygun. The coated foils are then baked to remove volatile matter. The transfer efficiency is calculated on a weight basis using the solids content and quantity of the paint sprayed and the amount of solids on the coated aluminum foil target.

5. Significance and Use

5.1 Subject to the limitations listed above, the procedure can be used as a research tool to optimize spray equipment and paint formulations as well as to study the relative effect on transfer efficiency of changing operating variables, spray application equipment, and types of coatings.

6. Apparatus

6.1 *Laboratory Scale*, accurate to ± 0.001 g for weight percent solids determination.

6.2 *Platform Scale*, accurate, or equivalent, to ± 0.01 g for mass of foil, mass of foil plus paint, and mass flow rate instrumentation calibration.

6.3 *Mass Flow Rate Meter*, or mass flow rate determination method, accurate to $\pm 2\%$ of the mass flow rate to be used during the test.

6.4 *Conveyor Timer* or conveyor timing method, accurate to $\pm 1\%$ of the conveyor speed to be used during the test. The equipment may consist of photoelectric cells or limit switches used in conjunction with a digital timer or timing marks on the conveyor used in conjunction with a stopwatch. Take at least two readings with a stopwatch and average the readings.

6.5 *Targets*, consisting of a set of ten steel panels 6 in. (15.2 cm) wide by 0.0625 in. (0.15875 cm) with 0.25-in.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory-Applied Coatings on Preformed Products.

Current edition approved October 27, 1989. Published December 1989.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ Available from National Fire Protection Assn., Battery March Park, Quincy, MA 02269.

(0.635-cm) radius corners. A minimum panel length of 48 in. (121.9 cm) should be used. The length of the panel should be set so that a minimum of 12 in. (30.4 cm) above and below the spray pattern is achieved.

6.5.1 It is essential to do this so that the entire height of the spray pattern is effectively captured.

6.6 *Aluminum Foil*, medium temper or equivalent, 1.5 mil (0.0037 cm) thick.

6.7 *Back-Draw Water Wash Spray Booth*, or equivalent. The booth should be a minimum of 6 ft (1.8 m) wide and capable of up to 120 ft/min (0.61 m/s) air velocity in the middle at the plane of the target. If a dry filter booth is used, filters should be changed as necessary to maintain uniform air velocity.

6.8 *Adjustable Rate Overhead Conveyor System*, capable of hanging targets as specified, and capable of up to 40 ft/min (0.20 m/s) or the maximum speed desired by the user.

6.9 *Forced Draft Curing Oven*, of sufficient size for curing targets, and capable of achieving and maintaining the cure temperatures specified by the paint supplier. All ovens should conform to NFPA 86.

6.10 *Curing Rack*.

6.11 *Stopwatch*.

6.12 *Air Velocity Measurement Equipment*.

6.13 *Humidity and Temperature Measurement Equipment*.

6.14 *Compressed Air Supply*.

7. Hazards

7.1 For specific hazard information and guidance, consult the supplier's Material Safety Data Sheet (MSDS) for the materials used.

8. Procedure

8.1 Set up the spray apparatus paint supply and the mass flow measurement equipment in accordance with the manufacturer's instructions.

8.1.1 In accordance with Chapter 9-11 of NFPA 33, all electrically conductive objects in the spray area, except those objects required by the process to be at high voltage, shall be adequately grounded.

8.2 Agitate paint in a closed container at least 30 min before any paint samples are taken.

8.3 Using an airtight container take a paint grab sample from the paint pot in accordance with Practice D 3925.

8.4 Determine and record the following from the paint sample:

8.4.1 Viscosity determined in accordance with Test Method D 1200.

8.4.2 Weight percent solids determined in accordance with Test Method D 2369. If the baking temperature in Test Method D 2369 is inadequate, use the manufacturers recommended cure schedule.

8.4.3 Resistivity for the samples being applied electrostatically (An ASTM method is under development).

8.5 Set up the conveyor speed measuring equipment.

8.6 Cut the aluminum foil to dimensions of 15 in. (38 cm) by approximately 50 in. (127 cm) or 2 in. (5 cm) longer than the length of the target panel.

8.7 Consecutively number each precut foil strip before weighing using a permanent marking pen.

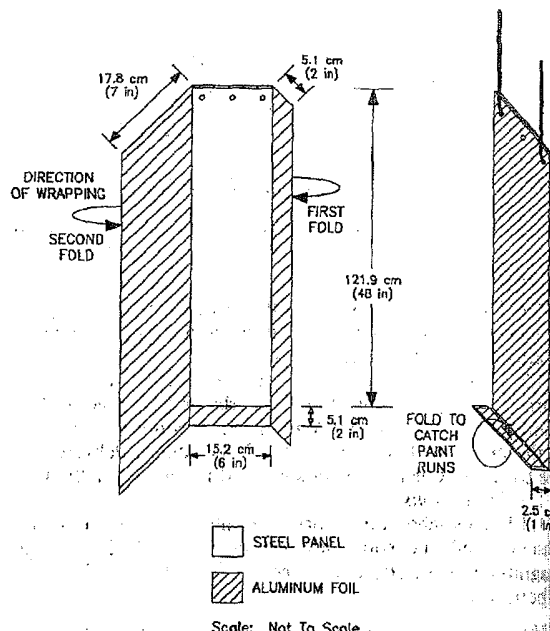


FIG. 1 Foil Attachment Technique

8.8 Weigh each test foil strip and record the uncoated weight and the foil number.

8.9 Attach the preweighed, labeled test foil to six targets using the technique shown in Fig. 1. Attach the unlabeled foil on four scavenger targets as shown in Fig. 1.

8.10 Mount the foil covered targets in consecutive order as shown in Fig. 2, with the foil seam on each target facing away from the spray gun.

8.10.1 If electrostatic equipment is being used the resistance shall be less than $1 \text{ by } 10^6 \Omega$ between the target and the earth ground in accordance with Chapter 9-8 of NFPA 33.

8.11 Adjust the following equipment operating parameters to the values desired for testing:

8.11.1 Paint fluid pressure (kilopascals) at spray gun.

8.11.2 Atomizing air pressure (kilopascals) at spray gun.

8.11.3 Rotating atomizer head speed (revolutions per minute) with and without paint fluid flow.

8.11.4 Operating voltage (kilovolts) if electrostatic equipment is used.

8.11.5 Ambient air temperature (degree Celsius).

8.11.6 Paint fluid temperature (degree Celsius).

8.11.7 Booth air velocity (feet per minute).

8.11.8 Relative humidity (percent).

8.11.9 Spray gun to target distance (centimeters).

NOTE 2: Precaution—If electrostatic equipment is being used, the gun-to-target distance shall be at least twice the sparking distance in accordance with Chapter 9-7 of NFPA 33.

8.11.10 Conveyor speed (centimeters per second).

8.11.11 Fluid mass flow rate (grams per minute).

8.11.12 Set the cure time and temperature in accordance with the manufacturer's instructions.

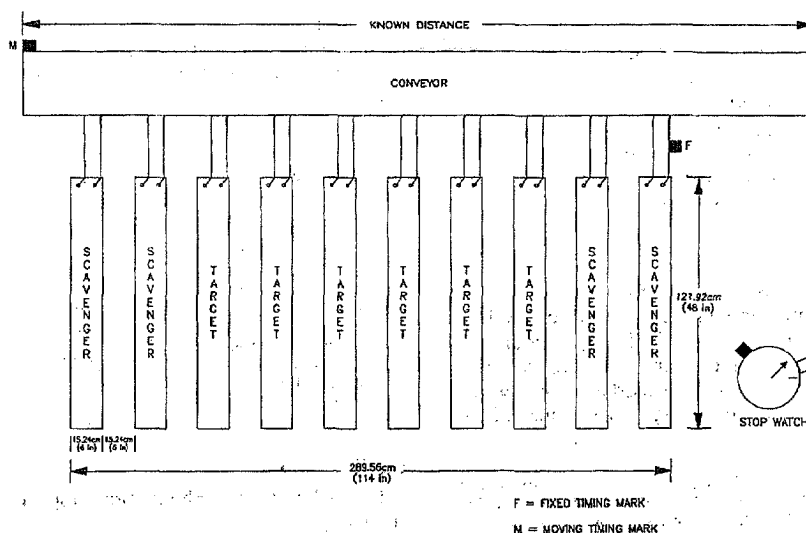


FIG. 2 Target Configuration for Transfer Efficiency Determination

8.12 For electrostatic spray equipment, measure the operating voltage and adjust it according to the manufacturer's instructions.

8.13 Turn on the spray booth and conveyor. At least 15 s before the first scavenger target passes in front of the gun, turn on the paint spray equipment. Maintain uniform paint flow during the test.

8.13.1 If mass flow measurement is used, begin the flow measurement at the leading edge of the first scavenger target and stop the mass flow measurement at the trailing edge of the last scavenger target.

8.13.2 If mass flow measurement equipment is not available use the following technique. Just before turning on the spray booth and the conveyor, spray the gun into a preweighed covered plastic container for a minimum of 30 s.

NOTE 3: Precaution—In addition to other precautions, turn off all high voltage to electrostatic spray guns to prevent personal injury.

8.13.3 Immediately weigh the container with paint, calculate the flow rate, and record the result. Just after turning off the spray booth and conveyor, repeat this procedure. Average the two results to obtain the average flow rate for the test.

NOTE 4—A gallon plastic bottle with the top cut off to conveniently fit around the front of the gun is recommended to use with the above procedure. A large plastic beaker covered with plastic wrap with a hole punched in the center of the plastic wrap works also.

Turn off all air sources to the spray gun before using the above procedure to prevent paint splattering out of the container.

8.14 Record the following data:

8.14.1 Application Equipment:

8.14.1.1 Paint fluid pressure (kilopascals) at the spray gun,

8.14.1.2 Atomizing air pressure (kilopascals) at the spray gun,

8.14.1.3 Rotating atomizer head speed (revolutions per minute) with and without paint fluid flow, and

8.14.1.4 Operating voltage (kilovolts) if electrostatic equipment is used.

8.14.2 Spray Booth and Conveyor:

8.14.2.1 Ambient temperature (degrees Celsius),

8.14.2.2 Paint fluid temperature (degrees Celsius),

8.14.2.3 Booth air velocity (foot per minute),

8.14.2.4 Relative humidity (percent),

8.14.2.5 Spray gun to target distance (centimeters),

8.14.2.6 Conveyor speed (centimeters per minute), and

8.14.2.7 Fluid mass flow rate (grams per minute).

8.15 After the paint flow and the conveyor are stopped, remove the painted targets from the conveyor and ensure that no paint is lost. Measure the wet film thickness to ensure that the proper amount of paint has been applied and record the wet film thickness.

8.16 Securely hang the coated targets on oven racks so all painted surfaces are exposed for uniform drying. Insert the racks into the oven and bake at the recommended manufacturer's cure schedule.

8.17 Remove the targets from the oven and let cool.

8.18 Remove the foil from each target, weigh and record the coated weight, the foil number, the percent vertical film coverage, and the dry film thickness at the center of the spray pattern.

8.19 The mass of the paint solids deposited is the difference in the total weight of the foils before painting and the total weight of the foils after painting and baking.

9. Calculation

9.1 Calculate the transfer efficiency using the following equation:

$$T = (100 \times C \times P) / (F \times S \times W)$$

where:

T = transfer efficiency, %

C = conveyor speed, cm/min,

P = mass of paint solids deposited, g,

F = fluid delivery rate, g/min,

S = weight percent solids expressed as a decimal, and

W = effective target width, 30.48 cm.

10. Report

10.1 Report the following information:

- 10.1.1 Transfer efficiency results,
- 10.1.2 Type of spray equipment,
- 10.1.3 Type of paint applied,
- 10.1.4 Paint application conditions, and
- 10.1.5 Conditions of test other than those specified in the procedure section of this test method.

11. Precision

11.1 This test method is derived from a study and report of transfer efficiency measurements conducted for the U. S. Environmental Protection Agency.⁴ The procedure described was subjected to round-robin evaluation following ASTM guidelines. The procedure was used at eight laboratories and results were obtained using conventional airless,

electrostatic air spray, and conventional air spray equipment. Statistical treatment of the results for the test sites and spray equipment type (gun) yields the following transfer efficiency results:

Type of Gun	Variance	Standard Deviation
<i>Conventional airless:</i>		
Within laboratory	1.22	1.10
Between laboratory	26.41	...
Gun	6.22	...
Total	33.85	5.82
<i>Electrostatic Air Spray:</i>		
Within laboratory	3.63	1.91
Between laboratory	72.02	...
Gun	13.01	...
Total	88.66	9.42
<i>Conventional Air Spray:</i>		
Within laboratory	2.30	1.50
Between laboratory	42.90	...
Gun	0.88	...
Total	46.08	6.79

12. Keywords

12.1 laboratory method; spray applied coatings; transfer efficiency

⁴ Development of Proposed Standard Test Method for Spray Painting Transfer Efficiency, Vols I and II, EPA Publication Nos. EPA-600/2-88-026a and EPA-600/2-88-026b, Environmental Protection Agency, Research Triangle Park, NC.

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Standard Guide for Testing Printing Inks and Related Materials¹

This standard is issued under the fixed designation D 5010; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers a list of test methods, practices, and specifications that can be used for the testing and evaluation of printing inks, printed ink films, and substrates used in their production (see Table 1).

1.2 This guide includes methods that were developed to test paints, paint films, and substrates, but may be adapted for use in testing printing inks and printed matter. Tests on raw materials and analytical methods in general have not been included.

NOTE 1—For the purpose of this guide, clear coatings such as overprint varnishes are classed as printing inks.

1.3 Other ASTM standards not specified here may also be applicable.

2. Terminology

2.1 Definitions:

2.1.1 *printing ink*—a colored or pigmented liquid or paste composition that dries to a solid film after application as a thin layer by printing machinery.

2.1.1.1 *Discussion*—Printing inks may contain vehicles, colorants, waxes, solvents, and other additives. Bulk inks are tested for dispersion, tinting strength, density, heat and storage stability, rheology, and printing properties.

2.1.2 *printed ink film*—thin layer of a printing ink deposited onto a substrate by means of a laboratory or production printing press, occasionally by a drawdown or roll-out technique. Printed matter is the usual medium by which inks are tested for appearance properties, drying, and resistance to various agents.

2.1.3 *printing substrate*—material onto which ink is deposited in the production of printed matter. Printing substrates include paper, paperboard, plastic film, glass, and metallic surfaces. In this guide, standards relating to substrates are largely restricted to properties associated with appearance and printability.

3. Test Categories

3.1 For convenience in selection, the test methods, practices, and specifications, listed in this guide are classified into three groups by type of printing process and in subgroups indicating whether the test is conducted on a bulk ink, a printed ink film, or a substrate (see Table 2). The group is given in the left column preceding the test method reference. The classification are as follows:

3.1.1 Group 1—Applicable in General:

Class A—Bulk inks.

Class B—Printed ink films.

Class C—Substrates.

3.1.2 Group 2—Applicable to Low Viscosity or Liquid Inks Associated With Flexography or Gravure:

Class A—Bulk inks.

Class B—Printed ink films.

Class C—Substrates.

3.1.3 Group 3—Applicable to High Viscosity or Paste Inks Associated With Letterpress, Lithography, or Silk Screen:

Class A—Bulk inks.

Class B—Printed ink films.

Class C—Substrates.

4. Precision and Bias

4.1 If available, precision for each test method listed can be found in the latest revision of that test method.

5. Keywords

5.1 printed matter; printing inks; printing substrates; test methods and practices (tabulation of)

¹ This guide is under the jurisdiction of the ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved May 15, 1991. Published July 1991.

TABLE 1 Numerical Listing of Ink-Related Standards

ASTM Designation	Volume	Title
D 16	06.01 06.02 06.03	Terminology Relating to Paint, Varnish, Lacquer, and Related Products
D 56	05.03 06.03	Test Method for Flash Point by Tag Closed Tester
D 93	04.09 05.01 06.03	Test Method for Flash Point by Pensky-Martin Closed Tester
D 185	06.01	Test Methods for Coarse Particles in Pigments, Pastes, and Paints
D 344	06.01	Test Method for Relative Dry Hiding Power of Paints by the Visual Evaluation of Brushouts
D 523	06.01	Test Method for Specular Gloss
D 528	15.09	Test Method for Machine Direction of Paper and Paperboard
D 562	06.01	Test Method for Consistency of Paints Using the Stormer Viscometer
D 644	15.09	Test Method for Moisture Content of Paper and Paperboard by Oven Drying
D 685	15.09	Method for Conditioning Paper and Paperboard Products for Testing
D 724	15.09	Test Method for Surface Wettability of Paper (Angle-of-Contact Method)
D 780	15.09	Test Method for Printing Ink Permeation of Paper (Castor Oil Test)
D 869	06.01	Test Method for Evaluating the Degree of Settling of Paint
D 918	15.09	Test Method for Blocking Resistance of Paper and Paperboard
D 971	05.01 10.03	Test Method for Interfacial Tension of Oil Against Water by the Ring Method
D 1200	06.01	Test Method for Viscosity by Ford Viscosity Cup
D 1210	06.01	Test Method for Fineness of Dispersion of Pigment-Vehicle Systems
D 1259	06.02	Test Methods for Nonvolatile Content of Resin Solutions
D 1310	05.01 06.03	Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus
D 1316	06.01	Test Method for Fineness of Grind of Printing Inks by the NPRI Grindometer
D 1331	15.04	Test Methods for Surface and Interfacial Tension of Solutions of Surface-Active Agents
D 1353	06.03	Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products
D 1474	06.01	Test Methods for Indentation Hardness of Organic Coatings
D 1475	06.01	Test Method for Density of Paint, Varnish, Lacquer, and Related Products
D 1535	06.01	Test Method for Specifying Color by the Munsell System
D 1544	06.01 06.02 06.03	Test Method for Color of Transparent Liquids (Gardner Color Scale)
D 1545	06.01	Test Method for Viscosity of Transparent Liquids by Bubble Time Method
D 1590	11.01	Test Methods for Surface Tension of Water and Waste Water
D 1640	06.01	Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature
D 1644	06.01	Test Methods for Nonvolatile Content of Varnishes
D 1647	06.01	Test Methods for Resistance of Dried Films of Varnishes to Water and Alkali
D 1653	06.01	Test Methods for Water Vapor Permeability of Organic Coating Films
D 1725	05.02	Test Method for Viscosity of Resin Solutions
D 1729	06.01	Practice for Visual Evaluation of Color Differences of Opaque Materials
D 1849	06.01	Test Method for Package Stability of Paint
D 1963	06.03	Test Method for Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials at 25/25°C
D 2066	06.01	Test Methods for Relative Tinting Strength of Printing Ink Dispersions
D 2091	06.01	Test Method for Print Resistance of Lacquers
D 2196	06.01	Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer
D 2243	06.01	Test Method for Freeze-Thaw Resistance of Water-Borne Coatings
D 2244	06.01	Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates
D 2248	06.01	Practice for Detergent Resistance of Organic Finishes
D 2337	06.01	Test Method for Freeze-Thaw Stability of Multicolor Lacquers
D 2369	06.01	Test Method for Volatile Content of Coatings
D 2482	15.09	Method for Wax Pick Test for Surface Strength of Paper
D 2574	06.01	Test Method for Resistance of Emulsion Paints in the Container to Attack by Microorganisms
D 2578	08.02	Test Method for Wetting Tension of Polyethylene and Polypropylene Films
D 2616	06.01 14.02	Test Method for Evaluation of Visual Color Difference with a Gray Scale
D 2620	06.01	Test Method for Light Stability of Clear Coatings
D 2794	06.01	Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)
D 2805	06.01	Test Method for Hiding Power of Paints by Reflectometry
D 3134	06.01	Practice for Establishing Color and Gloss Tolerances
D 3258	06.01	Test Method for Porosity of Paint Films
D 3278	06.03	Test Methods for Flash Point of Liquids by Setflash Closed-Cup Apparatus
D 3359	06.01	Test Methods for Measuring Adhesion by Tape Test
D 3363	06.01	Test Method for Film Hardness by Pencil Test
D 3424	06.01	Method of Evaluating the Lightfastness of Printed Matter
D 3732	06.01	Practice for Reporting Cure Times of Ultraviolet-Cured Coatings
D 3825	05.03	Test Method for Dynamic Surface Tension by the Fast Bubble Technique
D 3828	05.03	Test Method for Flash Point by Setflash Closed Tester
D 3924	06.01	Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquers, and Related Materials
D 3925	06.01	Practice for Sampling Liquid Paints and Related Pigmented Coatings
D 3928	06.01	Test Method for Evaluation of Gloss or Sheen Uniformity
D 3934	06.03	Test Method for Flash/No Flash Test—Equilibrium Method by a Closed-Cup Apparatus

TABLE 1 *Continued*

ASTM Designation	Volume	Title
D 3980	06.01	Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings
D 4017	06.01	Test Method for Water in Paints and Paint Materials by Karl Fischer Method
D 4040	06.01	Test Method for Viscosity of Printing Inks and Vehicles by the Falling-Rod Viscometer
D 4080	06.01	Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser
D 4086	06.01	Practice for Visual Evaluation of Metamerism
D 4141	06.01	Practice for Conducting Accelerated Outdoor Exposure Tests of Coatings
D 4144	06.01	Method for Estimating Package Stability of Coatings for Ultraviolet Curing
D 4212	06.01	Test Method for Viscosity by Dip-Type Viscosity Cups
D 4287	06.01	Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer
D 4302	06.01	Specification for Artists' Oil, Resin-Oil, and Alkyd Paints
D 4359	06.01	Test Method for Determining Whether a Material is a Liquid or a Solid
D 4361	06.01	Test Method for Apparent Tack of Printing Inks and Vehicles by the Inkometer
D 4366	06.01	Test Methods for Hardness of Organic Coatings by Pendulum Damping Tests
D 4449	06.01	Test Method for Visual Evaluation of Gloss Differences Between Surfaces of Similar Appearance
D 4459	08.03	Practice for Operating an Accelerated Lightfastness Xenon-Arc-Type (Water Cooled) Light-Exposure Apparatus for the Exposure of Plastics for Indoor Applications
D 4518	06.01	Test Methods for Measuring Static Friction of Coating Surfaces
D 4541	08.01	Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers
D 4674	08.03	Test Method for Accelerated Testing for Color Stability of Plastics Exposed to Indoor Fluorescent Light and Window-Filtered Daylight
D 4713	06.01	Test Methods for Nonvolatile Content of Printing Inks, Resin Solutions, and Vehicles
D 4758	06.02	Test Method for Nonvolatile Content of Latexes
D 4942	06.01	Test Methods for Water Pickup of Lithographic Printing Inks and Vehicles in a Laboratory Mixer
D 5039	15.09	Methods for Identification of Wire Side of Paper
D 5067	06.01	Specification for Artists' Watercolor Paints
D 5098	06.01	Specification for Artists' Acrylic Emulsion Paints
E 97	06.01	Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry
	14.02	
	15.09	
E 284	14.02	Definitions of Terms Relating to Appearance of Materials
E 308	14.02	Method for Computing the Colors of Objects by Using the CIE System
E 313	14.02	Test Method for Indexes of Whiteness and Yellowness of Near-White, Opaque Materials
E 429	14.02	Method for Measurement and Calculation of Reflecting Characteristics of Metallic Surfaces Using Integrating Sphere Instruments
E 430	06.01	Method for Measurement of Gloss of High-Gloss Surfaces by Goniophotometry
E 691	06.03	Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
	08.03	
	14.02	
E 805	14.02	Practice for Identification of Instrumental Methods of Color and Color-Difference Measurement of Materials
E 991	14.02	Practice for Color Measurement of Fluorescent Specimens
E 1331	14.02	Test Method for Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry
E 1347	14.02	Test Method for Color and Color Difference Measurement of Object-Color Specimens by Tristimulus (Filter) Colorimetry
E 1349	14.02	Test Method for Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry
F 34	15.09	Test Method for Liquid Extraction of Flexible Barrier Materials
F 149	15.09	Definitions of Terms Relating to Optical Character Recognition
F 151	15.09	Test Method for Residual Solvents in Flexible Barrier Materials
F 372	15.09	Test Method for Water Vapor Transmission of Flexible Barrier Materials Using an Infrared Detector Technique
F 413	15.09	Practice for Preparation of an Offset Duplicator for Use in Functional Testing of Lithographic Copy Products
F 425	15.09	Definitions of Terms Relating to Lithographic Copy Products
F 909	15.09	Definitions of Terms Relating to Printers
F 1125	15.09	Terminology of Image Quality in Impact Printing Systems
G 7	14.02	Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials
G 23	06.01	Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials
	07.02	
	08.03	
	14.02	
G 24	07.02	Practice for Conducting Exposures to Daylight Filtered Through Glass
	14.02	
G 26	06.01	Practice for Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials
	08.03	
	14.02	

TABLE 2 Index of Standards by Property

Group	Topic	ASTM Designation	ASTM Volume No.
Testing in General			
1ABC	Terminology Relating to Paint, Varnish, Lacquer, and Related Products	D 16	06.01 06.02 06.03
1C	Conditioning Paper and Paperboard Products for Testing	D 885	15.09
1ABC	Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials, Standard Environment for	D 3924	06.01
1A	Determining Whether a Material is a Liquid or a Solid	D 4359	06.01
1ABC	Interlaboratory Study to Determine the Precision of a Test Method	E 691	06.03 06.03 14.02 06.01
1A	Sampling Liquid Paints and Related Pigmented Coatings	D 3925	
Appearance Properties			
1ABC	Terminology Relating to Appearance of Materials	E 284	14.02
<i>Color and Reflectance</i>			
1ABC	Calculation of Color Differences From Instrumentally Measured Coordinates	D 2244	06.01
1ABC	Color and Color-Difference by Tristimulus (Filter) Colorimetry	E 1347	14.02
1ABC	Color of Fluorescent Specimens	E 991	14.02
1A	Color of Transparent Liquids (Gardner Color Scale)	D 1544	06.01 06.02 06.03
1ABC	Computing the Color of Objects by the CIE System	E 308	14.02
1ABC	Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry	E 97	06.01 14.02 15.09
1ABC	Establishing Color and Gloss Tolerances	D 3134	06.01
1ABC	Identification of Instrumental Methods of Color or Color-Difference Measurement of Materials	E 805	14.02
1ABC	Reflectance Factor and Color by Spectrophotometry Using Bidirectional Geometry	E 1349	14.02
1ABC	Reflectance Factor and Color by Spectrophotometry Using Hemispherical Geometry	E 1331	14.02
1C	Reflecting Characteristics of Metallic Surfaces Using Integrating-Sphere Instruments	E 429	14.02
1ABC	Specifying Color by the Munsell System	D 1535	06.01
1ABC	Visual Color Difference With a Gray Scale	D 2616	06.01
1ABC	Visual Evaluation of Color Differences of Opaque Materials	D 1729	06.01
1ABC	Visual Evaluation of Metamerism	D 4086	06.01
1C	Whiteness and Yellowness of Near-White Opaque Materials	E 313	14.02
<i>Gloss</i>			
1ABC	Gloss of High-Gloss Surfaces by Goniophotometry	E 430	06.01 14.02
1ABC	Specular Gloss (20°, 60°, 85°)	D 523	06.01
1ABC	Visual Evaluation of Gloss Differences Between Surfaces of Similar Appearance	D 4449	06.01
1ABC	Visual Evaluation of Gloss or Sheen Uniformity	D 3928	06.01
<i>Opacity and Strength</i>			
1B	Hiding Power of Paints by Reflectometry	D 2805	06.01
1B	Relative Hiding Power of Paints by the Visual Evaluation of Brushouts	D 344	06.01
3A	Relative Tinting Strength of Printing Ink Dispersions	D 2066	06.01
<i>Other Optical Properties</i>			
1B	Definitions of Terms Relating to Optical Character Recognition	F 149	15.09
1B	Terminology of Image Quality in Impact Printing Systems	F 1125	15.09
Chemical Resistance			
1B	Detergent Resistance of Organic Finishes	D 2248	06.01
1B	Resistance of Dried Films of Varnishes to Water and Alkali	D 1647	06.01
Density			
1A	Density of Paint, Varnish, Lacquer, and Related Products	D 1475	06.01
1A	Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials at 25/25°C	D 1963	06.03
Dispersion			
1A	Coarse Particles in Pigments, Pastes, and Paints	D 185	06.01
1A	Fineness of Dispersion of Pigment-Vehicle Systems	D 1210	06.01
1A	Fineness of Grind of Printing Inks by the NPIRI Grindometer	D 1316	06.01
Drying			
1B	Drying, Curing, or Film Formation of Organic Coatings at Room Temperature	D 1640	06.01
1B	Reporting Cure Times of Ultraviolet-Cured Coatings	D 3732	06.01

TABLE 2 Continued

Group	Topic	ASTM Designation	ASTM Volume No.
Heat Stability			
1A	Flash/No Flash Test—Equilibrium Method by a Closed-Cup Apparatus	D 3934	06.03
1A	Flash Point by Pensky-Martin Closed Tester	D 93	04.09 05.01 06.03
1A	Flash Point of Liquids by Setflash Closed-Cup Apparatus	D 3278	06.03
1A	Flash Point by Setflash Closed Tester	D 3828	05.03
1A	Flash Point by Tag Closed Tester	D 56	05.01 06.03
1C	Moisture Content of Paper and Paperboard by Oven Drying	D 644	15.09
1A	Flash Point and Fire Point by Tag Open-Cup Apparatus	D 1310	05.01 06.03
2A	Nonvolatile Content of Latexes	D 4758	06.02
1A	Nonvolatile Content in Printing Inks, Resin Solutions, and Vehicles	D 4713	06.01
1A	Nonvolatile Content of Resin Solutions	D 1259	06.02
1A	Nonvolatile Content of Varnishes	D 1644	06.01
1A	Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products	D 1353	06.03
1A	Volatile Content of Coatings	D 2369	06.01
1A	Volatile Organic Compound (VOC) Content of Paints and Related Coatings	D 3960	06.01
Light and Weather Fastness			
1B	Accelerated Outdoor Exposure Test of Coatings	D 4141	06.01
1B	Accelerated Testing for Color Stability of Plastics Exposed to Indoor Fluorescent Lighting and Window-Filtered Daylight	D 4674	08.03
1B	Atmospheric Environmental Exposure Testing of Nonmetallic Materials	G 7	14.02
1B	Light Stability of Clear Coatings	D 2820	06.01
1B	Lightfastness of Printed Matter	D 3424	06.01
1B	Natural Light Exposures Under Glass	G 24	07.02 14.02
1B	Operating an Accelerated Lightfastness Xenon-Arc-Type (Water Cooled) Light-Exposure Apparatus for the Exposure of Plastics for Indoor Applications	D 4459	08.03
1B	Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials	G 23	06.01 07.02 08.03 14.02
1B	Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials	G 26	06.01 08.03 14.02
1B	Specification for (Lightfastness of) Artists' Oil, Resin-Oil, and Alkyd Paints	D 4302	06.01
1B	Specification for (Lightfastness of) Artists' Watercolor Paints	D 5067	06.01
1B	Specification for (Lightfastness of) Artists' Acrylic Emulsion Paints	D 5098	06.01
Physical Strength and Resistance (Nonchemical)			
1B	Abrasion Resistance of Organic Coatings by Taber Abraser	D 4060	06.01
1B	Adhesion by Tape Test	D 3359	06.01
1B	Film Hardness (of Organic Coatings) by Pencil Test	D 3363	06.01
1B	Hardness of Organic Coatings by Pendulum Damping Test	D 4366	06.01
1B	Indentation Hardness of Organic Coatings	D 1474	06.01
1B	Print (Imprint) Resistance of Lacquers	D 2091	06.01
1B	Pull-off Strength of Coatings Using Portable Adhesion Tester	D 4541	06.01
1B	Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)	D 2794	06.01
1B	Static Friction of Coating Surfaces	D 4518	06.01
Porosity and Permeability			
1B	Porosity of Paint Films	D 3258	06.01
1B	Water Vapor Permeability of Organic Coating Films	D 1653	06.01
2C	Water Vapor Transmission Rate of Flexible Barrier Materials Using an Infrared Detection Technique	F 372	15.09
Printing Properties			
3A	Definitions of Terms Relating to Lithographic Copy Products	F 425	15.09
1B	Definitions of Terms Relating to Printers	F 909	15.09
1C	Blocking Resistance of Paper and Paperboard	D 918	15.09
1C	Machine Direction of Paper and Paperboard	D 528	15.09
3AC	Preparation of an Offset Duplicator for Use in Functional Testing of Lithographic Copy Products	F 413	15.09
1C	Printing Ink Permeation of Paper (Castor Oil Test)	D 780	15.09
3A	Water Pickup of Lithographic Printing Inks and Vehicles in a Laboratory Mixer	D 4942	06.01
3C	Wax Pick Test for Surface Strength of Paper	D 2482	15.09
1C	Wire Side of Paper	D 5039	15.09

TABLE 2 Continued

Group	Topic	ASTM Designation	ASTM Volume No.
Rheology			
3A	Apparent Tack of Printing Inks and Vehicles by the Inkometer	D 4361	06.01
1A	Consistency of Paints Using the Stormer Viscometer	D 562	06.01
1A	High-Shear Viscosity Using the ICI Cone/Plate Viscometer	D 4287	06.01
1A	Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer	D 2196	06.01
2A	Viscosity by Dip-Type Viscosity Cups	D 4212	06.01
1A	Viscosity by Ford Viscosity Cup	D 1200	06.01
3A	Viscosity of Printing Inks and Vehicles by the Falling-Rod Viscometer	D 4040	06.01
1A	Viscosity of Resin Solutions	D 1725	06.02
1A	Viscosity of Transparent Liquids by Bubble Time Method	D 1545	06.01
Storage Stability			
1A	Degree of Settling of Paint	D 869	06.01
2A	Freeze-Thaw Resistance of Water-Borne Paints	D 2243	06.01
1A	Freeze-Thaw Stability of Multicolor Lacquers	D 2337	06.01
1A	Package Stability of Coatings for Ultraviolet Curing	D 4144	06.01
1A	Package Stability of Paint	D 1849	06.01
2A	Resistance of Emulsion Paints in the Container to Attack by Microorganisms	D 2574	06.01
Surface Chemistry			
1A	Dynamic Surface Tension by the Fast Bubble Technique	D 3825	05.03
1A	Interfacial Tension of Oil Against Water by the Ring Method	D 971	05.01
1A	Surface and Interfacial Tension of Solutions of Surface-Active Agents	D 1331	15.04
1A	Surface Tension of Water and Waste Water	D 1590	11.01
1C	Surface Wettability of Paper (Angle-of-Contact Method)	D 724	15.09
2C	Wetting Tension of Polyethylene and Polypropylene Films	D 2578	08.02
Special Analytical Tests			
2C	Liquid Extraction of Flexible Barrier Materials	F 34	15.09
2C	Residual Solvents in Flexible Barrier Materials	F 151	15.09
1A	Water in Paints and Paint Materials by Karl Fischer Method	D 4017	06.01

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Conducting Tests on Paints and Related Coatings and Materials Using Enclosed Carbon-Arc Light and Water Exposure Apparatus¹

This standard is issued under the fixed designation D 5031; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This practice covers accelerated exposure testing of paint, varnish, lacquer, and related products. The apparatus types available are single enclosed carbon arcs (Types H and EH) and twin enclosed carbon arcs (types D and DH). Both types are manufactured with or without automatic humidity control.² Table 1 describes commonly used test conditions. Interlaboratory comparisons *must* only be made using the same device type and test conditions.

1.2 The procedures described in this practice were previously included in Practice D 822, which covered the use of both filtered open flame and enclosed carbon arcs for testing paints, varnishes, lacquers, and related products.

NOTE 1—Another procedure for exposing these products is covered by Practice D 3361, in which the specimens are subjected to radiation from an unfiltered open-flame carbon arc that produces much higher levels of short wavelength radiation than filtered open-flame or enclosed carbon arcs. Only automatic humidity controlled open-flame carbon-arc apparatus (Type EH) is applicable to Practice D 3361.

1.3 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 4.

Referenced Documents

2.1 ASTM Standards:

- D 358 Specification for Wood to Be Used as Panels in Weathering Tests of Coatings³
- D 523 Test Method for Specular Gloss³
- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products³
- D 610 Test Methods for Evaluating Degree of Rusting on Painted Steel Surfaces³
- D 659 Method of Evaluating Degree of Chalking of Exterior Paints³
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints³

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D1.27 on Accelerated Testing.

Current edition approved Dec. 29, 1989. Published April 1990.

² Apparatus and carbon arcs manufactured by Atlas Electric Devices Company, 14 N. Ravenswood Avenue, Chicago, IL 60613 have been found satisfactory for this purpose.

³ Annual Book of ASTM Standards, Vol 06.01.

- D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints³
- D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints³
- D 714 Test Method for Evaluating Degree of Blistering of Paints³
- D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints³
- D 822 Practice for Conducting Tests on Paint and Related Coatings and Materials using Filtered Open-Flame Carbon-Arc Light and Water Exposure Apparatus³
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³
- D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers³
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base³
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base³
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁴
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting⁵
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates³
- D 2616 Test Method for Evaluation of Visual Color Difference with a Gray Scale⁴
- D 3361 Practice for Operating Light- and Water-Exposure Apparatus (Unfiltered Open-Flame Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products Using the Dew Cycle³
- D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films³
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁶
- G 23 Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials⁶

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Annual Book of ASTM Standards, Vols 02.05 and 06.01.

⁶ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

TABLE 1 Cycles Commonly Used for Testing Paints, Varnishes, Lacquers, and Related Coatings in Enclosed Carbon-Arc Devices

Cycle Description	Black Panel Temp. ^A °F (°C)	Typical Uses
102 min light	145 ± 5	general coatings ^B
18 min light and water spray	(63 ± 2.5)	
18 h using:		general coatings
102 min light	145 ± 5	
18 min light and water spray	(63 ± 2.5)	
6 h at 95 ± 4 % relative humidity with no water spray	75 ± 3	original equipment manufactured coatings
48 min light	145 ± 5	
12 min light and water spray	(63 ± 2.5)	exterior pigmented paints
4 h light	145 ± 5	
4 h water spray	(63 ± 2.5)	exterior wood stains and clears
12 h light	145 ± 5	
12 h water spray	(63 ± 2.5)	marine enamels
8 h light	145 ± 5	
10 h light and water spray	(63 ± 2.5)	
6 h water spray		

^A Unless otherwise indicated, black panel temperature during light only portion of the cycle.

^B Historical convention has established this as a very commonly used test cycle.

3. Significance and Use

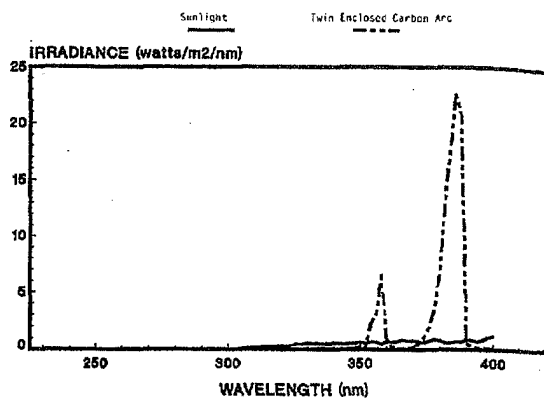
3.1 This practice is intended to evaluate coating films for their stability to ultraviolet light and moisture. If the spectral power distribution of the light source used for exposure tests does not adequately simulate that for terrestrial solar radiation, it may produce a different type of degradation and distort the ranking of materials obtained in outdoor exposures. Figures 1 and 2 compare representative spectral power distributions of a twin enclosed carbon arc with that of terrestrial sunlight.

3.2 No single operating procedure for light-exposure apparatus with or without water can be specified as a direct simulation of natural exposure. This practice does not express, or imply, a specific correlation with outdoor exposure.

3.3 Since natural environments vary with respect to climate, geography, and topography, it may be expected that the effects of natural exposures will vary accordingly. Furthermore, all materials are not affected equally by the same environment. Therefore, results obtained by use of this practice should not be represented as equivalent to those of any natural weathering test until the degree of correlation has been empirically established for the material in question.

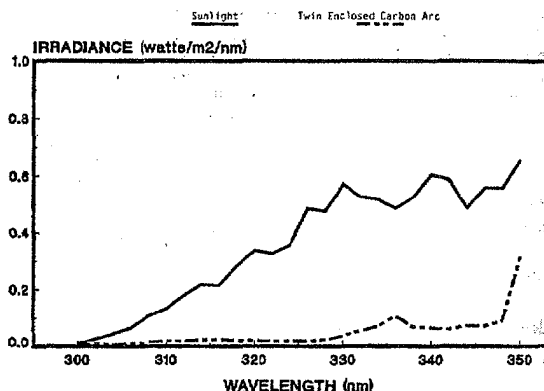
3.4 Variations in results are possible between instruments of the same type operating within the accepted limits of this practice. It is recommended that results obtained using this practice be compared with those for a control or reference material that is mutually agreed upon by the interested parties.

3.5 All references to exposures in accordance with this practice must include a complete description of the test cycle used in addition to the type of device used.



NOTE—The enclosed carbon-arc irradiance was measured at the sample plane at a position centered between the two carbon arcs. Sunlight was measured in Phoenix, AZ, at the summer solstice with clear sky at solar noon using a double grating monochromator (1-nm bandpass) with a quartz cosine receptor on an equatorial follow-the-sun mount. Because of momentary fluctuations in intensity due to flickering of the carbon-arc flame, the spectral power distribution shown in this figure is relative and is not to be used to calculate or estimate total radiant exposure for tests in enclosed carbon-arc devices.

FIG. 1 Representative Spectral Power Distributions (250–400 nm) for Twin Enclosed Carbon Arcs and Terrestrial Sunlight



NOTE—Measurements were made as described in Fig. 1. Because of momentary fluctuations in intensity due to flickering of the carbon-arc flame, the spectral power distribution shown in this figure is representative and is not meant to be used to calculate or estimate total radiant exposure for tests in enclosed carbon-arc devices.

FIG. 2 Representative Spectral Power Distributions (300–350 nm) for Twin Enclosed Carbon Arcs and Terrestrial Sunlight

4. Hazards

4.1 **Precaution**—In addition to other precautions, never look directly at the carbon arc because ultraviolet radiation can damage the eye. Most carbon-arc machines are equipped with door safety switches, but users of old equipment must be certain to turn the OPERATE switch OFF before opening the test-chamber door.

4.2 The burning carbon rods used in these devices become very hot during use. Make sure to allow at least 15 min for the arcs to cool after the device is turned off before

attempting to change the carbon rods. Avoid inhaling ash dust when changing carbon rods.

5. Test Specimens

5.1 Apply the coating to flat (plane) panels with the substrate, method of preparation, method of application, coating system, film thickness, and method of drying consistent with the anticipated end use, or as mutually agreed upon between the producer and user.

5.2 Panel specifications and methods of preparation include but are not limited to Methods D 609, Specification D 358, or Practices D 1730. Select panel sizes suitable for use with the exposure apparatus.

5.3 Coat test panels in accordance with Test Methods D 823 and measure the film thickness in accordance with an appropriate procedure selected from Test Methods D 1005, D 1186, or D 1400. Nondestructive methods are preferred because panels so measured need not be repaired.

5.4 Prior to exposing coated panels in the apparatus, condition them at $73 \pm 3^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity for one of the following periods in accordance with the type of coating:

Baked coatings	24 h
Radiation-cured coatings	24 h
All other coatings	7 days

NOTE 2—The procedures and specifications described in 5.2 through 5.4 are recommended but others may be used if agreed upon by all interested parties.

6. Procedure

6.1 Mount the test specimens both above and below a horizontal plane at the center of the single arc or centered between the two enclosed carbon arcs. When the exposure interval does not exceed 24 h, locate each specimen equidistant from the horizontal plane at the center of the single arc or centered between the arcs.

6.2 To ensure uniform exposure conditions for all specimens, reposition them vertically within their holders in a sequence that will provide each specimen with equivalent exposure periods in each location. For exposure intervals not exceeding 100 h, reposition specimens daily. For longer exposures, reposition specimens weekly. Other methods of achieving uniform total irradiation may be employed if mutually agreed upon between all concerned parties.

6.3 Table 1 lists test-cycle conditions commonly used for evaluation of paints, varnishes, lacquers, and related coatings.

6.4 It is recommended that the temperature of the water used for specimen spray be $60 \pm 9^\circ\text{F}$ ($16 \pm 5^\circ\text{C}$). Water used

for specimen spray must meet the purity levels specified in Practice G 23 in order to avoid unrealistic water spotting.

6.5 When mutually agreed upon, cycles other than those listed in Table 1 may be used. The term *cycle* is defined as the set of exposure conditions (light, light plus water spray, dark periods) that are repeated.

7. Periods of Exposure

7.1 Use one of the following methods to determine the duration of the exposure under this practice:

7.1.1 A mutually agreed upon specified number of total hours.

7.1.2 The number of total hours of exposure required to produce a mutually agreed upon amount of change in either the test specimen or an agreed upon control or reference material.

8. Evaluation of Specimens After Exposure

8.1 Evaluate or rate changes in exposed test specimens in accordance with Test Methods D 523, D 610, D 659, D 660, D 661, D 662, D 714, D 772, D 2244, D 2616, D 4214, E 97, and Practice D 1729. Consider product use requirements when selecting appropriate methods.

8.2 Evaluate test specimens by ranking their performance relative to a control or reference material exposed at the same time.

8.3 Plot properties of test specimens and controls as a function of exposure time and compare rate of change with that of the control or reference material. When this method of evaluation is used, the control or reference material must be exposed at the same time and in the same device as the test specimens.

8.4 Other methods for evaluating test specimens may be used if mutually agreed on by all interested parties.

9. Report

9.1 Report the following information:

9.1.1 Complete description of exposure procedure used, including:

9.1.1.1 Light/light plus water/dark cycle used, type of device used (single or twin enclosed carbon arc),

9.1.1.2 Operating black panel temperature during all portions of exposure cycle,

9.1.1.3 Operating relative humidity, and

9.1.1.4 Temperature of water used for water spray.

9.1.2 Total hours of test.

9.1.3 Test specimen preparation.

9.1.4 Identification of controls used.

9.1.5 Results of evaluation test or tests performed on specimens and control or standard samples.

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Standard Test Methods for Field Identification of Coatings¹

This standard is issued under the fixed designation D 5043; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover procedures and portable apparatus for determining the generic type of coating films most likely to be encountered on structures. The coating can either be weathered from exposure or be freshly applied.

1.2 Most commonly used coatings can be divided into the broad categories and subgroups shown in Table 1 on the basis of the nonvolatile component (generic types) of their vehicle (film forming resin, binder). Although the curing of some coatings involves more than one process and coatings may contain more than one type of resin, they can usually be assigned to one of the basic classes and generic types listed in Table 1.

1.3 For field exposed coatings, it is suggested that these test methods be used as part of a complete evaluation of a coated surface as it is frequently helpful to consider the environment of exposure and how the coating has performed in the environment when drawing conclusions from these tests.

1.4 These test methods will not result in the identification of components of a coating beyond general classification of the coating by generic type and are not appropriate if more detailed analysis is required, for example, as a part of failure analysis or to identify between different manufacturers of the same type of coating. They also may not be definitive enough to identify complex systems that include multiple layers of different generic types of coatings.

1.5 The evaluation of results is quite subjective. Practice and experience are required to minimize misinterpretation. Repeat tests may be required.

1.6 None of the tests is to be taken alone as grounds for identifying the generic type. Only the combination of results from several or all of the tests are to be used in conclusions regarding generic types.

1.7 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Notes 8, 9, and 11.

2. Summary of Test Methods

2.1 Samples of coatings films are tested with solvents and chemicals and subjected to pyrolysis to provide evidence of their generic type. Figure 1 shows a flow chart for suggested order of tests and classification of results.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

Current edition approved April 27, 1990. Published June 1990.

3. Significance and Use

3.1 Information about the generic type of coating on a surface is required to select compatible coatings for repainting and can be used when evaluating the performance of a coating in an environment in decisions on upgrading or replacing a coating system. These test methods provide information on generic type, can be performed in the field by personnel with limited laboratory experience, and require a minimum of equipment and materials.

4. Sampling

4.1 The sample of coating can be obtained by chipping or scraping with a knife or by sanding and then brushing the material into a specimen container or clean envelope. Care should be taken not to cut into substrates, such as plastic or asphalt, that contain polymeric or bituminous materials. Small portions of untreated wood, masonry, or steel do not ordinarily interfere with the tests. Some tests can be conducted directly on the coating surface. If a liquid sample of coating is to be evaluated, a film of the coating should first be cast on a glass plate or similar surface from which it can conveniently be removed after drying.

NOTE 1—To develop familiarity with the subjective evaluations that follow, it is good practice to make films of known resin composition by applying control paints to glass plates or similar surfaces from which they can be readily removed after drying.

5. Pyrolysis

5.1 *Summary of Test Method*—A sample of coating placed in a small test tube is burned over a hot flame. The way the coating burns and the odor and other characteristics of the fume generated are recorded. The Beilstein test identifies the presence of chlorinated and other halogens. In coatings, chlorine-containing material is most often encountered. For coatings not containing halogens, the odor is recorded.

5.2 Apparatus:

5.2.1 *Flame Source*, including butane or propane utility torch. (Lighters do not provide a hot enough flame.)

5.2.2 *Test Tubes*—A suitable size is 10 by 75 mm (disposable culture tubes).

5.2.3 *Copper Wire*—A length of single-strand 16 to 18 gauge. AWG copper electrical wire, stripped of insulation sufficiently far that melted insulation cannot interfere with the test, is satisfactory. Leave about 6 in. of insulation as a heat insulator or provide a wrapping or handle for protection from heat.

5.2.4 *Lead Acetate Paper*.

5.2.5 *Test Tube Clamp*.

5.3 Procedure:

5.3.1 Put a small specimen of coating, preferably of one

TABLE 1 Classification of Coatings Frequently Used

Basic Class	Examples
Drying or baking (oxidizing) paint and enamel	unmodified drying oil oleoresinous (oil-modified, alkyd, epoxy ester, phenolic and other resins)
Lacquer (drying by solvent evaporation)	vinyl (poly(vinyl chloride-vinyl acetate)) poly(vinyl butyral) chlorinated rubber styrene-butadiene rubber and similar rubbers bituminous (coal tar, asphalt) cellulose nitrate poly(vinyl acetate) acrylic styrenated acrylic epoxy
Glass (drying by evaporation of water)	
Chemically curing single package and multi-component coating	bituminous epoxy urethane polyester silicates and cement flame-sprayed silicones
Inorganic	
Cellulosic	

piece, in the test tube and hold the tube briefly in the hot flame. Limit flame contact to the end of the test tube immediately around the specimen. As the specimen begins to burn, observe the nature of deterioration and identify coating type as follows:

Observation	Identification
Change in shape; possible change in color; continued heating causes sample to glow red; rapid deterioration, almost explosive in nature; charring	inorganic cellulose nitrate, or similar some vinyl-type coatings

NOTE 2—Melting, bubbling, and charring are common with most generic types and not definitive.

5.3.2 Continue heating until fume (smoke) fills the test tube. Most fumes are white or near-white; slight condensation of a clear liquid on the upper test tube wall is sometimes observed. Other observations and identifications include:

Observation	Identification
Dark fume; clear brown liquid condensate	possibly epoxy
Very dark, possibly sooty fume; dark condensate	bituminous

NOTE 3—Bituminous coatings may be asphalt, coal tar, or combinations. The test is not definitive.

NOTE 4—Silicone coatings will form an ash upon pyrolysis at 800°C. Such temperatures are outside the scope of this test.

5.3.3 *Beilstein Test*—Conduct the Beilstein test by first heating the bare copper wire in the flame until no color is imparted to the flame. Insert the heated wire into hot fume in the test tube briefly (1 to 2 s). Withdraw the copper wire from the test tube and immediately hold it in the flame again. Observe the flame over the copper wire for color and make identifications as follows:

Observation	Identification
No color	no chlorine or chloride (or other halogen) content
Traces of green color	chloride contaminants from environment or minor component of coating
Strong green color	chlorinated resin or chlorinated resin modifier

With practice, the intensity of the green flame can be used to determine whether the chlorine containing component is major or minor.

5.3.3.1 *Example 1*—A very intense, relatively long-lasting repeatable green flame indicates chlorinated rubber or vinyl coating.

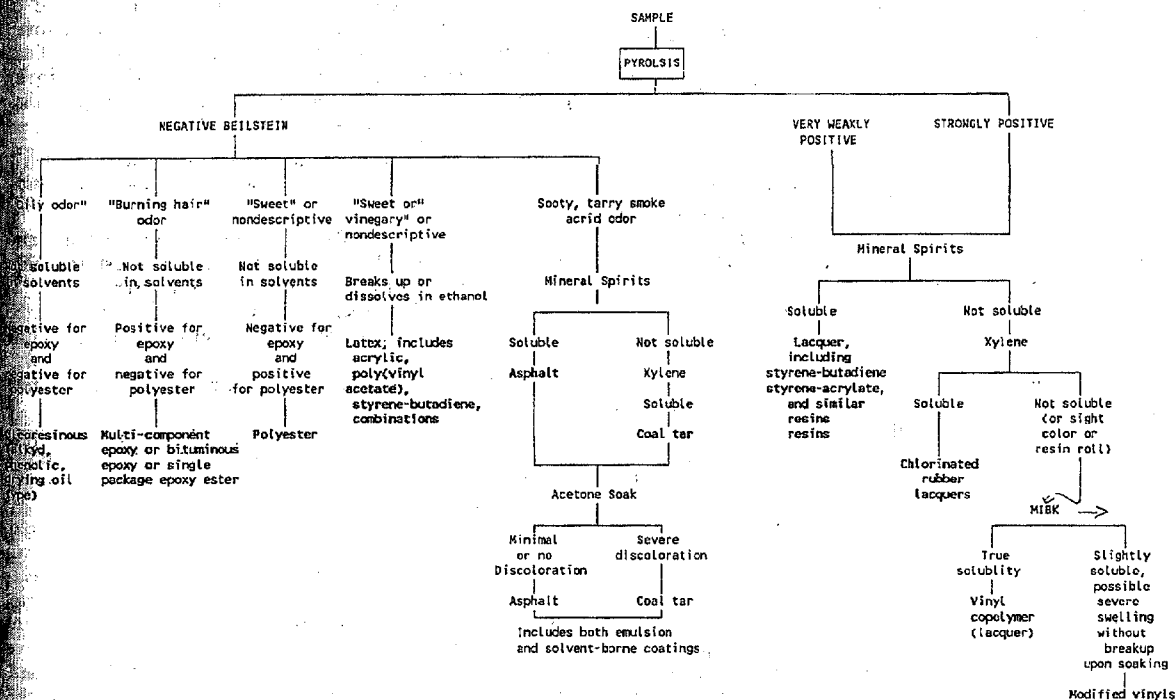


FIG. 1 Suggested Test Flow Chart for Coatings Identification

5.3.3.2 *Example 2*—An intense or moderately intense relatively short-lived green flame, which may or may not be repeated, indicates chlorinated plasticizer in a nonchlorinated resin binder.

NOTE 5—Although fluorinated resins also give a positive Beilstein test, they are less likely to be encountered in industrial applications than chlorinated resins.

NOTE 6—If the sample includes hydrated material, for example, concrete or plaster, water will be liberated by burning and will condense on the wall of the test tube. Halogen liberated from the paint will be absorbed into the condensate. The copper wire must be brought into contact with the condensate to avoid a false negative.

NOTE 7—Those experienced with the Beilstein test may prefer to run it on a specimen not subjected to pyrolysis.

5.3.4 *Odor Test*—Conduct the odor test *only* if the Beilstein test is negative (no green flame).

NOTE 8: *Warning*—Hot chlorine or fluorine-containing fumes are extremely irritating and potentially hazardous. Tip the test tube so that the fumes flow toward the open end of the tube. Gently wave a hand over the mouth of the test tube and carefully smell the odor of the fumes as they dissipate from the mouth of the test tube. Avoid overexposure to the fumes.

5.3.4.1 Indications are subjective, but the following classes can be assigned:

Observation	Identification
Oily	oleoresinous
Very sweet	acrylic latex
Vinegary; acetic acid	poly(vinyl acetate)
Burning hair	epoxy, epoxy ester, bituminous epoxy
Burning rubber	polysulfide
No strong odor	inorganic, cementitious
Acrid (biting) odor, with sooty or tarry smoke	bituminous

5.3.5 Use the lead acetate paper to verify the presence of a sulfide component by holding a piece of moistened lead acetate paper over or in the mouth of the test tube. A sulfur-containing component is present if the paper rapidly darkens.

6. Solubility Tests

6.1 Chemically cured, inorganic, and aged oleoresinous coatings are not resolvable in the solvents originally used in producing the coatings. Lacquers and some latex coatings are resolvable and the strength of the solvent required to cause the coating to dissolve can be used to classify the coating.

6.2 *Reagents*—Solvents used, listed in order of increasing power of solvency (that is, ability to dissolve a resin), are as follows:

6.2.1 *Denatured Ethanol (Ethyl Alcohol)*:

6.2.2 *Mineral Spirits (Petroleum Spirits)*—Aliphatic hydrocarbon solvent with typical Kauri-Butanol value (KB) of 25 to 45.

6.2.3 *Xylene (Xylol)*—Aromatic hydrocarbon solvent with typical KB of 98.

6.2.4 *Methyl Isobutyl Ketone (MIBK, 4-Methyl-2-Pentanone)*.

6.2.5 *Acetone (Dimethyl Ketone, 2-Propanone)*.

6.3 *Procedure*:

6.3.1 Although a stirring rod or a gloved finger-tip can be used, the test is best done by a finger-rub technique on the coating film itself. The sensations perceptible by touch are valuable in interpreting results. Alternatively, the solubility tests may be done by soaking portions of the film in solvents,

in which case porcelain spot plates and glass stirring rods can be used.

NOTE 9: *Warning*—These solvents can cause skin irritation and dermatitis. Minimize time of contact of solvents with skin and discontinue use if irritation occurs.

6.3.2 *Finger-Rub Test*—Beginning with ethanol, dampen a fingertip and rub the surface of the film briskly in a circular motion 5 to 10 mm (1/4 to 1/2 in.) in diameter. Renew the test solvent frequently as required. Continue rubbing at least 30 s or until definite effects are observed. Continue with each solvent in increasing power of solvency by cleaning the fingertip in each succeeding solvent before using that test solvent and selecting a new spot on the film or an untested chip of paint for each solvent used.

NOTE 10—If the coating film on the test surface is chalky, the first finger-rub test done with ethanol will liberate much of the chalk, which will dry quickly on the finger as a powder. Repeating the test will reveal much less or no color and the test surface will appear unchanged. If chalk is liberated, use ethanol to clean the test spot for subsequent solvent tests.

6.3.3 *Solvent-Soak Test*—The full series of solvents can be run concurrently. Place five chips in a spot plate dish and pour a small amount of each solvent over one of the chips. Periodically stir the solvent and rub the chip with a stirring rod until definite changes occur. Add additional solvent, if necessary due to evaporation, and observe extent of discoloration of the solvent and whether the chip softens, breaks apart, swells, dissolves, or a combination thereof. Note whether a color different from the color of the topcoat is imparted to the solvent, indicating dissolution of an intermediate or primer coat. If portions of the chip dissolve or discolor the solvent, soak up the solvent with a paper towel and add fresh solvent if undissolved chip remains. If no further effect occurs, wash the remainder of the chip by gentle swirling, soak up the solvent, allow the chip to dry, and proceed with pyrolysis or other tests.

NOTE 11: *Precaution*—Do not attempt to burn a solvent-wet chip or heat solvent in a test tube, as the liquid may suddenly boil, possibly causing burns or loss of specimen.

6.3.4 Observe the effects of rubbing or soaking and classify as follows (the results for the soaking test are more difficult to interpret than for the finger-rub test):

6.3.4.1 No effect or small amount of color transfer to the fingertip or rod (due to chalk or film surface abrasion while rubbing).

6.3.4.2 Softening of the film, with resin rolling into small balls under the fingertip or rod.

6.3.4.3 True solubility with film dissolving, becoming sticky, and transferring in relatively large liquified quantity to the fingertip or rod.

6.3.5 Succeeding layers in a coating system may be individually tested if they are visually different; for example, colored topcoat, white or grey intermediate coat, and brown or red primer coat. To test visually different layers, repeatedly rub the spot with an effective solvent and wipe away dissolved coating periodically until a sublayer of different color is clean, then continue the test with the effective solvent on the sublayer. If that solvent is ineffective, repeat the test on the same spot with the next stronger solvent in the series. If that solvent is effective, repeat the test on a new

men or area, and when the sublayer is uncovered, allow solvent to evaporate, then test the coating with the next solvent in the series, continuing up the series until solubility is again observed. If a sublayer of coating is not affected by any solvent in the series, that layer may be removed and collected by chipping or scraping for a solubility test.

2. Interpretation of Results:

2.1 **No Effect with Any Solvent**—Chemically cured, aged (aged) oleoresinous, or inorganic.

NOTE 12—With prolonged contact, oleoresinous coatings may soften or wrinkle. Absence of wrinkling, however, is not indicative of absence of oleoresinous coatings.

2.2 **Breaks Up or Dissolves in Ethanol**—Latex coating poly(vinyl butyral).

NOTE 13—Latex coatings are normally not soluble in mineral spirits but are only slightly affected in the short time of the test by xylene and MIBK (resulting in surface slickness). Ethanol will not dissolve any common coating type in this test, but it may affect considerable color transfer from weathered epoxy films. Bituminous emulsions do not break up or dissolve in ethanol.

2.3 **Dissolves in Mineral Spirits**—Asphalt coatings and other coatings other than chlorinated rubber and vinyls. Acceptable resins include styrene-butadiene, styrene-acrylate, and similar resins, but do not include polymers such as neoprene which are not normally used in solvent resistant coatings.

NOTE 14—Lacquer coatings other than chlorinated rubber and vinyls contain chlorinated plasticizers that give a positive Beilstein test.

NOTE 15—Some coal tar coatings strongly discolor mineral spirits but are not significantly dissolved by the solvent.

2.4 **Dissolves in Xylene but Not in Mineral Spirits**—Coal tar coatings and chlorinated rubber-based coatings. Frequently associated with solution of a chlorinated rubber is the ability of the resin to "string" between surface of film and finger when the finger is pulled away from the coating.

2.5 **Dissolves in MIBK but Not in Xylene**—Poly(vinyl chloride-vinyl acetate) solution coatings.

NOTE 16—Vinyls may soften with resultant "resin roll" in the finger-rub test with xylene. Some vinyls, modified with polymers such as ethylene, may not readily dissolve in MIBK, but feel slick with little color transfer in the finger-rub test and may swell up to 2½ times without dissolving in the MIBK soak test. They may also feel similarly with some color transfer in xylene.

NOTE 17—Latex binders merely soften in the short time the test is

2.6 It may be possible to further differentiate between asphalt and coal tar coatings using acetone. Soak a chip of bituminous coating in a test tube of acetone for several minutes. Agitate gently and observe for extent of discoloration. Asphalts only very slightly discolor acetone while coal tars strongly discolor it, but there can be intermediate degrees of discoloration that do not permit discrimination. It is not possible by simple methods to differentiate between a solvent-borne (cutback) and water-borne (emulsion) bituminous coating.

Test for Polyester Coatings

1 This test identifies polyester-based coatings from the

group of chemically cured coatings that are not affected by the solubility test.

7.2 Apparatus:

7.2.1 Test Tube.

7.2.2 Medicine Droppers.

7.3 Reagents:

7.3.1 Potassium Hydroxide in Methanol.

7.3.2 Hydroxylamine Hydrochloride Solution in Methanol, 10 %.

7.3.3 Ferric Chloride Solution, saturated in distilled water.

7.3.4 Hydrochloric Acid, 3 %.

7.3.5 Warm Water, 125°F (50°C).

7.4 **Procedure**—Place a small quantity of the film sample in a test tube. Add 10 drops of potassium hydroxide solution and 6 drops of hydroxylamine hydrochloride solution. Place the test tube in a container of warm water for 2 min. Add 10 drops of hydrochloric acid and one drop of ferric chloride solution.

7.5 Interpretation of Results:

Observation	Identification
Muddy violet color	dicastic polyester is present
Absence of color or light yellow color	dicastic polyester is not present

8. Test for Epoxy Coatings

8.1 All common epoxy coatings give positive results in this test. Epoxy ester coatings may also give positive results.

8.2 Apparatus:

8.2.1 **Ashless or Low-Ash Filter Paper**, 90 to 110-mm diameter.

8.2.2 Medicine Droppers.

8.3 Reagent:

8.3.1 Sulfuric Acid, concentrated.

8.4 **Procedure**—Support the filter paper off surfaces that may be damaged or could cause interference in the test. A watch glass can be used to support the paper. Place a specimen of coating in the filter paper. Place 2 or 3 drops of sulfuric acid directly on the coating. Place 1 or 2 drops of acid elsewhere on the filter paper not in contact with the coating. Let stand for 1 to 2 min. Carefully hold and tilt the filter paper toward the vertical until the acid runs down the paper away from the specimen. Wait 10 to 30 s or until there is development of color in the acid itself, not on the coating.

8.5 Interpretation of Results

Observation	Identification
Development of red to violet color in the acid	presence of epoxy
Absence of color in the acid	coating is not epoxy

8.5.1 If red to violet color develops in the drop of acid not in contact with the specimen, then the paper is contaminated or was placed on an epoxy or epoxy-coated surface. Discard and repeat the test. A very slight pink color may develop in the acid. This is not a positive result. Bitumen-filled epoxies may discolor the acid enough to mask color development. If the acid stream is discolored brown to black, carefully rinse the filter paper briefly in water or under running water if available. Color from a positive test will remain in the filter paper after the discoloration is washed off. The filter paper itself will be charred brown by the acid and eventually dissolve. The color of a positive test should occur early enough to be seen before the paper chars.

9. Test for Pigments that Contain Lead and Hexavalent Chromium

9.1 Summary of Test Method:

9.1.1 Knowledge of the presence of pigments that contain lead and hexavalent chromium in an existing coating system may be important in a decision on whether the coating system is to be retained and recoated or on the method of removal and disposal of the coating system residue.

9.1.2 Pigments that contain lead or hexavalent chromium, or both, may be used both in primers as rust-inhibitive pigments and in topcoats as weather-resistant colored pigments. Depending on the ease with which a coating system can be separated, topcoats and primer coats may be individually tested for lead or hexavalent chromium, or both.

9.1.3 The presence of lead and hexavalent chromium containing pigments can be qualitatively determined in the field with the following two tests. Both tests can be done directly on the coated surface or on chips or dust of the coating placed in the well of a spot plate.

9.2 *Apparatus*—Glass beakers, jars or bottles, porcelain spot plates, sandpaper, razor blade or knife.

9.3 Reagents:

9.3.1 *Sodium Sulfide Powder or Crystals.*

9.3.2 *Hydrochloric Acid, concentrated.*

9.3.3 *Diphenylcarbohydrazide Powder.*

9.3.4 *Phosphoric Acid, concentrated (85 %).*

9.3.5 *Acetone.*

9.3.6 *Denatured Ethanol.*

9.3.7 *Distilled or Deionized Water.*

9.4 Preparation of Test Solutions:

9.4.1 A solution of sodium sulfide in water is used to test for presence of lead. Prepare the solution by dissolving 1.5 g of sodium sulfide in 20 mL of distilled water and adding hydrochloric acid dropwise while swirling the solution until a white precipitate forms and remains with continued swirling (pH should be about 8). A proportionally smaller or larger amount of solution can be made up. The solution loses strength with age. Test the solution by placing a drop on a strip of lead acetate paper and observing the paper for the development of the black color of lead sulfide. If color development does not occur, discard the solution and make a fresh one.

9.4.2 A solution of 1,5-diphenylcarbohydrazide is used to test for the presence of hexavalent chromium. Prepare the solution using the following or proportionally smaller amounts of ingredients. Dissolve 0.5 g of 1,5-diphenylcarbohydrazide in a mixture of 20 mL acetone and 20 mL ethanol in a beaker, warming the beaker in warm water if necessary to facilitate solution. Carefully add 20 mL of phosphoric acid to 20 mL of cold distilled water in a separate container. Slowly add the acetone-ethanol mixture to the dilute acid solution and mix thoroughly by swirling. The 1,5-diphenylcarbohydrazide solution is not stable. It

may be stored for short periods of time in an opaque glass bottle but is best prepared just prior to use. The solution can be tested by placing a drop on a material known to contain a hexavalent chromium pigment. If a blue to violet color does not rapidly develop in the drop of solution, discard the solution and prepare a fresh solution.

9.5 Procedure:

9.5.1 Abrade two separate spots on the coating film with sandpaper or knife if the tests are to be done on the coating surface. Alternatively, abrade one spot of the coating and collect the dust and flakes in two wells of a spot plate. Abrasion of the film is required to expose pigments in an aged, weathered film.

9.5.2 On one abraded spot or on the specimen in one well of the spot plate, place 1 or 2 drops of sodium sulfide solution. Development of a black or dark grey color on the film or flakes indicates the presence of lead. Lack of color development indicates less than 0.1 % by weight lead (approximate practical limit of sensitivity of this test procedure).

9.5.3 Metals other than lead give a positive sulfide test but may not be common to coatings or usually do not interfere in this test in the form present. If there is doubt, laboratory testing beyond the scope of this test method is required to confirm the presence of lead.

9.5.4 On one abraded spot or to the second well of the spot plate place 1 or 2 drops of 1,5 diphenylcarbohydrazide solution. Rapid development of blue to violet color in the solution droplets indicates the presence of hexavalent chromium.

9.6 *Interpretation of Results*—The combination of results from the two tests, together with consideration of the color of the coating, may be used to establish more definitely the pigments that are present, as shown in the following scheme, which is not all inclusive:

Observation	Identification
Positive for lead, negative for chromium	pigment is red lead, white lead, or lead suboxide (white)
Negative for lead, positive for chromium	pigment is zinc or strontium chromate (yellow)
Positive for lead, positive for chromium	chrome yellow, chrome green, chrome orange, or molybdate orange (all color pigments containing lead chromate) or basic lead silichromate (orange-red inhibitive pigment)

10. Precision and Bias

10.1 Precision cannot be determined for these test methods because the variability of coatings on a surface does not permit the collection of reliably uniform samples and because few specimens will be tested from one particular coating.

10.2 Bias cannot be established for these test methods because accepted reference standards are not available.

11. Keywords

11.1 coatings; field; generic resin; identification

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Resin Solution Dilutability¹

This standard is issued under the fixed designation D 5062; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (^{e1}) indicates an editorial change since the last revision or reapproval.

^{e1} NOTE—Keywords were added editorially in March 1991.

1. Scope

1.1 This test method covers a titrametric/gravimetric determination of resin solution dilutability which gives a numerical value for the overall solubility of the resin expressed as percent dilutability.

1.2 This test method is applicable only if the test solution is of sufficient clarity to allow accurate visual judgement of the end point and of low enough viscosity for efficient mixing to take place.

1.3 This test method is primarily for, but not limited to, resins used in the printing ink industry.

1.4 The percent solvent tolerance of a resin can be determined using this test method if the solvent in the resin solution and the dilution solvent are the same.

1.5 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
 - D 1725 Test Method for Viscosity of Resin Solutions²
 - E 1 Specification for ASTM Thermometers³

3. Terminology

3.1 Definition of Term Specific to This Standard:

3.1.1 *resin solution dilutability*—the maximum amount of diluent tolerated to reach a defined degree of turbidity; beyond this point, resin precipitation will occur.

4. Summary of Test Method

4.1 A sample of resin solution is weighed into a glass beaker that is placed over a piece of 10 point print (standard newspaper print).

4.2 The dilution solvent is added slowly from a buret until the newsprint can no longer be read (cloud point) when viewed from the top of the beaker.

4.3 The percent dilutability is calculated by weight.

5. Significance and Use

5.1 This test method provides a means for resin producers and users as well as solvent and varnish manufacturers to rate various types of resins for solubility by assigning a numerical dilutability value. This percent dilutability value can be used to differentiate resin types for end users and can be utilized as a quality control tool by resin manufacturers.

5.2 When running a series of these tests the same lot or batch of dilution solvent must be used throughout to ensure reproducible results.

6. Apparatus

6.1 *Glass Beaker*, 150-mL (51-mm diameter, 79-mm height).

6.2 *Graduated Buret*, 50 mL.

6.3 *Constant Temperature Water Bath* at 25°C.

6.4 *Thermometer*, 0 to 40°C range with subdivisions of 0.5°C conforming to Specification E 1.

6.5 *Magnetic Stirring Bar and Stirring Plate or Stirring Rod*.

6.6 *Sheet of Newsprint*, with 10-point (Note), No. 3 old-style type, lower case letters 1.5-mm high with normal spacing, upper and lower case with no italicized or bold letters.

NOTE—The term point is derived from the American Point System. 72 points = 1 in.

7. Reagents and Materials

7.1 *Resin Solution*—A resin solution prepared in such a way as to provide an accurate percent solids and to have sufficient clarity to allow accurate visual judgment of the cloud point. The solution must also have low enough viscosity at 25°C to allow efficient mixing during the addition of the dilution solvent.

7.2 *Dilution Solvent*—The dilution solvent for this test is typically, but not limited to, a hydrocarbon. The dilution solvent should have minimal loss by evaporation at room temperature.

8. Procedure

8.1 Preparation of Resin Solution:

8.1.1 The resin solution may be prepared by either heating the resin/solvent combination as described in Test Method D 1725, or dispersing using a high-speed mixer such as a blender. Please note results may vary dependent on method of preparation.

8.1.2 The ratio of resin to solvent in the preparation should correspond as much as possible to the intended end

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.37 on Ink Vehicles.

Current edition approved May 25, 1990. Published July 1990.

² *Annual Book of ASTM Standards*, Vol 06.02.

³ *Annual Book of ASTM Standards*, Vol 14.03.

use of the material, but should be chosen to avoid difficulty in effecting solution at a low enough viscosity for efficient mixing during dilution.

8.1.3 The precision of weighing the resin and solvent should ensure a maximum deviation of $\pm 1\%$ in the desired concentration. This percent solids must not be altered during resin solution preparation by solvent loss.

8.2 Weigh a 150-mL glass beaker with the magnetic stirrer bar or stirring rod. Record this tare weight as W_1 .

8.3 Accurately weigh 10 ± 0.1 g of the resin solution into the beaker. Record this sample weight as W_2 .

8.4 Place the beaker in a 25°C constant temperature bath until the resin solution reaches $25 \pm 1^\circ\text{C}$ as measured with the thermometer specified in 6.4.

8.5 Prepare the dilution solvent by bringing it to a temperature of $25 \pm 1^\circ\text{C}$ before adding it to the buret.

8.6 Place the beaker over the piece of 10-point print and begin adding solvent slowly (5 to 6 mL/min) while stirring. Ensure that the sample temperature remains at $25 \pm 1^\circ\text{C}$, which may require periodic return of the beaker to the water bath. If the sample has started to form a haze it should not be returned to the water bath.

8.7 Observe the 10-point print from the top of the beaker looking through the solution.

8.8 If the resin solution begins to cloud, add solvent slowly one drop at a time while continuing to mix. Do not return the sample to the water bath at this point. If more than 100 g is required, the resin solution can be considered infinitely soluble unless otherwise specified in an agreement between the purchaser and supplier.

8.9 The end point is reached when the 10-point print cannot be read looking through the solution from the top of the beaker.

8.10 Record the final weight of the beaker and its contents (W_3).

8.11 Record the sample temperature at the end point.

9. Calculation

9.1 Calculate the percent resin solution dilutability, D , as follows:

$$D = \frac{W_3 - (W_1 + W_2)}{W_2} \times 100 \quad (1)$$

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9.2 Calculate the percent solvent tolerance of the resin, S , (percent resin solids at cloud point) as follows:

$$T = \frac{[W_2 \times (S/100)]}{W_2 + [W_3 - (W_1 + W_2)]} \times 100 \quad (2)$$

where S = resin in solution, %.

10. Report

10.1 Report the following information:

10.1.1 The identification of the resins and the solvent in the solution as well as the dilution solvent,

10.1.2 The method used to prepare the resin solution,

10.1.3 The percent solids of the resin solution, and

10.1.4 The percent resin dilutability value and operating conditions including air temperature, relative humidity, initial resin solution temperature, final resin solution temperature, initial dilution solvent temperature, and final dilution solvent temperature.

11. Precision

11.1 An interlaboratory study was conducted in which one operator in each of eight laboratories determined the percent dilutability of two common resin solutions and two dilution solvents completed in duplicate on two different days. The pooled within-laboratory standard deviation was found to be 3.25 % of the value measured with 12 df. The between-laboratory standard deviation was 7.63 % of the value measured with 28 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

11.2 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator should be considered suspect if they differ by more than 9.19 % of the value measured.

11.3 *Reproducibility*—Two results, each the mean of duplicate determinations obtained by operators in different laboratories, should be considered suspect if they differ by more than 23.38 % of the value measured.

12. Keywords

12.1 dilutability value; percent dilutability; printing ink resins; resins; resin solution cloud point; resin solution dilutability; solvent tolerance



Standard Guide for Use of Certification of Coating Conformance Form¹

This standard is issued under the fixed designation D 5063; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This certification of coating conformance form provides procurement information concerning products being furnished in accordance with a specific coating specification and additional requirements contained in the purchase order (see Fig. 1).

1.2 The completed form can be utilized to help evaluate the acceptability of the paint being furnished.

2. Referenced Documents

2.1 ASTM Standards:

D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.45 on Marine Coatings.

Current edition approved May 25, 1990. Published July 1990.

² Annual Book of ASTM Standards, Vol 06.01.

3. Instructions for Completing Certification Form

3.1 Use only known and industry accepted descriptions. Standard definitions of terms relating to paint, varnish, lacquer, and related materials are provided in Terminology D 16 (see Fig. 2).

3.2 The sections are self-explanatory. Applicable information in Section II should be provided by the buyer. Applicable information in Section III should be provided by the seller.

3.3 For two-component coatings, separate forms should be provided for each component, if appropriate.

4. Keywords

4.1 certification form; conformance form

CERTIFICATION OF COATING CONFORMANCE FORM		Number:
		Date:
GENERAL TYPE AND DESCRIPTION:		
Generic Type:		
Manufacturer's Designation:		
Specification Number:		
Formula Number:		
Number of Components:		
Component Identification (if applicable):		
Color:		
BUYER INFORMATION:		
Name and Address:		
Purchase Order Number:		
Release Number (if applicable):		
Part Number (if applicable):		
Item Number (if applicable):		
Stock Number (if applicable):		
SELLER INFORMATION:		
Name and Address:		
Seller's Representative:		
(Include Telephone Number)		
Batch Number:		
Lot No.:		
Date of Manufacture:		
Date of Expiration of Certification:		
CERTIFICATION:		
This is to certify that the above material being furnished has been manufactured in accordance with the specification and meets all specification requirements contained in the above purchase order.		
NAME AND TITLE	SIGNATURE	DATE

FIG. 1 Certification Form

CERTIFICATION OF CONFORMANCE FORM		Number: A001
		Date: 6/26/88
I. GENERAL TYPE AND DESCRIPTION:		
Generic Type:		Polyamide Epoxy
Manufacturer's Designation:		Perfect Paint 1234
Specification Number:		MIL-P-24441
Formula Number:		Formula 150
Number of Components:		Two
Component Identification:		Component A
Color:		Green
II. BUYER INFORMATION:		
Name and Address:		American Shipbuilders 1234 Easy St. Anytown, USA
Purchase Order Number:		0001-0002
Release Number (if applicable):		#1
Part Number (if applicable):		11B
Item Number (if applicable):		#3
Stock Number (if applicable):		4321
III. SELLER INFORMATION:		
Name and Address:		Perfect Paint Co. 11 Profit St. Paint City, USA
Seller's Representative:		I.B. Responsive
(Include Telephone Number)		703/123-4567
Batch Number:		88-F-01
Lot Number:		0001
Date of Manufacture:		6/20/88
Date of Expiration of Certification:		6/19/89
IV. CERTIFICATION:		
This is to certify that the above material being furnished has been manufactured in accordance with the specification and meets all specification requirements contained in the above purchase order.		
I.M. Xpert, Tech Director		6/26/88
NAME AND TITLE	SIGNATURE	DATE

FIG. 2 Example of Completed Form

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1016 Race St., Philadelphia, PA 19103.



Standard Practice for Conducting a Patch Test to Assess Coating Compatibility¹

This standard is issued under the fixed designation D 5064; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the procedures for testing coating compatibility when maintenance of an in-place coating system is being contemplated. It does not cover procedures for assessing the integrity of the existing coating to determine if it can be repainted, nor does it establish the compatibility of the maintenance coating system with the substrate or corrosion products. The practice is intended for use in the field.

NOTE 1—Pass-Fail Criteria (for example, adhesion requirements) are not established by this practice. These should be established by the user or specifier with input from the supplier.

1.2 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- D 3359 Test Methods for Measuring Adhesion by Tape Test²
- D 4138 Test Method of Measurement of Dry Film Thickness of Protective Coating Systems by Destructive Means²
- D 4414 Practice for Measurement of Wet Film Thickness by Notch Gages²

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to the Federation of Societies for Coatings Technology (FSCT) *Paint/Coatings Dictionary*.³

4. Summary of Test Method

4.1 The materials under test are applied to the previously painted surface after proper surface preparation. After the appropriate time has elapsed, the test patch is examined for

visual defects and adhesion is determined.

5. Significance and Use

5.1 In performing maintenance of a coating system, the new coating being applied must be compatible with the existing coating. While general guides exist which indicate compatibility of different generic types of coatings, differences in manufacturer's formulation and the condition of the in-place coating will affect compatibility.

6. Procedure

6.1 Select test locations for evaluation that properly characterize differences in configuration of the structure and exposure, that is, vertical versus horizontal surfaces and bold versus sheltered exposure. A minimum of three test locations with one test patch in each is recommended.

6.2 The size of each test patch will be determined by the size and configuration of the test locations. Each test patch shall be as large as possible, with a minimum size of 10 ft² (0.93 m²) recommended.

6.3 Clean the surface of the test areas using the methods specified for the maintenance painting procedure (Note 2). Alternative methods of preparation may also be evaluated in separate, adjacent tests.

NOTE 2—This test method assesses compatibility with the existing coating only and does not apply to areas where the substrate is exposed by the methods of preparation.

6.4 Measure the existing coating thickness in accordance with Test Methods D 1186, D 1400, or D 4138, as appropriate for the substrate.

6.5 Measure the ambient conditions and surface temperature and assure the conditions are within the limits specified by the coatings manufacturer for the product being tested.

6.6 Apply the test coating to the thickness recommended for the particular job. Use the application technique as intended for use on the full-scale job. If agreed upon between the purchaser and the seller, the method of coating application may be different from that used on the job, that is, brush application of the test patch even though spray application will be used on the job. However, this can cause some error and is not generally recommended.

6.7 Immediately after application, measure the wet-film thickness in accordance with Practice D 4414. Make corrections to the application, if necessary, by either applying more material if the expected dry-film thickness is low or applying another test patch if the expected dry-film thickness is above the recommended maximum. Inspect each patch for application defects such as runs, sags, and holidays. If such defects cannot be corrected as a part of the initial application process, prepare a new test patch.

6.8 After the coating has dried, measure the dry-film

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¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

Current edition approved May 25, 1990. Published July 1990.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Paint/Coatings Dictionary*, Federation of Societies for Coatings Technology, Philadelphia, PA, 1978.

thickness in accordance with Test Method D 1186, D 1400, or D 4138.

NOTE 3—When using Test Methods D 1186 or D 1400, the dry film thickness is the difference in average thickness of the coating system less the average thickness of the in-place coating.

6.9 Allow the coating to cure or weather prior to testing. Cure durations are defined as long term and short term. Long-term curing provides the most reliable assessment of compatibility. Short-term curing provides for more rapid evaluation of results.

6.9.1 *Long-Term Curing*—Curing for as long a time as possible, with a minimum of six months preferred. Curing should span seasonal weather changes.

6.9.2 *Short-Term Curing*—Curing at the following minimum times based on average daily (24 h) temperatures:

50°F (10°C)	70°F (21°C)	90°F (32°C)
14 days	10 days	7 days

6.10 After curing, examine the total surface of each test patch for wrinkling, blistering, mudcracking, checking, cracking, peeling, lifting, and disbonding. Measure or rate

the adhesion in a minimum of five locations per test patch in accordance with Test Methods D 3359.

6.11 Examine the test patches for the defects noted in 6.10 on a regular schedule, discounting rust caused by previous tests such as adhesion measurements and destructive film thickness measurements.

7. Report

7.1 Report the following information:

7.1.1 The identity of the structure tested, the location and size of the test patches, the identity of the test coating, the method and grade of surface preparation, and the method of coating application.

7.1.2 For each test patch, the dry film thickness measurements and average of the in-place coating, the dry film thickness measurements and average of the test coating, the elapsed time for each evaluation, the visual defects noticed, and the results of adhesion tests.

8. Keywords

8.1 coating; coating compatibility; coating test patch; paint; paint compatibility; test patch

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Standard Guide for Assessing the Condition of Aged Coatings on Steel Surfaces¹

This standard is issued under the fixed designation D 5065; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides general guidelines for a detailed assessment of the condition of aged coatings on steel structures and the extent of rust breakthrough of the coated surface. Additional assessment may be required to support coating failure analyses or other job specific needs.

1.2 This guide does not address the problem of determining the structural condition of a steel substrate. It provides procedures to determine the percent of the surface rusted, but not the severity, condition, or cause of such rusting.

NOTE 1—A more comprehensive condition assessment procedure, Practice F 1133, based upon two sets of visual standards, one for level and one for extent of deterioration, has been developed for determining the condition of coatings on ship hulls.

1.3 This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- D 610 Test Methods for Evaluating Degree of Rusting on Painted Steel Surfaces²
- D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 3359 Test Method for Measuring Adhesion by Tape Test²
- D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films²
- D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers²
- D 5043 Test Methods for Field Identification of Coatings²
- F 1133 Practice for Inspecting the Coating System of a Ship's Underwater Hull and Boottop During Drydocking³

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.46 on Industrial Protective Coatings.

Current edition approved May 25, 1990. Published July 1990.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 01.07.

2.2 Steel Structures Painting Council Standard:⁴

SSPC-PA-2 Measurement of Dry Paint Thickness with Magnetic Gages

3. Summary of Practice

3.1 This practice for assessing the condition of coatings consists of identifying general types of components of a structure and assessing each separately for commonly occurring modes of coating deterioration and rust breakthrough of the coating using visual standards and simple evaluation tools. A form for recording the results of the assessment procedure (Fig. 1) is provided.

4. Significance and Use

4.1 Assessment of the condition of aged coated surfaces strengthens decisions on when coating maintenance is required, aids in the selection of effective coating maintenance procedures, and provides a means to characterize performance of coating systems.

5. Procedure

5.1 Survey the structure to (1) determine the general types of unique components (for example, for fuel tanks the components may be shell, roof, ladders, and piping) and the service exposure environment for each, (2) visually identify areas having a typical level of coating deterioration and rust breakthrough for each component and (3) identify areas having a much greater visual level of deterioration than typical and unique environmental conditions that may correspond to these areas (for example, bridge expansion joints). Record a description of the components and their general environment on an inspection form and describe areas having greater deterioration, as well as any unique associated environments in the remarks column. A suggested general format for data collection is shown in Fig. 1. Modification of the form (for example, adding or deleting specific items) will be required for each specific application.

5.2 Based upon the knowledge of what constitutes typical deterioration for each component as determined in the initial survey, examine the condition of the coating on a representative sample of each component. Rate the condition of the coatings using the appropriate ASTM visual standard for rust breakthrough (Test Methods D 610), blistering (Test Method D 714), peeling (use Test Methods D 610 to report amount), chalking (Test Methods D 4214), and cracking/checking (Test Method D 660) of the coating film. Record the rating in the appropriate column of the report form for each

⁴ Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

Structure _____	Date _____	Inspector _____	Overall Environment _____
Original Coating System _____	1st Maintenance Coating System _____	2nd Maintenance Coating System _____	
Surface Preparation _____	Surface Preparation _____	Surface Preparation _____	
Year Applied _____	Year Applied _____	Year Applied _____	
Primer _____	Primer _____	Primer _____	
Midcoat _____	Midcoat _____	Midcoat _____	
Midcoat _____	Midcoat _____	Midcoat _____	
Topcoat _____	Topcoat _____	Topcoat _____	

Structure	Description	Ratings						Measurements		
		Local Environment	Rust ^A	Under film Condition ^A	Peeling	Blistering	Cracking	Chalking	Thickness	Adhesion

^A Rusting corresponds to Test Methods D 610, that is, that observed upon visual inspection of the coated surface while underfilm condition corresponds to substrate condition under an intact coating as described in Section 4.

FIG. 1. Example 1 Report Form

component. Determine and record the type of peeling, for example, intercoat delamination. Rate the condition in enough areas to ensure that for each component the coating evaluation is representative of the condition over the entire structure. If additional areas of greater deterioration are detected during this assessment, make note of them in the remarks column.

NOTE 2—For the purpose of an initial general assessment, cracking and checking can be assessed as one type of failure, using the pictorial standards in Test Method D 660 to define type and extent.

5.3 When rusting beneath an intact coating film is suspected, examine the condition of the underlying substrate. Remove apparently intact coatings using chemical strippers or closely spaced parallel knife cuts. For structural steel, determine the type of previous surface preparation from the presence of millscale or profile. Identify evidence of corrosion from the presence of pits, black anodic spots or corrosion scale, or from results of a test for ferrous ion using potassium ferriferrocyanide paper. Record the results of the examination on the report form.

5.4 Using one of the procedures described in Test Methods D 1186, determine the coating thickness in enough areas of each component to ensure a representative measure. Record the measured thicknesses.

5.5 Using one of the procedures described in Test Methods D 3359 or D 4541, determine coating adhesion in enough areas of each component to ensure a representative measure. Record the adhesion reading and the type of procedure and equipment used.

NOTE 3—The number of areas in which coating thickness and adhesion is measured will depend upon the desired precision of the measurement. More measurements would be made on structures in which precise knowledge of the thickness and adhesion of the coating is required. SSPC-PA-2 states the required number of thickness measurements as a function of coating area for conformance of thickness to a specification.

5.6 If required for a maintenance decision, identify the generic type(s) of the existing coating film component from records or using the procedure in Test Method D 5043. To the extent possible, each layer of the film should be characterized.

6. Report

6.1 Prepare an inspection report. Figure 1 provides an example of the types of information to be included.

7. Keywords

7.1 assessment; coatings; condition; field; paint; weathered

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Standard Test Method for Determination of the Transfer Efficiency Under Production Conditions for Spray Application of Automotive Paints— Weight Basis¹

This standard is issued under the fixed designation D 5066; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides procedures for determination of the transfer efficiency (using a weight method) under production conditions for in-plant spray application of automotive paints.

1.2 The transfer efficiency is calculated from the weight of the paint solids sprayed and that deposited on the painted part. The recommended approach involves painting the part directly. Also described is an alternative approach for painting parts covered with aluminum foil.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in 10.9.*

2. Referenced Documents

2.1 ASTM Standards:

D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products²

D 2369 Test Methods for Volatile Content of Coatings²

2.2 USEPA/MVMA (Motor Vehicle Manufacturers Association) Standards:

EPA 450/3-88-018, U.S. Environmental Protection Agency Protocol for Determining the Daily Volatile Organic Compound Emission Rate of Automobile and Light Duty Truck Topcoat Operations,³

EPA Federal Reference Method 24—Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids, of Surface Coatings⁴

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *paint*—the liquid material that is applied onto the part to cover or coat the surfaces.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.55 on Factory Applied Coatings on Preformed Products.

Current edition approved May 15, 1991. Published July 1991. Originally published as D 5066 - 90. Last previous edition D 5066 - 90.

² Annual Book of ASTM Standards, Vol 06.01.

³ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Refer to EPA 450/3-88-018 dated December 1988. This protocol makes reference to the determination of production spray transfer efficiency.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Refer to CFR 40, Part 60, Appendix A.

3.1.2 *transfer efficiency*—the ratio of the weight of paint solids deposited to the total weight of paint solids used during the application process, expressed as a percent.

3.1.3 *paint weight solids content*—the weight of the non-volatile materials in the liquid paint material divided by the total weight of the paint, times 100, determined by Test Method D 2369.

3.1.4 *paint density*—the mass of a unit volume of the liquid paint material at any given temperature, determined by Test Method D 1475.

3.1.5 *satellite paint supply system*—a smaller, paint-circulating system separate from the main production paint-circulating supply system capable of supplying paint under the same conditions.

4. Summary of Test Method

4.1 The weight of the part to be painted is determined before and after the paint application process. The weight of liquid paint used per part is determined from material usage and part processing records. The determined weight solids content of the paint material is determined and used to calculate the paint solids sprayed per part. The transfer efficiency of the process is calculated by dividing the weight of paint solids deposited by the weight of the paint solids sprayed.

5. Significance and Use

5.1 This test method is specifically directed at the spray painting of automobile car and light duty truck bodies. The general principles are applicable to the painting of other automotive parts.

5.2 This test method may also be used to measure transfer efficiency in full-sized painting facilities simulating production conditions and operations.

6. Interferences

6.1 Limitations include the ability of the weighing device to determine accurately the weight of the paint solids deposited on the part and the capability of accurate measurement of the amount of paint sprayed (see Section 7).

6.2 It may be difficult to cover the surface of complex shaped parts with aluminum foil (see 11.6.11).

7. Apparatus

7.1 *Tension Load Cells*, with upper/lower transition pieces.

7.1.1 1500-lb (682 kg) capacity with 0.05-lb (0.02 kg) precision for weighing automobile body and support frame.

7.1.2 500 to 800-lb (227 to 364-kg) capacity with 0.05-lb (0.02 kg) precision for weighing mix tanks or automobile components.

7.1.3 A safety cable is required for upper to lower transition.

7.2 *Electronic Digitizer/Readout*, readability of 0.05 lb (0.02 kg) and special filtering.

7.2.1 The electronic digitizer/readout shall meet OIML International Organization of Legalized Metrology specifications.⁵

7.3 *Voltage Regulator*.

7.4 *Swivel Devices*, as required.

7.5 *Rule and Calipers* for measuring diameter of paint supply tank shaft, etc.

7.6 *Sample Containers*, clean and airtight for paint material.

7.7 *Lifting Device and Support Frame Apparatus* to lift body.

7.7.1 Total weight must not exceed capacity of load cell or scale.

7.8 *Standard Calibration Weight*, approximately 2 lb (0.9 kg).

7.9 *Measure Stick*, Starret precision spring tempered, satin chrome finish, 36 in. (91.4 cm), with 4R graduations, or equivalent.

Paint Usage Measurement Procedures

8.1 Transfer efficiency measurement requires that accurate measurements be made of the quantity of paint material used in the application process during the time period associated with the coating of specific vehicles or parts. Two general procedures are applicable for accurately measuring paint usage.

8.1.1 The preferred procedure is to determine the weight of paint used during the application study period. Under typical production conditions, such weighing may be difficult, due to the large number of applicators requiring paint supply from a common tank. If a separate, isolated paint supply tank is used in the test; it is important to control paint viscosity, temperature, and flow rate consistent with the regular production system (see 9.1).

8.1.2 Where direct paint usage measurement by weight is not practical, an alternative procedure for determining paint usage by volume is suggested. This procedure involves measuring the drop in paint level in the paint supply tank during the test. To provide sufficient accuracy, it may be necessary to paint a "block" (isolated group) of similar vehicles or parts from the paint supply system while usage measurements are taken. Typically, this may require painting approximately 30 vehicles (see 9.2).

9. Paint Usage Determination by Weight Procedure

9.1 This procedure for determining paint usage during the test is preferred when a satellite paint supply system is available for the process to be tested. With this procedure, it is usually easier to isolate paint usage for measurement purposes, and accurate results can be obtained with a smaller

number of vehicles or parts (see 9.1.1).

9.1.1 At least 5.0 lb (2.27 kg) of paint material must be used during the test with the specified weighing equipment.

9.2 Before a satellite paint supply system can be used, it must be demonstrated that the system is capable of supplying the paint at sufficient volume and pressure to maintain normal process conditions (fluid deliveries of the spray devices) and that the paint can be maintained at a representative temperature and viscosity for the duration of the test period. These requirements can best be assured by mounting the satellite tank on a load cell or scale and directly piping it into the production supply system of the spraybooth. For this procedure the requirements are as follows:

9.2.1 Level and calibrate a weighing device (see Section 7) for weighing the satellite supply tank.

9.2.2 If an electronic weighing device is used, it must be turned on long enough to achieve stability, following the manufacturer's directions. All weighing devices must be situated to minimize disturbance from vibration or air movement.

9.2.3 Introduce the paint material, reduced to spraying viscosity, into the tank to be weighed. Before the test is conducted, be certain that fluid flows are properly set, that all supply and return lines are filled with the paint, and that no leaks are present in the piping system.

9.2.4 Shut off the agitator to minimize vibration during the weighing of the system.

9.2.5 Calibrate the weighing device in accordance with manufacturer's instructions. Weigh and tare a lifting frame, if used to support the satellite paint supply tank.

9.2.6 Weigh the satellite paint supply tank before the test vehicles or parts are run. Flexible connections are required to minimize strain. Carefully note the configuration of the tank so that the same configuration is used for both initial and the final weighing (that is, hose connections, height, etc.). Weigh the tank until 2 consecutive measurements are obtained within the measurement error of the weighing system ± 0.05 lb (0.02 kg). Average the two readings. The satellite tank may be left on the weighing device during the painting operation to monitor painting usage on a continuous basis.

9.2.7 After painting the test vehicles or parts, reweigh the satellite paint supply tank in accordance with 9.2.5 and 9.2.6.

9.2.8 Obtain representative paint samples immediately after completion of the test for solids and density analysis.

10. Paint Usage Determination by Tank Level Measurement (Volume Procedure)

10.1 This procedure for determining the paint usage requires that the drop in the liquid level in the system supply tank be measured accurately. This procedure is applicable with either a satellite system or a main-mix room supply system. A sufficient number of parts must be processed in the test block to provide at least a 3-in. (7.6-cm) drop in the liquid level in the paint supply tank. Careful measurement is critical to the overall accuracy of the transfer efficiency test. This procedure consists of the following:

10.1.1 Accurately measure the inside diameter of the paint supply tank(s) to be used for the various test materials. If the agitator shafts, fill pipes, or any other objects are located in the measurement zone, the occupied volume of

⁵ Electronic digitizer, Model 5322, available from Sterling Scale Co., 20950 Boening, Southfield, MI 48075, has been found suitable for this purpose.

these items must be determined and subtracted from the total volume. Careful selection of the section of the tank for the test measurement will minimize the difficulty of this task. The measurements are used to develop a conversion factor between level drop and volume of paint.

10.1.2 Most main paint supply systems consist of two tanks connected together to maintain the material supply. During the test, the paint supply tank must be isolated. Check to ensure that there is no leakage or overflow between the two tanks and that the directional valves for paint return function properly. Make sure that these checks are made just prior to and after the test, and are done by measuring the volume in both tanks.

10.1.3 Place the test paint, which has been reduced to spraying viscosity, into the paint supply tank to be measured. No material additions or reductions may be made to the tank during the test. Before the test is conducted, make certain that valving is set so that paint is being drawn from the test tank and returned to the test tank, and that all air has been removed from the delivery system.

10.1.4 With satellite paint supply tank systems, special care must be taken to ensure that all fluid lines are completely filled.

10.1.5 Prior to taking the volume measurement, turn off any agitation within the tank that will interfere with the measuring process and then turn it back on after the measurement.

10.1.6 Record the tank levels before and after the test. Take the initial reading just prior to the first test vehicle or part of the block that enters the first application station in the tested process. Take the final reading just after the last vehicle or part has left the last application area in the tested process.

10.1.7 With clearcoat (or other paint materials that cannot be isolated to a specific block of cars due to other connected operations such as repair), take the initial reading as the first vehicle or part in the test block enters the first station applying clearcoat. Take the final reading when the vehicle after the last job in the test block enters the first station, applying clearcoat (the same point at which the initial reading was taken). If this is not done, the block size for clearcoat will be different from the block size for basecoat (this is acceptable but must be accurately reflected in the calculations).

10.1.8 The measurements are to be taken to the nearest $\frac{1}{16}$ in. with a measuring stick with $\frac{1}{16}$ -in. (1.6-mm) or less graduations. Measurements are made from the top of the tank to the top of the liquid level at least 3 to 4 in. (7.6 to 10.2 cm) from the sides of the tank. The top of the tank is to be determined by laying a straight edge across the top of the tank in the same position for each measurement.

NOTE—Caution—Any measuring instruments used in this procedure must be effectively grounded before contacting the coating tank or coating liquid surface. Review all measuring instruments for suitability and resistance to the paint solvents prior to the test.

10.1.9 It may be necessary to provide two-way radio communication between the mix-room monitor and the tested process operation to coordinate the measurement process and timing.

10.1.10 Obtain representative paint samples immediately

after completion of the test for density and solids determinations.

11. Paint Solids Deposited Measurement Procedure

11.1 To determine the transfer efficiency, it is necessary to determine both the weight of paint solids sprayed per vehicle or part and the weight of paint solids actually deposited onto the object in the tested process. These are obtained by weighing vehicles or parts incorporated in the same sample (block) used to obtain the paint usage measurement.

11.2 *Vehicle or Part Weighing Procedure*—The weight of paint solids deposited during the application process is determined by weighing the vehicle(s) or part(s) prior to and after the paint is applied and baked. At least two vehicles or parts are required to be tested. A control part is also run and weighed before and after the application process. No paint is applied to the control part in the process. The control is required to determine weight loss from miscellaneous materials (primarily sealants and plastics applied prior to the tested process) that may occur in the drying oven. The difference in the weight of the measurement vehicles adjusted for the weight loss in the control vehicle or part, is the weight of paint solids applied in the tested process.

11.3 Weighing of the parts requires the use of a precision weighing device and digitizer as described in 7.1 and 7.2. A lifting frame and hoist are required to support the test vehicle. The measurement site(s) must be selected to avoid disturbance caused by air movement or production activities and to allow convenient removal of the test vehicles or parts from the production line and their subsequent replacement to the production line. Fork trucks or other mechanical handling aids along with sufficient manpower to move the vehicle bodies into and out of the weighing frame will be required.

11.3.1 Passenger car bodies generally will weigh 800 to 900 lb (364 to 409 kg). Since the maximum capacity of the specified load cell is 1500 lb (682 kg), the weight of the lifting frame and rigging must be kept to less than 600 lb (273 kg) or a higher capacity load cell with the specified accuracy must be utilized.

11.4 It is critical that the vehicles or parts be allowed to cool completely to room temperature prior to weighing. Closely monitor heavy-metal sections (usually around the door frames) as these areas will cool more slowly than exterior body surfaces. If during the weighing process there is any doubt that the vehicle or part is fully cooled, weigh the test part and allow it to stand 15 to 20 min and weigh again. If a weight increase of more than 0.05 lb (0.02 kg) is noted, allow the body to cool an additional 15 to 20 min and repeat the weighing procedure. Repeat this process until consistent weights are obtained.

11.5 Check that all accessories and miscellaneous materials (hangers, spacers, etc.) remain in the same position (either on or off) for both the initial and final weighings.

11.6 Weighing Procedure:

11.6.1 Turn on the weighing device and digitizer and allow them to warm up in accordance with the manufacturer's instructions.

11.6.2 Zero the instrument in accordance with manufacturer's instructions.

11.6.3 Attach the lifting frame to the load cell. Lift and

the frame until two consecutive readings within 0.05 (2 kg) are obtained. Record this tare weight, W_t .

11.6.4 Mount a completely cooled vehicle or part into the frame. Lift the test unit until it is completely clear of the carrier and fully suspended by the load cell. If the test does not hang level, adjust the suspension point on the frame and recheck for proper tare weight. If necessary, calibration weights may be used to help level the test but be certain to include this weight in the initial tare weight.

11.6.5 Allow the suspended vehicle or part to completely stabilize (usually 2 to 3 min). Be certain that nothing is touching the test unit or suspension frame and record the weight, W_i .

11.6.6 Add a standard calibration weight to the suspended vehicle or part and check for instrument accuracy. Recalibrate the system if a discrepancy is noted.

11.6.6.1 Select the calibration weight to represent the estimated increase in weight of the vehicle or part in the weighing process.

11.6.7 Lower the vehicle or part back onto the carrier to remove the weight from the load cell. Relift the vehicle or part, as just indicated until two consecutive readings within 0.05 lb (0.02 kg) are obtained. Record the weight of two consecutive initial weights, that is W_i for part and $W_{c,i}$ for the control.

11.6.8 Remove the first body and repeat 11.6.5 through 11.6.7 for the other vehicles and the weight loss control vehicle (if included in this test run). Between test measurements and after the last unit is weighed, resuspend the empty weighing frame and verify the tare weight, W_t .

11.6.9 Apply the paint to the specified vehicles or parts, allowing the control unit to proceed through the spraybooth oven uncoated.

11.6.9.1 Considerable care must be taken in handling the weighed units to eliminate any weight gain or loss due to handling, parts addition, parts removal, or miscellaneous items.

11.6.10 After the vehicles or parts have been painted and dried in the oven, allow them to cool to room temperature and then reweighing. Repeat the weighing procedure in 11.6.1 through 11.6.8 and record the weights, $W_{f,i}$ for the coated part (1) and $W_{c,f}$ for the control.

11.6.11 *Foiled Part Procedure*—The procedure is identical to that described in 11.2 except that aluminum foil is applied to the vehicle or part. All the surfaces to be painted are covered with foil as smoothly and tightly as possible to ensure that the covered surface is representative of the area painted in the normal production environment.

12. Measurement of Other Paint Usage

12.1 When the measurement of paint used cannot be completely isolated to the application process under test because of the process and facilities arrangement, it may be necessary to obtain an estimate of paint solids deposited from the same paint supply system in the nonisolated operations (such as repair, etc.). With this estimated value, the total paint solids deposited in the main process can be directly correlated to the amount of paint used in calculating the resultant transfer efficiency. Such a correlation can be established by either making a separate estimate or measurement of the paint used

or the paint solids deposited outside the tested process. The amount of material involved should be small (less than 5 %) relative to the total paint used or deposited.

12.2 While an estimate of paint used is generally preferred, in practice it is usually easier to obtain an estimate of the amount of paint solids deposited onto the test unit. For repair type operations, an estimate of paint solids applied may be obtained by counting the number of panels repaired while the test block is being processed and multiplying by the square feet per panel and the average film build applied (determined from measurements or historical data). This technique works well since almost all repairs involve complete body panels for which data can readily be obtained.

13. Analysis of Paint Samples

13.1 Make the following analytical determinations for each paint sample obtained:

13.1.1 *Weight Fraction Solids*, in accordance with Test Method D 2369 per EPA Federal Reference Method 24.

13.1.2 *Paint Density*, in accordance with Test Method D 1475.

13.2 These determinations are required to calculate transfer efficiency. Obtain separate determinations for each paint material used in each test.

14. Calculations and Report Submission

14.1 Upon completion of the test and receipt of the paint analytical results, the transfer efficiency calculations can be made in accordance with the following procedure:

14.2 Calculate the average weight gain of the vehicle or part, corrected for the weight gain or loss of the control unit.

$$G_{avg} = \frac{\text{Sum}[W_f - W_i]}{n} - [W_{c,f} - W_{c,i}] \quad (1)$$

where:

G_{avg} = average weight gain, lb (kg),

W = weight, lb (kg),

W_f = final weight, lb (kg),

W_i = initial weight, lb (kg),

n = number of test units coated,

c = control,

$W_{c,f}$ = final weight of control vehicle or part, lb (kg), and

$W_{c,i}$ = initial weight of control vehicle or parts, lb (kg).

14.3 Calculate the average amount of paint used during the test period by either the weight procedure or the volume procedure.

14.3.1 *Weight Procedure* (see Section 9):

$$P_{avg} = [P_i - P_f]/m \quad (2)$$

where:

P_{avg} = average weight of paint used during the test, lb (kg),

P_i = initial weight, lb (kg),

P_f = final weight, lb (kg), and

m = number of units measured.

14.3.2 *Volume Procedure* (see Section 10):

$$P_{avg} = (V \times P)/m \quad (3)$$

where:

V = volume of paint used during the test,

D = paint density in accordance with Test Method D 1475, and

m = number of units measured.

14.4 Calculate the average paint solids used during the test period.

$$S_{avg} = P_{avg} \times F \quad (4)$$

where:

S_{avg} = average weight of paint solids used during the test, lb (kg), and

F = weight fraction solids in the paint material from Test Method D 2369.

14.5 Calculate the transfer efficiency T result.

$$T = G_{avg}/S_{avg} \times 100 \quad (5)$$

15. Precision and Bias

15.1 *Precision*—Engineering estimates based on error assumptions indicate an accuracy of $\pm 5\%$ can be expected for typical automotive painting operations.

15.2 *Bias*—Since there is no accepted reference procedure suitable for determining the bias for this test method, no statement on bias is being made.

16. Keywords

16.1 automotive painting; production method; spray-applied; transfer efficiency.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Specification for Artists' Watercolor Paints¹

This standard is issued under the fixed designation D 5067; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (^e) indicates an editorial change since the last revision or reappraisal.

^{e1} NOTE—Table X1.1 was editorially corrected in March 1991.

This specification establishes requirements for composition, physical properties, performance, and labeling of watercolor paints.

This specification covers pigments, vehicles, and additional requirements are included for pigment identification, fineness, and consistency.

Table 1 lists some pigments meeting the lightfastness requirements in this specification. In order to identify other pigments that meet these requirements, instructions are given for test specimen preparation. Test methods for determining relative lightfastness are referenced.

Referenced Documents

ASTM Standards:

D 55 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 1010 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems²

D 136 Practice for Labeling Art Materials for Chronic Health Hazards²

D 203 Test Methods for Lightfastness of Pigments Used in Artists' Paints²

D 284 Terminology of Appearance of Materials³

Terminology

Definitions:

1.1 **Colour Index Name**—consists of the category (type of dye or pigment), general hue, and an assigned number added to a colorant in the Colour Index⁴ as an international identification system.

1.1.1 **Discussion**—For example, the Colour Index name of one phthalocyanine blue pigment is Pigment Blue 15 (PB 15).

1.2 **Colour Index Number**—a five-digit number given in the Colour Index that describes the chemical constitution of a colorant.

1.2.1 **Discussion**—For example, the Colour Index

Number of one phthalocyanine blue pigment is 74160.

3.1.3 Appearance terms used in this standard are defined in Terminology E 284.

3.2 Descriptions of Terms Specific to this Standard:

3.2.1 **watercolor paint**—a pigment dispersion in a water soluble gum/resin vehicle that dries water soluble and is intended primarily for transparent applications.

4. Significance and Use

4.1 This specification establishes quality requirements and provides a basis for common understanding among producers, distributors, and users.

4.2 It is not intended that all paints meeting the requirements be identical nor of uniform excellence in all respects. Variations in manufacture, not covered by this specification, may cause some artists to prefer one brand over another, either of which may be acceptable under this specification.

5. Labeling Requirements

5.1 Pigment(s) Identification:

5.1.1 Every label shall include for each pigment contained in the paint (1) the information underlined in Table 1 (which includes the Common Name, Colour Index Name, and any additional terms necessary to identify the form of the pigment) and (2) the appropriate Lightfastness Category.

5.1.2 The complete pigment identification given in Table 1, which also includes the Colour Index Number and a simple chemical description, shall be given in an appropriate producer publication. Manufacturers are encouraged to put this complete identification on the container label when label size permits.

5.1.3 The Common Name shall be placed on the front of the label and shall be the name of the paint except as described in 5.1.5 and 5.1.6. Other identification may be placed elsewhere on the container.

5.1.4 The Colour Index Name may be spelled out in full or abbreviated depending on the size of the label. Example: Pigment Blue 15, or Pig. Blue 15 or PB 15.

5.1.5 **Substituted Pigments**—In the case of substituted pigments, the word "Hue" in equal size letters shall follow in the title, on the front of the tube, immediately after the name of the pigment that has been simulated. Directly below the title, the Common Name of the significant pigment used shall be given in letters no less than the next type size smaller than the title. For example:

COBALT BLUE HUE
(ULTRAMARINE BLUE).

5.1.6 Proprietary names or optional names may be used

¹This specification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

Current edition approved May 25, 1990. Published September 1990.

Annual Book of ASTM Standards, Vol 06.01.

Annual Book of ASTM Standards, Vol 14.02.

²Colour Index, 3rd ed., The Society of Dyers and Colourists, London, 1975, five vols and revisions. Available from the American Association of Textile Chemists and Colorists, PO Box 12215, Research Triangle Park, NC 27709.

TABLE 1 Suitable Pigments List

Note—Underlined information and the lightfastness category in the table shall be included on every label.

Key:

Lightfastness Category:

Lightfastness I Excellent Lightfastness
Lightfastness II Very Good Lightfastness

Abbreviations Used for Colour Index Names:

NR Natural Red
PB Pigment Blue
PBk Pigment Black
PBr Pigment Brown
PG Pigment Green
PO Pigment Orange
PR Pigment Red
PV Pigment Violet
PW Pigment White
PY Pigment Yellow

Pigment Notations:

(CC) Concentrated cadmium pigments may contain up to 15 % barium sulfate for color control. Cadmium-barium pigments contain a much higher amount of barium sulfate.
(NA) Colour Index name or number not assigned.
(SM) Sensitive to moisture in direct sunlight.

Colour Index Name	Lightfastness Category	Common Name and Chemical Class	Colour Index Number
Watercolors			
YELLOWS			
PY 3	II	Arylide Yellow 10G, with option of adding the name Hansa Yellow Light, arylide yellow	11710
PY 31	I	Barium Chromate Lemon, barium chromate	77103
PY 34	I	Chrome Yellow Lemon, lead chromate and lead sulfate	77600
PY 35	I	Cadmium Yellow Light, concentrated cadmium zinc sulfide (CC) (SM)	77205
PY 35:1	I	Cadmium-Barium Yellow Light, cadmium zinc sulfide coprecipitated with barium sulfate (SM)	77205:1
PY 37	I	Cadmium Yellow Medium or Deep, concentrated cadmium sulfide (CC) (SM)	77199
PY 37:1	I	Cadmium-Barium Yellow Medium or Deep, cadmium sulfide coprecipitated with barium sulfate (SM)	77199:1
PY 40	II	Aureolin, with option of adding the name Cobalt Yellow, potassium cobaltinitrite	77357
PY 42	I	Mars Yellow, with option of adding the name Yellow Iron Oxide, synthetic hydrated iron oxide	77492
PY 42	I	Mars Orange, synthetic hydrated iron oxide	77492
PY 43	I	Yellow Ochre, natural hydrated iron oxide	77492
PY 97	IIA	Arylide Yellow FGL, arylide yellow	11767
ORANGES			
PO 20	I	Cadmium Orange, concentrated cadmium sulfo-selenide (CC)	77202
PO 20:1	I	Cadmium-Barium Orange, cadmium sulfo-selenide coprecipitated with barium sulfate	77202:1
PO 62	I	Benzimidazolone Orange H5G, monoacetolone	NA
REDS			
PR 101	I	Indian Red, synthetic red iron oxide (bluish hue)	77491
PR 101	I	Light or English Red Oxide, synthetic red iron oxide (yellowish hue)	
PR 101	I	Mars Red, with option of adding the name Red Iron Oxide, synthetic red iron oxide	77491
PR 101	I	Mars Violet, with option of adding the name Violet Iron Oxide, synthetic iron oxide (violet hue)	77015
PR 101	I	Venetian Red, synthetic iron oxide (yellowish hue)	77491
PR 104	I	Chrome Orange, lead chromate and lead molybdate	77605
PR 108	I	Cadmium Red Light, Medium, or Deep, concentrated cadmium-seleno sulfide (CC)	77202:1
PR 108:1	I	Cadmium-Barium Red Light, Medium, or Deep, cadmium seleno-sulfide coprecipitated with barium sulfate	77202:1
PURPLES			
PV 14	I	Cobalt Violet, cobalt phosphate, cobalt ammonium phosphate	77360
PV 15	I	Ultramarine Red, complex silicate of sodium and aluminum with sulfur	77362
PV 15	I	Ultramarine Violet, complex silicate of sodium and aluminum with sulfur	77007
PV 16	I	Manganese Violet, manganese ammonium pyrophosphate	77007
PV 19	IIA	Quinacridone Violet, quinacridone violet b	77742
BLUES			
PB 15	IIA	Phthalocyanine Blue, copper phthalocyanine	74160
PB 27	I	Prussian Blue, with the option of adding the name Milori Blue, ferriammonium ferrocyanide	77510
PB 28	I	Cobalt Blue, oxides of cobalt and aluminum	77346
PB 29	I	Ultramarine Blue, complex silicate of sodium and aluminum with sulfur	77007
PB 33	I	Manganese Blue, barium manganate with barium sulfate	77112
PB 35	I	Cerulean Blue, oxides of cobalt and tin	77368
PB 36	I	Cerulean Blue, Chromium, oxides of cobalt and chromium	77343
GREENS			
PG 7	I	Phthalocyanine Green, chlorinated copper phthalocyanine	74260
PG 17	I	Chromium Oxide Green, anhydrous chromium sesquioxide	77268
PG 18	I	Viridian, hydrous chromium sesquioxide	77269
PG 19	I	Cobalt Green, oxides of cobalt and zinc	77335

TABLE 1 Continued

Pigment Index Number	Lightness Category	Common Name and Chemical Class	Colour Index Number
	Watercolors		
BROWNS			
PBr 7	I	Burnt Sienna, calcined natural iron oxide	77492
PBr 7	I	Burnt Umber, calcined natural iron oxide containing manganese	77492
PBr 7	I	Raw Sienna, natural iron oxide	77492
PBr 7	I	Raw Umber, natural iron oxide containing manganese	77492
BLACKS			
PBk 6	I	Lamp Black, nearly pure amorphous carbon	77266
PBk 7	I	Carbon Black, nearly pure amorphous carbon	77266
PBk 8	I	Ivory Black, amorphous carbon produced by charring animal bones	77267
PBk 9	I	Gray Hydrated Aluminum Silicate, hydrated aluminum silicate	77017
PBk 19	I		
WHITES			
PW 4	I	Zinc White, zinc oxide with option of adding the name Chinese White	77947

These pigments were put into the lightfastness II category pending results of retesting.

provided the Common Name(s) given in Table 1 appears on front of the label directly under the proprietary or optional name in letters no less than the next type size smaller than the proprietary or optional name.

5.1.7 *Mixed Pigments*—Artists' paints containing more than one colorant comply with this specification if all colored pigments used are on the suitable pigment list (Table 1) and provided the mixture itself has passed all other test requirements in this specification. The lightfastness category shall be that of the least lightfast pigment. This lightfastness category may be changed if these paints are tested for lightfastness in accordance with Test Methods D 4303 and results indicating a different category are submitted to ASTM Subcommittee D01.57 for evaluation.

5.2 Provide on the label the identification of the gum/medium used.

5.3 *Lightfastness*—The label shall contain the word "lightfastness" followed by the appropriate rating, I or II, as given for each pigment in Table 1.

5.3.1 Lightfastness I pigments, when made into paint specimens as described in Section 7 and exposed, tested, and rated in accordance with Test Methods D 4303, shall have a color difference (ΔE^*_{ab}) of 4 or less CIELAB units between the specimens measured before and after exposure.

5.3.2 Lightfastness II pigments, when made into paint specimens as described in Section 7 and exposed, tested, and rated in accordance with Test Methods D 4303, shall have a color difference (ΔE^*_{ab}) of more than 4.0 but not more than 10 CIELAB units between the specimens measured before and after exposure.

5.3.3 Pigments were placed in a lightfastness category on the basis of either known historical performance in art works or the ratings from four lightfastness tests conducted as described in Test Methods D 4303. Results from further tests on these, or other pigments, are solicited by Subcommittee D01.57.

5.3.3.1 The lightfastness category of a pigment shall be changed if results from several further tests conducted in accordance with Test Methods D 4303 and approved by ASTM Subcommittee D01.57, establish a different lightfastness category than the one given in Table 1.

5.3.3.2 Additional pigments shall be placed in Table 1 after they have been tested for lightfastness in accordance

with Test Methods D 4303 and the test results submitted to ASTM Subcommittee D01.57 for evaluation, provided the results demonstrate that the pigments have the lightfastness ratings required for Lightfastness I or Lightfastness II, as described above.

5.3.4 For information and to establish nomenclature, pigments in Lightfastness III category are given in Table X1.1 in Appendix X1, but are not to be used in paint conforming to this specification. These pigments have a color difference before and after exposure of more than 8.0 but not more than 16.0 CIELAB units.

5.4 *Toxicity*—All products and labeling must conform to the Federal Hazardous Substances Act and to Practice D 4236.

5.5 *Statement of Conformance*—"Conforms to ASTM Specification D 5067," or "Conforms to ASTM D 5067," or "Conforms to the quality requirements of ASTM D 5067." This statement may be combined with other conformance statements, such as, "Conforms to the quality and health requirements of ASTM Specification D 5067 and Practice D 4236."

5.6 *Address*—Include on the label (1) the name and address of the manufacturer or importer and (2) the country of manufacture.

6. Quality Assurance for Artists' Watercolor Paints

6.1 *Conditions not Covered in This Specification That Affect Artists' Watercolor Paints:*

6.1.1 *Substrate*—The effective pH of the paper used will affect the long-term color of the applied watercolor.

6.1.2 *Environmental Conditions*—Factors such as temperature, humidity, airflow, and light conditions affect application properties, drying time, and adhesion.

6.1.3 *Storage*—With aging and elevated temperatures there may be a change in consistency and a discernible separation of vehicle.

6.2 *Vehicles*—Only water soluble gums/resins shall be used.

6.3 *Pigments*—Pigments used in watercolors shall be limited to those in Table 1. Their lightfastness rating shall be the numeral given in the same row.

6.4 *Additives*—Thickeners, preservatives, surfactants, and humectants may be used to achieve consistency, prevent

microbe deterioration, and control application results.

6.5 *Inerts*—Inerts shall only be used to produce desirable working qualities.

6.6 *Preparation of Sample*—For paste and fluid paints, empty the contents of the previously unopened container onto a glass slab and mix thoroughly with a spatula to a homogeneous sample. For cake paints, take a piece of the cake on a glass slab and add water and mix until a homogeneous paint is formed.

6.7 *Coarse Particles*—Paints shall be free of oversize particles and shall form a uniform film. The maximum content of coarse particles shall be 1 weight % as determined by Test Methods D 185.

6.8 *Fineness of Dispersion*—Determine the fineness of dispersion by Test Method D 1210. For paste paint, on a glass plate, using a spatula, mix the paint with an equal volume of water until homogeneous. The maximum allowable grind reading is 1.5 mils (40 μm).

6.9 *Consistency*—Paints shall be smooth and easily solubilized with water to a homogeneous color.

6.10 *Freeze-Thaw Stability*—Using a freezer that has a temperature of 20°F (−7°C) or lower, subject the paint to five freeze-thaw cycles. A freeze-thaw cycle shall consist of freezing the paint to a solid state (minimum of 18 h) and then thawing the paint to room temperature (minimum of 5 h). The paint shall then meet the requirements of 6.7, 6.8, and 6.9.

7. Lightfastness Determination

7.1 If a pigment is not listed in Table 1, test specimens of a watercolor containing the pigment shall be prepared. These test specimens shall be tested in accordance with the requirements for exposure and evaluation given in Test Methods D 4303.

NOTE—A report of the results of these tests may be submitted to Subcommittee D01.57 for inclusion of the pigments in Table 1. The report shall include information on test conditions and instruments used and shall be accompanied by the test specimens (which will be returned).

7.2 Materials:

7.2.1 *Filter Paper*, 15.0-cm diameter, ashless.⁵

7.2.2 *Drawdown Bar* with 3-mil (75- μm) aperture.

7.2.3 *Posterboard*, lightweight, approximately 20 mils (0.5 mm) thick, having a glossy finish on one side.

7.2.4 *Distilled Water*.

7.2.5 *Acrylic Latex Adhesive*.

7.3 Preparation of Test Paints:

7.3.1 The pigment to be tested may be milled in a soft

paste consistency. If a prepared artists' paint of known composition is available it may be used for this test instead of preparing a standard.

7.3.2 Dilute the watercolors with water and drawdown on paper until the spectrophotometric measurement of the dry paint shows from 35 to 45 % reflectance at the wavelength of maximum absorption for that pigment. The wavelength of maximum absorption is located at the point of lowest reflectance on the spectral curve between 420 and 620 nm. If using a tristimulus filter colorimeter, the lowest of the three filter readings is the region of maximum absorption and the dilution should be adjusted so that a reading of 35 to 45 % reflectance is obtained with this filter. The diffuse white reference standard for all measurements should have an absolute reflectance between 97 and 100 %.

7.3.3 Use an applicator with a 3-mil (75- μm) aperture to make a drawdown on the filter paper. Tape the filter paper to a smooth surface such as a piece of glass. Place the drawdown bar just above the upper edge of the paper so it is ready to use. Pour a small amount of the diluted paint, which is thoroughly mixed, onto the top of the filter paper. Using the drawdown bar, draw the paint down, running the excess off the edge of the filter paper at the bottom. Quickly untape the filter paper and hang it to dry at room temperature. There will be a dark puddle area where the paint was originally applied, but the remaining part of the paper will be uniform in color for use in obtaining spectrophotometric measurements.

7.3.3.1 Prepare four specimen panels for each pigment under test. Two are used in the first lightfastness tests and two are retained in subdued light, one for visual comparisons with the exposed panels and one in case a third test is needed to supplement results from the first two tests, as described in Test Methods D 4303.

7.3.3.2 Apply the test paints to the filter paper as described in 7.3.3. The panels should be air dried for 2 h and then put in an oven at 50°C for overnight drying.

7.3.3.3 Cut the uniform color section of the filter paper drawdown panel into 1½-in. (38-mm) square panels. Adhere the panels to the light posterboard (see 7.2.3) using a thin coat of an acrylic-latex adhesive. The size of the posterboard shall conform to the dimensions of the exposure equipment test racks.

8. Exposure

8.1 Conduct exposure tests, calculate mean color difference, and assign pigments to lightfastness categories as described in Test Methods D 4303.

9. Keywords

9.1 lightfastness; quality requirements; specimen preparation; watercolors

⁵ Whatman No. 42 Filter Paper, available from Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219, has been found satisfactory for this purpose.

APPENDIX

(Nonmandatory Information)

X1. LIGHTFASTNESS III

The pigments in Table X1.1 are not sufficiently common terminology. They may be satisfactory when used fast to be used in paints that conform to this specifica- full strength (without dilution) or with extra protection from These pigments are listed here solely to establish exposure to light.

TABLE X1.1 Lightfastness III

—Underlined information and the lightfastness category in the table shall be included on every label.

Notations:

Lightfast type

Colour Index name or number not assigned

Colour Index Name	Lightfastness Category	Common Name and Chemical Class	Colour Index Number
RY1:1	III	Arylide Yellow G, with option of adding the name Hansa Yellow Medium, arylide yellow	11680
RY74 (LF)†	III	Arylide Yellow 5GX, with option of adding Hansa Yellow 5GX, arylide yellow	11741
RY110	III	isoindolinone Yellow R, tetrachloroisoindolinone	NA
RB106	III	Vermilion, mercuric sulfide	77766
RB112	III	Naphthol AS-D, naphthol AS-D	12370
RB122	III	Quinacridone Magenta, quinacridone	73915

Editorially corrected.

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Standard Practice for Preparation of Paint Brushes for Evaluation¹

This standard is issued under the fixed designation D 5068; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes the preparation of paint brushes for evaluation.

1.2 This practice is applicable to paint brushes 2 to 4 in. (50 to 100 mm) in width.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Practice

2.1 Using a paint chosen for the evaluation, the brush to be tested is repeatedly loaded with this paint and brushed out over a specified area in a specified application time.

3. Significance and Use

3.1 Until a paint brush is fully wetted with paint, only part of the paint loaded onto the brush can be transferred to the surface being painted. By properly preparing the brush before using, the amount of paint delivered to the surface can be made more uniform and reflect real use.

4. Apparatus

- 4.1 *Container*, to hold paint, for example, a quart can.
- 4.2 *Test Brush*, 2 to 4 in. (50 to 100 mm) in width.

5. Materials

- 5.1 *Test Paint*.
- 5.2 *Brush-Out Panels*, or other typical panels to be used.²
- 5.3 *Masking Tape*, to secure the panel to a flat surface.

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.61 on Paint Application Tools.

Current Edition Approved May 25, 1990. Published July 1990.

² Any smooth type panel, for example, upson boards, can be used. A Leneta 8H-BW Chart, available from The Leneta Co., P. O. Box 86, Ho-Ho-Kus, NJ 07427, has been found satisfactory for this purpose.

6. Procedure

6.1 Secure the brush-out panel with masking tape to a flat, smooth, horizontal surface.

6.2 Dip the test brush into the specified paint to the depth shown below:

Brush Width, in. (mm)	Depth, in. (mm)
2 and 2½ (50 and 62.5)	1½ (38)
3 and 3½ (75 and 87.5)	1¾ (45)
4 (100)	2 (50)

6.3 Hold the brush at the specified depth in the paint for 10 s. Remove and hold the brush vertically for 30 s allowing any excess paint to drain.

6.4 After the drain period, immediately apply paint to the specified initial area on the brush-out panel as indicated below:

Brush Width, in. (mm)	Initial Area, cm ²
2 and 2½ (50 and 62.5)	250
3 to 4 (75 to 100)	500

6.5 Application time shall be 15 s for 2 and 2½-in. (50 and 62.5-mm) brushes and 20 s for 3 to 4-in. (75 to 100-mm) brushes.

6.6 Repeat this procedure three times.

6.7 Maintain the level of paint in the container. Keep the angle of the handle and displacement of the filaments of the test brush uniform throughout the entire series. When applying paint, always displace the test brush filament about one third to one half of the filament length and maintain the handle perpendicular to the paint-out surface.

6.8 When applying paint, always start in the middle of the area to be painted with each loading. This leaves the excess paint in the middle where it is easiest to spread to the area being covered and will result in a more uniform coverage (see Fig. 1).

7. Keyword

- 7.1 brush preparation

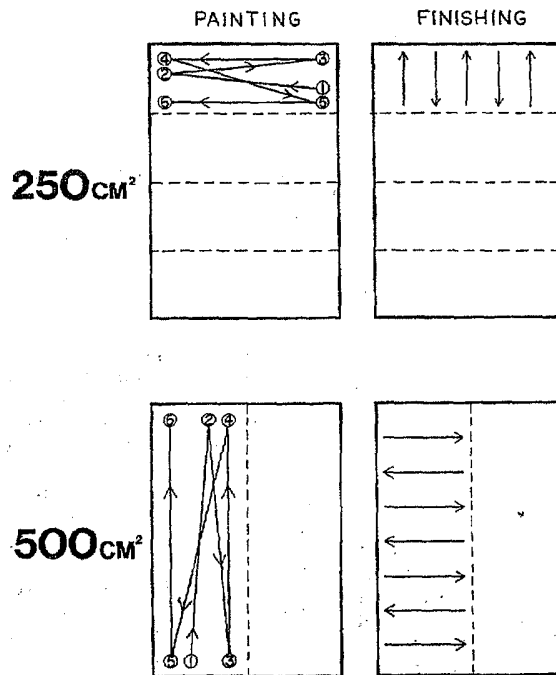


FIG. 1 Paint Application

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Standard Practice for Preparation of Paint-Roller Covers for Evaluation¹

This standard is issued under the fixed designation D 5069; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes the preparation or breaking-in of paint-roller covers for evaluation.

1.2 This practice is applicable to paint-roller covers having nap lengths up to $\frac{1}{2}$ in. (13 mm).

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Practice

2.1 Using the paint chosen for the evaluation, the paint-roller cover to be tested is repeatedly and generously loaded with paint and painted out over the same area of approximately 4 to 6 ft (0.4 to 0.6 m²) until it is saturated with paint, that is, the paint-roller cover cannot pick up any more paint, nor apply any more paint to the area being used for break-in.

3. Significance and Use

3.1 Until a paint-roller cover is saturated with paint, only part of the paint loaded onto the paint-roller cover can be transferred to a surface being painted. The remainder of the paint is absorbed into the fabric of the paint-roller cover. The amount of paint absorbed by a paint-roller cover is inversely proportional to the amount already present within the paint-roller cover. By saturation of the paint-roller cover before testing, quantitative inaccuracies of the amount of paint delivered to a surface are eliminated.

3.1.1 Using a saturated paint-roller cover enables the user to apply paint at controllable spreading rates.

3.1.2 Using a saturated paint-roller cover affords reproducibility when repeating a test.

4. Apparatus

4.1 *Paint Tray.*

4.2 *Paint Roller Frame*, of the same size as the paint-roller cover being prepared.

5. Materials

5.1 *Paint*, to be used in test.

5.2 *Primed or Painted Surface*, to be used for the roller cover break-in.

6. Procedure

6.1 Place the paint-roller cover on the frame.

6.2 Load the paint-roller cover with the paint from the tray by rolling the paint-roller cover over the surface of the tray so that just the nap is submersed. See Fig. 1.

6.3 Roll out the roller cover on the surface being used for break-in in an upward and downward motion in no larger an area than 2 ft (0.6 m) high by the width of the roller cover.

6.4 Reload the paint-roller cover with paint and roll out in the same manner over the same area. Do not increase the area except as necessary to control excess dripping of paint.

6.5 Repeat the above procedure as necessary until the following conditions are met:

6.5.1 Reloading the paint-roller cover does not result in increased paint pickup.

6.5.2 There is so much paint on the surface being used that the fully loaded paint-roller cover cannot transfer any more paint to the surface.

6.5.3 When the above conditions are met, there will be so much paint on the roller cover that constant movement (turning over) is necessary to prevent dripping, and the panel will show profuse sagging of the paint. About six roller-cover loadings are usually necessary to achieve this.

6.6 The roller cover is now prepared for immediate testing.

7. Keywords

7.1 break-in; paint roller; paint-roller cover; test preparation; roller

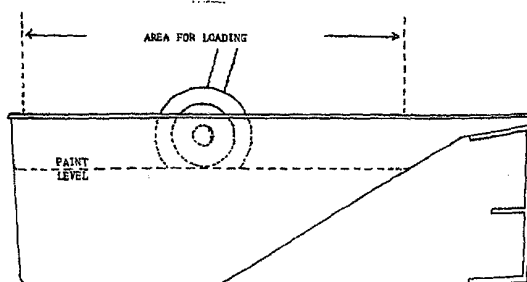


FIG. 1 Loading the Paint Roller

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.61 on Paint Application Tools.

Current edition approved May 25, 1990. Published July 1990.

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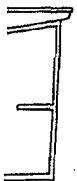
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Standard Test Method for Determining Amount of Volatile Organic Compound (VOC) Released from Solventborne Automotive Coatings and Available for Removal in a VOC Control Device (Abatement)¹

This standard is issued under the fixed designation D 5087; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the amount of volatile organic compound (VOC) released from applied solventborne automotive coatings that is available for delivery to a VOC control device. This is accomplished by measuring the weight loss of a freshly coated test panel subjected to solvent evaporation or baking in a laboratory simulation of the production process.

1.2 This test method is applicable to the VOC released from flashoff and baking operations after the paint has been applied.

1.3 This test method is applicable to solventborne automotive coating materials and is intended to represent or simulate the production process. The same general principles apply to waterborne coatings that contain volatile organic compounds (VOC), although specific procedural details are not presented herein to differentiate between VOC and water.

1.4 *This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- B 499 Test Method for Measurement of Coating Thickness by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals²
- D 1005 Test Methods for Measurement of Dry Film Thickness of Organic Coatings Using Micrometers³
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base³
- D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base³
- D 2369 Test Method for Volatile Content of Coatings³

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.2 on Chemical Analysis of Paints and Paint Materials.

Current edition approved July 15, 1991. Published September 1991. Originally published as D 5087 - 90. Last previous edition D 5087 - 90.

² Annual Book of ASTM Standards, Vol 02.05.

³ Annual Book of ASTM Standards, Vol 06.01.

E 145 Specification for Gravity-Convection and Forced Ventilation Ovens⁴

2.2 EPA Standard:

EPA Federal Reference Method 24—Determination of Volatile Matter Content, Water Content, Density, Volume Solids, and Weight Solids, of Surface Coatings, 40 CFR, Part 60, Appendix A⁵

EPA 450/3-88-018, PB89152276—Protocol for Determining the Daily Volatile Organic Compound Emission Rate of Automobile and Light Duty Truck Topcoat Operations⁶

3. Summary of Test Method

3.1 This test method simulates the loss of VOC from a freshly coated surface by using the difference in weight of a coated test panel before and after baking. Metal test panels are subjected to a simulation of the production coating process including application, flashing, and baking. The weight loss value obtained is then related to the volume of solids deposited on the test panel to compute the number of pounds of VOC per gallon (grams per litre) of solids applied that is available at the inlet of the VOC control device.

3.2 The information needed to determine the amount of VOC available for removal by the VOC control device is determined by identification of the points in the production process at which coated objects would enter flashoff/baking zones whose effluents are vented to the VOC control device.

4. Significance and Use

4.1 This test method provides basic engineering data that may be used to determine the amount of VOC available for delivery at the inlet of a VOC control device (particularly for the automobile industry).⁷ Typically, the procedure is useful for establishing the quantity of VOC that is evolved from the coating in the baking oven and available to be incinerated, although the same procedure can be followed for other forms of VOC abatement (that is, carbon, adsorption, etc.).

4.2 This test method may be adaptable for use directly in a production environment if all the critical factors (for

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁶ National Technical Information Services, 5285 Port Royal Rd. Springfield, VA 22151.

⁷ United States Environmental Protection Agency Protocol for Determining the Daily Volatile Organic Compound Emission Rate of Automobile and Light Duty Truck Topcoat Operations, Ref EPA 450/3-88-018 dated December 1988. This protocol provides for VOC emissions reduction credit for abatement processes.

expressed in pounds (grams) VOC per gallon (litre) solids applied:

11.1.1 The weight of coating solids deposited, W_s , is equal to the coated panel weight after full bake minus the panel weight before coating application as follows:

$$W_s = W_3 - W \quad (3)$$

or

$W_s = W_2 - W$ if W_2 represents a fully cured paint film.

where:

W_s = weight of coating solids deposited, lb (g),

W_2 = weight of panel at point which exhaust air is no longer vented to VOC control device,

W_3 = weight of panel with fully baked paint film, and

W = weight of initial unpainted test panel.

11.1.2 Determine the weight of VOC available for abatement control (W_A) by taking the difference in the weight of the panel representing the point in the process at which the exhaust air is vented to the control device (W_1) and the weight of the panel at the point where the exhaust air is no longer vented to the VOC control device (W_2) as follows:

$$W_A = W_1 - W_2 \quad (4)$$

where:

W_A = weight of VOC available for control, lb (g),

W_1 = weight of panel at point at which exhaust air is first vented to VOC control device, and

W_2 = weight of panel at point at which exhaust air is no longer vented to VOC control device.

11.1.3 Calculate the pounds VOC available for control per gallon of coating solids applied, C_L , by combining the weight of VOC available (W_A), weight of coating solids applied (W_s) and the coating solids density (D_{CS}) as follows:

$$C_L = \left(\frac{W_A}{W_s} \right) \times D_{CS} \quad (5)$$

where:

C_L = VOC loading lb/gal (g/L),

W_A = VOC available for removal, lb,

W_s = coating solids applied, lb, and

D_{CS} = coating solids density, lb/gal (g/L).

NOTE 2—Coating materials must be the specific formulations (as-applied condition) used in the process for which the VOC loading to the VOC control device, is to be determined.

12. Precision and Bias

12.1 *Precision*—The precision of the procedure in this test method is being determined.

12.2 *Bias*—Since there is no accepted reference procedure suitable for determining the bias for the procedure in this test method, no statement on bias is being made.

13. Keywords

13.1 automotive coatings; solvent release; VOC abatement

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Standard Test Method for Determination of the Nonvolatile Content in Silanes, Siloxanes and Silane-Siloxane Blends Used in Masonry Water Repellent Treatments¹

This standard is issued under the fixed designation D 5095; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method describes a procedure for the determination of the nonvolatile content (N_v) of silanes, siloxanes, and blended silane-siloxane materials used in masonry water repellent treatments and is applicable to both solvent- and water-borne materials.

1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see Section 7.

Referenced Documents

- 2.1 *ASTM Standards*:
D 1193 Specification for Reagent Water²
D 3980 Practice for Interlaboratory Testing of Paint and Related Materials³
E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens⁴

Summary of Test Method

3.1 A designated quantity of test material is weighed into a preweighed aluminum dish containing the catalyst solution, mixed, allowed to stand for 60 min at room temperature, and then heated in an oven at $110 \pm 5^\circ\text{C}$ for 60 min. The nonvolatile content of the test material is calculated by subtracting the solids of the catalyst solution from the total solids by weight of the test solution.

Significance and Use

4.1 This test method is used to determine the nonvolatile content of silanes, siloxanes, and silane-siloxane blended materials used in masonry water-repellent treatments. It can be used for the purpose of calculating the volatile organic compound (VOC) content of these materials under specified test conditions.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.47 on Masonry Treatments.

Current edition approved Sept. 15, 1991. Published November 1991. Originally published as D 5095 - 90. Last previous edition D 5095 - 90.

² *Annual Book of ASTM Standards*, Vols 6.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

5. Apparatus

5.1 *Aluminum Dishes*, 58-mm diameter by 18-mm high with a smooth (planar) bottom surface. Precondition the dishes for 30 min in an oven at $110 \pm 5^\circ\text{C}$ and store in a desiccator prior to use.

5.2 *Forced Draft Oven*, Type IIA or Type IIB as specified by Specification E 145.

5.3 *Syringes*, 1-mL and 5-mL.

5.4 *Analytical Balance*, capable of weighing to 0.1 mg.

6. Reagents

6.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.2 *p-Toluenesulfonic Acid*, monohydrate.⁵

6.3 *Alcohol*, technical grade ethanol or isopropanol.

7. Hazards

7.1 In addition to other precautions, provide adequate ventilation, consistent with accepted laboratory practice, to limit the accumulation of solvent vapors.

8. Procedure

8.1 *Catalyst Solution*—Prepare a catalyst solution containing a mixture of 0.5 % *p*-Toluenesulfonic acid in either ethanol or isopropanol. Thoroughly mix the solution. Prepare sufficient catalyst solution to perform all tests.

8.1.1 The nonvolatile content of the test material can be calculated only if the same catalyst solution is used throughout the test. Each time a new batch of catalyst solution is used, its nonvolatile content must be determined.

8.2 Determine the nonvolatile matter, in triplicate, of the catalyst solution as follows:

8.2.1 Weigh an aluminum dish to 0.1 mg. Record the weight as W_1 .

8.2.2 Using a 5-mL syringe, weigh 3 ± 1.0 g, to 0.1 mg, by difference, of the catalyst solution into the preweighed aluminum dish. Record the weight of catalyst solution as W_2 .

8.2.3 Heat the aluminum dish containing the catalyst solution in a forced draft oven for 60 min at $110 \pm 5^\circ\text{C}$.

8.2.4 Remove the dish from the oven and immediately place in a desiccator. Seal the desiccator and allow the dish to cool to ambient temperature.

⁵ Solutions of *p*-Toluenesulfonic acid in isopropanol may be obtained from King Industries, Science Road, Norwalk, CT 06852.

8.2.5 Reweigh the dish to 0.1 mg. Record the weight as W_2 .

8.2.6 Calculate the nonvolatile matter of the catalyst solution, N_c , in accordance with 9.1.

8.3 Determine the percent nonvolatile content, in triplicate, of the test specimen as follows:

8.3.1 Thoroughly mix the test materials before use.

8.3.2 Weigh an aluminum dish to 0.1 mg. Record the weight as W_4 .

8.3.3 Using a 5-mL syringe, weigh 3 ± 1.0 g, to 0.1 mg, by difference, of the catalyst solution into the preweighed aluminum dish. Record the weight as W_6 .

8.3.4 Using a 1-mL syringe, weigh 1.0 ± 0.1 g, to 0.1 mg, by difference, of the test specimen into the weighing dish containing the catalyst solution. Record the weight of the test specimen as S .

8.3.4.1 Weighings must be done quickly to limit weight loss due to volatilization. If there is insufficient moisture present when testing solvent-borne silane materials, it is advisable to add up to 0.3 g of reagent grade water to the dish containing the mixture of catalyst and test solutions.

8.3.5 Gently swirl the dish to mix the materials. Allow the materials to stand at room temperature for 60 min.

8.3.6 Heat the dish containing the mixture of catalyst and test solutions in a forced-draft oven for 60 min at $110 \pm 5^\circ\text{C}$.

8.3.7 Remove the dish from the oven and immediately place in a desiccator. Seal the desiccator and allow the dish to cool to ambient temperature.

8.3.8 Reweigh the aluminum dish to 0.1 mg and record the weight as W_5 .

8.3.9 Calculate the nonvolatile content, N_s , of the test specimen in accordance with 9.2.

9. Calculation

9.1 Calculate the nonvolatile matter, N_c , in the catalyst solution as follows:

$$N_c = (W_2 - W_1)/W_3 \quad (1)$$

where:

N_c = nonvolatile matter of catalyst solution expressed as a decimal fraction,

W_1 = weight of aluminum dish, g,

W_2 = weight of dish plus catalyst solution after heating, g, and

W_3 = weight of catalyst solution before heating, g.

9.2 Calculate the nonvolatile content, N_s , in the test specimen as follows:

$$N_s = 100 \frac{[(W_5 - W_4) - (W_6)(N_c)]}{S}$$

where:

N_s = nonvolatile content of test specimen, percent,

W_4 = weight of the aluminum dish, g,

W_5 = weight of aluminum dish plus the catalyzed material after heating, g,

W_6 = weight of catalyst solution used in test specimen before heating, g,

N_c = nonvolatile matter of catalyst solution, decimal fraction, (average of two determinations), and

S = weight of test specimen before heating, g.

10. Report

10.1 Report the following information:

10.1.1 The average values obtained for the nonvolatile content of the catalyst solution, N_c .

10.1.2 The average values obtained for the percent nonvolatile content of the test specimen, N_s .

11. Precision and Bias

11.1 The precision estimated for this test method is based on an interlaboratory study in which one operator in each of five laboratories tested in triplicate on two different days four water repellent materials containing between 14 to 65% nonvolatiles. The results were analyzed statistically in accordance with Practice D 3980. The intralaboratory standard deviation was found to be 0.283% absolute with 17 df and the interlaboratory coefficient of variation 7.02% relative with 16 df. Based on the standard deviation and coefficient of variation, the following criteria should be used for judging at the 95% confidence level, the acceptability of results.

11.1.1 *Repeatability*—Two results, each the mean of triplicates, obtained by the same operator should be considered suspect if they differ by more than 0.84% absolute.

11.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 7.02% relative.

11.2 *Bias*—Bias has not been established for this test method.

12. Keywords

12.1 masonry water repellents; nonvolatile matter content; silanes; siloxanes

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Standard Specification for Artists' Acrylic Emulsion Paints¹

This standard is issued under the fixed designation D 5098; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This specification establishes requirements for composition, physical properties, performance, and labeling of artists' acrylic emulsion paints.

1.2 This specification covers pigments, vehicles, and additives. Requirements are included for pigment identification, lightfastness, consistency, and drying time.

1.3 Table 1 lists some pigments meeting the lightfastness requirements in this specification. In order to identify other pigments that meet these requirements, instructions are given for test specimen preparation. Test methods for determining relative lightfastness are referenced.

1.4 The values stated in inch-pound units are to be regarded as standard. The SI units given in parentheses are for information only.

Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 476 Specification for Titanium Dioxide Pigments²
- D 602 Specification for Barium Sulfate Pigments²
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems³
- D 1640 Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature³
- D 3168 Practice for Qualitative Identification of Polymers in Emulsion Paints³
- D 4236 Practice for Labeling Art Materials for Chronic Health Hazards³
- D 4303 Test Methods for Lightfastness of Pigments Used in Artists' Paints³
- D 4838 Test Method for Determining the Relative Tinting Strength of Chromatic Paints⁴
- E 284 Terminology of Appearance⁵

Terminology

3.1 Definitions:

3.1.1 *colour index name*—consists of the category (type of color or pigment), general hue, and an assigned number given

to a colorant in the Colour Index⁶ as an international identification system.

3.1.1.1 *Discussion*—For example, the Colour Index Name of one phthalocyanine blue pigment is Pigment Blue 15 (PB 15).

3.1.2 *Colour Index Number*—a five-digit number given in the Colour Index that describes the chemical constitution of a colorant.

3.1.2.1 *Discussion*—For example, the Colour Index Number of one phthalocyanine blue pigment is 74160.

3.1.3 Appearance terms used in this specification are defined in Terminology E 284.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *acrylic emulsion paint*—paint containing a stable aqueous dispersion of polymers or copolymers of acrylic acid, methacrylic acid, esters of these acids, or acrylonitrile; sometimes termed latex, acrylic latex, or polymer emulsion paint.

3.2.2 *glycols*—general term for dihydric alcohols used to provide freeze-thaw stability in acrylic and other water-based vehicle systems.

4. Significance and Use

4.1 This specification establishes quality requirements and provides a basis for common understanding among producers, distributors, and users.

4.2 It is not intended that all paints meeting the requirements be identical nor of uniform excellence in all respects. Variations in manufacture, not covered by this specification, may cause some artists to prefer one brand over another, either of which may be acceptable under this specification.

5. Labeling Requirements

5.1 Pigment(s) Identification:

5.1.1 Every label shall include for each pigment contained in the paint the information underlined in Table 1 which includes the Common Name, Colour Index Name, and any additional terms necessary to identify the form of the pigment.

5.1.2 The complete pigment identification given in Table 1, which also includes the Colour Index Number and a simple chemical description, shall be given in an appropriate producer publication. Manufacturers are encouraged to put this complete identification on the container label when label size permits.

5.1.3 The Common Name shall be placed on the front of the label and shall be the name of the paint except as

¹ This specification is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.57 on Artist Paints and Related Materials.

Current edition approved July 27, 1990. Published November 1990.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 06.03.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Colour Index, 3rd ed., 5 Vols and Revisions. The Society of Dyers and Colourists, London, 1971-75. Available from the American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

TABLE 1 Suitable Pigment List

Note—Underlined information and the lightfastness rating in the table shall be included on every label.

Key:

Lightfastness Category:

Lightfastness I Excellent Lightfastness

Lightfastness II Very Good Lightfastness

Abbreviations used in Colour Index Names:

PB Pigment Blue
PBk Pigment Black
PBr Pigment Brown
PG Pigment Green
PO Pigment Orange
PR Pigment Red
PV Pigment Violet
PW Pigment White
PY Pigment Yellow

Pigment Notations:

(CC) Concentrated cadmium pigments may contain up to 15 % barium sulfate for color control. Cadmium-barium pigments contain a much higher amount of barium sulfate.
(DL) May darken in strong light
(LF) Lightfast type
(NA) Colour Index name or number not assigned
(RS) Red shade
(SM) Sensitive to moisture in direct sunlight
(SS) Sensitive to hydrogen sulfide

Colour Index Name	Lightfastness Category	Common Name and Chemical Class	Colour Index Number
	Acrylic		
YELLOWS			
PY 1, PY 1.1	II	Arylide Yellow G, with option of adding the name Hansa Yellow Medium, arylide yellow	11680
PY 3	II	Arylide Yellow 10G, with option of adding the name Hansa Yellow Light, arylide yellow	11710
PY 35	I	Cadmium Yellow Light, concentrated cadmium zinc sulfide (CC) (SM)	77205
PY 35:1	I	Cadmium-Barium Yellow Light, cadmium zinc sulfide coprecipitated with barium sulfate (SM)	77205:1
PY 37	I	Cadmium Yellow Medium or Deep, concentrated cadmium sulfide (CC) (SM)	77199
PY 37:1	I	Cadmium-Barium Yellow Medium or Deep, cadmium sulfide coprecipitated with barium sulfate (SM)	77199:1
PY 42	I	Mars Yellow, with option of adding the name Yellow Iron Oxide, synthetic hydrated iron oxide	77492
PY 42	I	Mars Orange, synthetic hydrated iron oxide	77492
PY 43	I	Yellow Ochre, natural hydrated iron oxide	77492
PY 53	I	Nickel Titanate Yellow, oxides of nickel, antimony and titanium	77788
PY 65	I	Arylide Yellow RN, with option of adding the name Hansa Yellow RN, arylide yellow	11740
PY 73	I	Arylide Yellow GX, with option of adding the name Hansa Yellow GX, arylide yellow	11738
PY 74 (LF)	I	Arylide Yellow 5GX, with option of adding the name Hansa Yellow 5GX, arylide yellow	11741
PY 83 (HR70)	I	Diarylide Yellow HR70, diarylide yellow	21108
PY 97	I	Arylide Yellow FGL, arylide yellow	11767
PY 98	I	Arylide Yellow 10GX, with option of adding the name Hansa Yellow 10GX, arylide yellow	11727
PY 108	I	Anthrapyrimidine Yellow, anthrapyrimidine	68420
PY 109	I	Isindolinone Yellow G, tetrachloroisindolinone	NA
PY 110	I	Isindolinone Yellow R, tetrachloroisindolinone	56280
PY 112	I	Flavanthrone Yellow, flavanthrone	70600
PY 138	I	Quinophthalone Yellow, quinophthalone	NA
PY 139	I	Isindoline Yellow, isindoline	NA
PY 150	I	Nickel Azo Yellow, nickel complex azo	NA
PY 151	I	Benzimidazolone Yellow H4G, benzimidazolone	13880
PY 153	I	Nickel Dioxine Yellow, dioxine yellow nickel complex	NA
PY 154	I	Benzimidazolone Yellow H3G, benzimidazolone	11781
PY 175	I	Benzimidazolone Yellow H6G, benzimidazolone	11784
ORANGES			
PO 5	II	Dinitraniline Orange, dinitraniline (SM)	12075
PO 20	I	Cadmium Orange, concentrated cadmium sulfo-selenide (CC)	77202
PO 20:1	I	Cadmium-Barium Orange, cadmium sulfo-selenide coprecipitated with barium sulfate	77202:1
PO 23	I	Cadmium Vermilion Orange, concentrated cadmium mercury sulfide (CC)	77201
PO 23:1	I	Cadmium-Barium Vermilion Orange, cadmium mercury sulfide coprecipitated with barium sulfate	77201:1
PO 36	I	Benzimidazolone Orange HL, benzimidazolone	11780
PO 43	I	Perinone Orange, perinone	71105
PO 48	I	Quinacridone Gold, quinacridone	NA
PO 49	I	Quinacridone Deep Gold, quinacridone	NA
PO 80	I	Benzimidazolone Orange HGL, benzimidazolone	11782
PO 62	I	Benzimidazolone Orange H5G, monoacetolone	11775
REDS			
PR 5	II	Naphthol ITR, naphthol ITR	12490
PR 7	I	Naphthol AS-TR, naphthol AS-TR	12420
PR 9	I	Naphthol AS-OL, naphthol AS-OL	12460
PR 14	II	Naphthol AS-D, naphthol AS-D	12380

TABLE 1 *Continued*

Index Name	Lightfastness Category Acrylic	Common Name and Chemical Class	Colour Index Number
REDS (cont'd)			
MRS ^A	I	Thioindigold Violet, thioindigold	73312
	I	Indian Red, synthetic red iron oxide (bluish hue)	77491
	I	Light or English Red Oxide, synthetic red iron oxide (yellowish hue)	77491
	I	Mars Red, with option of adding the name Red Iron Oxide, synthetic red iron oxide	77491
	I	Mars Violet, with option of adding the name Violet Iron Oxide, synthetic iron oxide (violet hue)	77015
	I	Venetian Red, synthetic iron oxide (yellowish hue)	77491
	I	Light Red, calcined yellow ochre	77492
	I	Vermilion, mercuric sulfide (DL)	77766
	I	Cadmium Red Light, Medium, or Deep, concentrated cadmium-seleno sulfide (CC)	77202.1
	I	Cadmium-Barium Red Light, Medium, or Deep, cadmium seleno-sulfide coprecipitated with barium sulfate	77202.1
	II	Naphthol AS-D, naphthol AS-D	12370
	I	Cadmium Vermillion Red Light, Medium or Deep, concentrated cadmium mercury sulfide (CC)	77201
	I	Cadmium-Barium Vermillion Red Light, Medium or Deep, cadmium mercury sulfide coprecipitated with barium sulfate	77201.1
	I	Naphthol Red, naphthol	NA
	I	Quinacridone Magenta, γ quinacridone	73915
	II	Perylene Vermillion, perylene	71145
	I	Perylene Red, perylene	77137
	I	Brominated Anthranthrone, brominated anthranthrone	58300
F3RK-70	I	Naphthol Red, naphthol carbanilide	12475
F5RK	II	Naphthol Crimson, naphthol carbanilide	12475
	I	Benzimidazolone Maroon, benzimidazolone	71513
	I	Perylene Maroon, perylene	71130
	I	Thioindigold Magenta, thioindigold	73360
	I	Naphthol AS, naphthol AS	12467
	I	Perylene Red, perylene	71140
	I	Quinacridone Red, γ quinacridone red	NA
	I	Pennone Red Deep, pennone	71100
	I	Quinacridone Scarlet, quinacridone red	NA
	I	Quinacridone Yellow Red, quinacridone red γ	73902
	I	Quinacridone Red, γ quinacridone red	73900
PURPLES			
	I	Ultramarine Red, complex silicate of sodium and aluminum with sulfur	77007
	I	Ultramarine Violet, complex silicate of sodium and aluminum with sulfur	77007
	I	Quinacridone Violet, quinacridone violet b	73900
23 (RS)	II	Dioxazine Purple, carbazole dioxazine	51319
	I	Isoviolanthrone Violet, isoviolanthrone	60010
BLUES			
	I	Phthalocyanine Blue, copper phthalocyanine	74160
	I	Phthalocyanine Blue, metal free phthalocyanine	74100
	I	Indanthrone Blue, indanthrone	69810
	I	Cobalt Blue, oxides of cobalt and aluminum	77346
	I	Ultramarine Blue, complex silicate of sodium and aluminum with sulfur	77007
	I	Manganese Blue, barium manganate with barium sulfate	77112
	I	Cerulean Blue, oxides of cobalt and tin	77368
	I	Cerulean Blue, Chromium, oxides of cobalt and chromium	77343
	I	Indanthrone Blue, indanthrone	69800
GREENS			
	I	Phthalocyanine Green, chlorinated copper phthalocyanine	74260
	I	Green Gold, with option of adding the name Nickel Azo Yellow, nickel chelated azo	12775
	I	Chromium Oxide Green, anhydrous chromium sesquioxide	77288
	I	Cobalt Green, oxides of cobalt and zinc	77335
	I	Green Earth or Terra Verde, natural green	77009
	I	Phthalocyanine Green, chlorinated and brominated phthalocyanine	74265
	I	Light Green Oxide, oxides of nickel, cobalt, and titanium	77377
BROWNS			
	I	Mars Brown, with option of adding the name Brown Iron Oxide, synthetic brown iron oxide or mixtures of synthetic iron oxides	77499
	I	Burnt Sienna, calcined natural iron oxide	77492
	I	Burnt Umber, calcined natural iron oxide containing manganese	77492
	I	Raw Sienna, natural iron oxide	77492
	I	Raw Umber, natural iron oxide containing manganese	77492

TABLE 1 Continued

Colour Index Name	Lightfastness Category	Common Name and Chemical Class	Colour Index Number
	Acrylic		
BLACKS			
PBk 6	I	Lamp Black, nearly pure amorphous carbon	77266
PBk 7	I	Carbon Black, nearly pure amorphous carbon	77266
PBk 9	I	Ivory Black, amorphous carbon produced by charring animal bones	77267
PBk 11	I	Mars Black, with option of adding the name Black Iron Oxide, synthetic black iron oxide	77499
WHITES			
PW 4	I	Zinc White, zinc oxide	77947
PW 6	I	Titanium White, titanium dioxide (rutile or anatase) with option of including some barium sulfate or zinc oxide	77891

* Applies only to Permanent Red Violet MRS, product of American Hoechst Corp., Coventry, RI 02616. Pigments described as thioindigoids have varying degrees of lightfastness.

described in 5.1.5 and 5.1.6. Other identification may be placed elsewhere on the container.

5.1.4 The Colour Index Name may be spelled out in full or abbreviated depending on the size of the label. Example: Pigment Blue 15, or Pig. Blue 15 or PB 15.

5.1.5 *Substituted Pigments*—In the case of substituted pigments, the word "Hue" in equal size letters shall follow in the title, on the front of the tube, immediately after the name of the pigment that has been simulated. Directly below the title, the Common Name of the significant pigment used shall be given in letters no less than the next type size smaller than the title. For example:

COBALT BLUE HUE
(ULTRAMARINE BLUE).

5.1.6 Proprietary names or optional names may be used provided the Common Name(s) given in Table 1 appears on the front of the label directly under the proprietary or optional name in letters no less than the next type size smaller than the proprietary or optional name.

5.1.7 *Mixed Pigments*—Artists' paints containing more than one colorant comply with this specification if all colored pigments used are on the suitable pigment list (Table 1) and provided the mixture itself has passed all other test requirements in this specification. The lightfastness category shall be that of the least lightfast pigment. This lightfastness category may be changed if these paints are tested for lightfastness in accordance with Test Methods D 4303 and results indicating a different category are submitted to ASTM Subcommittee D01.57 for evaluation.

5.2 Provide on the label identification of polymer used in the paint.

NOTE 1—The type of polymer can be identified by using Practice D 3168.

5.3 *Lightfastness*—The label shall contain the word "Lightfastness" followed by the appropriate rating, I or II, as given for each pigment in Table 1.

5.3.1 Lightfastness I pigments, when made into paint specimens as described in Section 7 and exposed, tested, and rated in accordance with Test Method D 4303, shall have a color difference (ΔE^*_{ab}) of 4 or less CIELAB units between the specimens measured before and after exposure.

5.3.2 Lightfastness II pigments, when made into paint specimens as described in Section 7 and exposed, tested, and rated in accordance with Test Methods D 4303, shall have a color difference (ΔE^*_{ab}) of more than 4.0 but not more than

8.0 CIELAB units between the specimens measured before and after exposure.

5.3.3 Pigments were placed in a lightfastness category on the basis of either known historical performance in art works or the ratings from four lightfastness tests conducted as described in Test Methods D 4303. Results from further tests on these, or other pigments, are solicited by ASTM Subcommittee D01.57.

5.3.3.1 The lightfastness category of a pigment shall be changed if results from several further tests conducted in accordance with Test Methods D 4303 and approved by ASTM Subcommittee D01.57, establish a different lightfastness category than the one given in Table 1.

5.3.3.2 Additional pigments shall be placed in Table 1 after they have been tested for lightfastness in accordance with Test Methods D 4303 and the test results submitted to ASTM Subcommittee D01.57 for evaluation, provided the results demonstrate that the pigments have the lightfastness ratings required for Lightfastness I or Lightfastness II, as described in 5.3.1 and 5.3.2.

5.3.4 For information and to establish nomenclature, pigments in Lightfastness III category are given in Table X1.1 in Appendix X1, but are not to be used in paint conforming to this specification. These pigments have a color difference before and after exposure of more than 8.0 but not more than 16.0 CIELAB units.

5.4 *Toxicity*—All products and labeling must conform to the Federal Hazardous Substances Act and to Practice D 4236.

5.5 *Statement of Conformance*—"Conforms to ASTM Specification D 5098," or "Conforms to ASTM D 5098," or "Conforms to the quality requirements of ASTM D 5098." This statement may be combined with other conformance statements, such as, "Conforms to the quality and health requirements of ASTM Specification D 5098 and Practice D 4236."

5.6 *Address*—Include on the label (1) the name and address of the manufacturer or importer and (2) the country of manufacture.

6. Quality Assurance for Artists' Acrylic Emulsion Paints

6.1 *Conditions not Covered in This Specification that Affect the Quality of Artists' Acrylic Emulsion Paints:*

6.1.1 *Substrate*—Factors such as the texture, gloss, effective pH, porosity, chemical composition, and condition of the substrate will affect gloss, gloss uniformity, drying time,

hesion, and the flexibility of the dried film.

6.1.2 *Environmental Conditions*—Factors such as temperature, humidity, air flow, and light conditions affect application properties, film formation, drying time, and adhesion.

6.1.3 *Storage*—Factors such as aging, and high and low temperatures may cause changes in consistency.

6.2 *Vehicles*—Only acrylic polymer emulsions or acrylic polymer emulsions may be used (see Note 1).

6.3 *Pigments*—The pigments shall be limited to those recommended for use in acrylic emulsion paints in the list of pigments in Table 1. Their lightfastness rating shall be the numeral given in the same row.

6.4 *Additives*—Surfactants, preservatives, defoamers, glycol solvents, and thickeners may be used to achieve aging stability, to control foaming, to ensure freeze-thaw stability of film coalescence, and to obtain a desired consistency.

6.5 *Inerts*—Inerts shall be included only to adjust product gloss or sheen, or to produce desirable working qualities.

6.6 *Preparation of Sample*—Empty the contents of a previously unopened container onto a glass slab and mix thoroughly with a spatula to a homogeneous sample.

6.7 *Coarse Particles*—Paints shall be free of oversize particles and shall form a uniform film. The maximum content of coarse particles shall be 1 weight % as determined by Test Methods D 185.

6.8 *Fineness of Dispersion*—Determine the fineness of dispersion by Test Method D 1210. For paste paint: on a glass plate, using a spatula, mix the paint with an equal volume of water until homogeneous. The maximum allowable grind reading is 1.5 mils (40 μ m).

6.9 *Consistency*—Paints shall be smooth and creamy. The paste type of paint shall not flow or level when applied with a palette knife.

6.10 *Freeze-Thaw Stability*—Using a freezer that has a temperature of 20°F (−7°C) or lower, subject the paint to five freeze-thaw cycles. A freeze-thaw cycle shall consist of freezing the paint to a solid state (minimum of 18 h) and then thawing the paint to room temperature (minimum of 5 h). The paint shall then meet the requirements of 6.7, 6.8, and 6.9.

6.11 *Drying*—Use a 6-mil (150- μ m) clearance film applicator to make a uniform drawdown on a lacquer-sealed panel. At a relative humidity of 50 to 75 % and a temperature of 65 to 80°F (18 to 27°C), the dust-free drying time, determined in accordance with Test Method D 1640, shall be not less than 10 min.

6.12 *Tinting strength requirements* will be included in this specification as appropriate tinting strength standards for individual pigments are established. Test Method D 387 may be used to determine the tinting strength of pigments or tints when all ingredients are known. Test Method D 4838 may be used to determine the relative tinting strength of aromatic paints containing a single pigment and the same vehicle but where other ingredients are unknown.

Lightfastness Determination

7.1 If a pigment is not listed in Table 1, test specimens of paint containing the pigment shall be prepared. These test specimens shall be tested in conformance with the requirements for exposure and evaluation given in Test Methods D 4303.

NOTE 2—A report of the results of these tests may be submitted to ASTM Subcommittee D01.57 for inclusion of the pigments in Table 1. The report shall include information on test conditions, instruments used, and be accompanied by the test specimens, which will be returned.

7.2 Materials:

7.2.1 *Aluminum Exposure Panels*,⁷ 3 by 6 in. (75 by 150 mm).

7.2.2 *Posterboard*, lightweight, approximately 20 mils (0.5 mm) thick, having a glossy finish on one side.

7.2.3 *Titanium Dioxide Acrylic Emulsion Paint*.

7.2.4 *Spray Apparatus*, for applying ground coats.

7.2.5 *Paint Brush*, 2.5 in. (60 mm) wide for applying ground coats if spray apparatus is not available.

7.2.6 *Soft Hair Brush*, 1 in. (25 mm) wide for applying ground coats to aluminum panels. An oxhair artist's "stroke" brush is suitable.

7.3 Specimen Preparation:

7.3.1 Ground Coats:

7.3.1.1 For the first ground coat under acrylic emulsion paints, prepare the following enamel:

	Weight %
Medium oil length soya alkyd, 50 % nonvolatile ⁸	20
Rutile titanium dioxide ⁹ (conforming to Type II of Specification D 476)	40
Blanc fixe (conforming to Specification D 602)	40
Driers: 0.15 % zinc and 0.15 % zirconium as metal on the alkyd nonvolatile	
Sufficient mineral spirits for milling	

7.3.1.2 Mill to a Hegman fineness of 7 as measured by Test Method D 1210. Thin with mineral spirits to appropriate viscosity for spraying or for flow coating by brush.

7.3.2 For the second ground coat for acrylic emulsion paints, prepare the following enamel:

	Weight %
Medium oil length soya alkyd, 50 % nonvolatile ⁸	40
Rutile titanium dioxide ⁹ (conforming to Type II of Specification D 476)	30
Colloidal silica ¹⁰	30
Driers: 0.15 % zinc and 0.15 % zirconium as metal on the alkyd nonvolatile	
Sufficient mineral spirits for milling	

7.3.2.1 The colloidal silica may be dispersed separately in mineral spirits using a high-speed mixer and added to the milled alkyd-titanium pigment paste. Mill to a Hegman fineness of 7 as measured by Test Method D 1210. Use sufficient mineral spirit for proper application.

NOTE 3—These soya alkyd enamels are used for the ground coats because of their color stability, nonabsorbency, adhesion of the specimen coats under humid conditions, and freedom from blistering that can occur with an acrylic ground coat under high humidity.

⁷ The No. A-36 aluminum panel manufactured by Q Panel Co., 26200 First St., Cleveland, OH 44145, has been found satisfactory for this purpose.

⁸ The following alkyd resins have been found satisfactory for this purpose: Cargill, No. 5196; JMC McWhorter, DURAMAC 2419; Reichhold, No. 11-035; and Spencer-Kellog, AROPLAZ 1082 M50.

⁹ DuPont R900 rutile titanium dioxide, available from E.I. du Pont de Nemours & Co., 1007 Market St., Wilmington, DE 19898, has been found satisfactory for this purpose.

¹⁰ Syloid 308, available from Davison Chemical, Div. W.R. Grace Co., PO Box 2117; Baltimore, MD 21203, has been found satisfactory for this purpose.

7.3.3 Application of Ground Coats:

7.3.3.1 Degrease aluminum substrates before applying the ground coat.

7.3.3.2 Apply one coat of the enamel described in 7.3.1.1, followed by one coat of the enamel described in 7.3.2. To flow coat the aluminum panels for sun exposure use a 1-in. (25-mm) soft hair brush. Coat the posterboard to be used for laboratory exposure on the less absorbent, glossy side.

NOTE 4—Coating the posterboard is most conveniently done on the whole sheet before cutting to size.

7.3.3.3 Allow a minimum of five days drying time following the first coat and two weeks or more after applying the second coat and before applying the paint to be tested.

7.4 Mixing Whites for Dilution of Colors:

7.4.1 Use a white containing the same emulsion and additives as the formulation in which the pigment is incorporated if possible.¹¹ The following basic composition has been found satisfactory:

	Weight %
Acrylic emulsion nonvolatile	23 to 24
Rutile titanium dioxide ⁹ (conforming to Type II of Specification D 476)	34.6

7.4.1.1 Keep the viscosity of the mixing white to 250 P or slightly lower.

7.5 Preparation of Test Paints:

7.5.1 The pigment to be tested may be milled in a compatible paste for acrylic emulsion. If a prepared artists' paint of known composition is available, it may be used for this test instead of preparing a dispersion.

7.5.2 Dilute the pigment paste or paint with the white containing the same type of vehicle until the spectrophotometric measurement of the dried film shows 35 to 45 % relative reflectance at the wavelength of maximum absorption for that pigment. The wavelength of maximum absorption is located at the point of lowest reflectance on the spectral curve between 420 and 620 nm. If using a tristimulus filter colorimeter, the lowest of the three filter readings is the region of maximum absorption and the dilution should be adjusted so that a reading of 35 to 45 % reflectance is obtained with this filter. The diffuse white reference standard for all measurements should have an absolute reflectance between 97 and 100 %.

7.5.2.1 To obtain this reflectance, use the Kubelka-Munk

Single Constant Method described in Appendix X2 or use a trial and error method.

7.5.3 Make instrumental readings with the specular reflectance included to minimize the effect on readings of any change in gloss and to minimize the effect of brush strokes.

7.5.4 Use an applicator with a 6-mil (150- μ m) aperture to make a minimum size drawdown, 1½ by 1½ in. (40 by 40 mm), or other minimum size appropriate for the viewing area of the instrument used. Return all recoverable paint to the batch to allow for repeat mixes and measurements.

7.5.5 For acrylic emulsion paints, 80 g of white paint is needed since paint used in the drawdown is lost.

7.5.6 For the initial weights of white stated in 7.5.4 and 7.5.5, the weighing must be accurate to 0.05 g.

7.6 Application of Paints to Panels:

7.6.1 Prepare four specimen panels on appropriate substrates for each pigment under test. Two are used in the first lightfastness tests and two are retained in subdued light, one for visual comparisons with the exposed panels and one in case a third test is needed to supplement results from the first two tests, as described in Test Methods D 4303.

7.6.2 Apply the test paints by brush to the aluminum panel if exposure is to be under glass to the sun or by brush to the posterboard panel if exposure is to be to laboratory apparatus.

7.6.3 Using the No. 12 artist's flat bristle brush, brush the panels lengthwise, then crosswise, and again lengthwise, this time with a light touch to produce a film as smooth as possible. The mixing whites must be fluid enough to facilitate leveling, but not have excess vehicle that can affect the test. Do not use thinners. Apply two coats to all specimen panels to achieve complete opacity.

7.6.4 Allow specimens to dry between coats as described in the Procedure section (Dry-Through or Dry-to-Handle Time) of Test Methods D 1640. After recoating, allow specimens to dry hard, as described in the Procedure section (Dry-Hard Time) of Test Methods D 1640, before measuring them prior to exposure. Acrylic paints can be recoated the next day and read two days after recoating.

8. Exposure

8.1 Conduct exposure tests, calculate mean color difference, and assign pigments to lightfastness categories as described in Test Methods D 4303.

9. Keywords

9.1 artists' acrylic paints; lightfastness; quality requirements; test specimens

¹¹ If the acrylic vehicle composition of the paint is unknown, Rhoplex AC-234, from Rohm & Haas Co., Independence Mall W., Philadelphia, PA 19105, is compatible with most acrylic emulsion paints.

APPENDIXES

(Nonmandatory Information)

X1. LIGHTFASTNESS III

The pigments in Table X1.1 are not sufficiently fast to be used in paints that conform to this specification. These pigments are listed here solely to establish

common terminology. They may be satisfactory when used full strength (without dilution) or with extra protection from exposure to light.

TABLE X1.1 Lightfastness III

Colour Index Name	Lightfastness Category	Common Name and Chemical Class	Colour Index Number
PR 17	III	Naphthol Red AS-D, naphthol	12390
PR 146	III	Naphthol Red, naphtholic arylide	12485
PY 23BS	III	Dioxazine Purple, carbazole dioxazine	51319
PG 8	III	Hooker's Green, ferric-nitroso-beta-naphthol	10006

X2. KUBELKA-MUNK SINGLE CONSTANT METHOD FOR PREPARING PAINT FILMS $40 \pm 5\%$ REFLECTANCE

X2.1 Prepare a mixture of white and colorant in proportion estimated to give a paint having a reflectance of 30 to 40 % at the wavelength of maximum absorption. Calculate the concentration of colorant in white in this batch as follows:

$$C_c = W_c/W_w + W_c$$

where:
 C_c = concentration of colorant, and
 W_c = weight of white and colorant, respectively, used in the batch.

Now or brush a film of this paint over contrast paper at a thickness sufficient to give complete visual hiding.

X2.1.1 After allowing the film to dry, measure the reflectance of this film at the wavelength of maximum absorption with a spectrophotometer or, if using a colorimeter, the reflectance with the filter that gives the lowest reading (see Note X2.1). Calculate the Kubelka-Munk Single Constant value of this reflectance as follows:

$$(K/S)_m = [1 - (R_m - 0.04)]^2 / 2(R_m - 0.04)$$

where:
 $(K/S)_m$ = Kubelka-Munk Value,
 R_m = reflectance measurement of the mixture, made with the specular component included expressed as a decimal value, and
 0.04 = decimal value correction for surface reflectance.

NOTE X2.1—To speed drying, accelerated methods may be used. It is recommended that water-based acrylic paints be warmed to 50 to 55°C for 2 h after 1 h of air drying before measuring the reflectance. Placing drawdowns of oil paints near fluorescent lamps, for example in the fluorescent light exposure racks described in Annex A1 of Test Methods D303, will speed their drying time.

X2.1.2 Calculate the Kubelka-Munk absorption coefficient for unit concentration as follows:

$$K_c = (K/S)_m / C_c$$

where K_c = unit absorption coefficient.

X2.2 Based on the results of the trial mixture, calculate the concentration of colorant in white required to give the

desired 35 to 45 % reflectance at the wavelength of maximum absorption as follows:

$$C_d = (K/S)_{40\%} / K_c = 0.4500 / K_c$$

where C_d = concentration of colorant in white predicted to give a paint with the desired 35 to 45 % reflectance value.

X2.3 Adjust the concentration of the trial batch so that the concentration of colorant equals C_d . Determine the changes in colorant concentration, ΔC as follows:

$$\Delta C = C_d - C_c$$

These changes are positive if the trial batch has a reflectance greater than 40 % and negative if the reflectance is less than 40 %. If ΔC is positive, add more colorant to the trial batch to bring the concentration of colorant to C_d . If ΔC is negative, add more white to bring the concentration of colorant to C_d . Because it may be necessary to add large amounts of white to correct for concentrations of colorant giving reflectance values far below 40 %, it is usually best to prepare a trial batch expected to give a ΔC value that is moderately positive or only slightly negative.

X2.3.1 For the case where ΔC is positive, the weight of colorant needed to prepare a second batch is,

$$\Delta W_c = W_c - C_d(W_w + W_c) / C_d - 1 \times (L.F.)$$

where:

ΔW_c = weight of colorant to be added to the trial batch to obtain a corrected batch with a colorant concentration of C_d , and

L.F. = "loss factor" = weight of paint from trial batch used to prepare the corrected batch/weight of paint prepared as trial batch.

The loss factor, L.F., allows for paint removed to prepare drawdowns, loss by transfer to different containers, etc. This term may be neglected if losses are small compared to the batch size. The other terms are as defined in the previous sections of this appendix.

X2.3.2 For the case where ΔC is negative, the weight of white needed to prepare a corrected batch corresponds to,

$$\Delta W_w = W_c - C_d(W_w + W_c) / C_d \times (L.F.)$$

where ΔW_w = weight of white needed to adjust the colorant concentration in the batch to C_d . Other terms are as defined above or in previous sections of this appendix.

X2.4 Prepare a paint film from the adjusted batch. After drying, measure the reflectance at the wavelength of max-

imum absorption. If this value does not fall within 35 to 45 %, repeat X2.3 through X2.4, where C_d now becomes the C_c for the next batch correction. Normally only a single iteration is necessary and a second batch correction will not be needed.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Standard Practice for Preparatory Surface Cleaning of Architectural Sandstone¹

This standard is issued under the fixed designation D 5107; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This practice covers non-abrasive surface cleaning of architectural sandstone to remove grease, dirt, loose material, and surface deposits such as soot, fly ash, hydrocarbon residues, and algae and other biological growth in preparation for the application of water repellent coatings. Procedures include broom cleaning, vacuum cleaning, air blast cleaning, water cleaning (and detergent water cleaning), and chemical cleaning.

1.2 **Limitations**—This practice is intended to clean architectural sandstone without damaging it or altering the surface profile.

1.3 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 4.

Referenced Documents

- 2.1 **ASTM Standards:**
 C 119 Definitions of Terms Relating to Natural Building Stones²
 D 4262 Test Method for pH of Chemically Cleaned or Etched Concrete Surfaces³
 D 4263 Test Method for Indicating Moisture in Concrete by the Plastic Sheet Method³
 D 4285 Method for Indicating Oil or Water in Compressed Air³

Significance and Use

3.1 Surface cleaning is necessary to prepare architectural sandstone surfaces for application of coatings intended for water repellent protection. Surface cleaning of the sandstone substrate helps to ensure proper adhesion of the coating.

3.2 Use of procedures described in this practice may not be adequate where protective systems will be used for continuous or intermittent immersion or mechanical loading.

Hazards

4.1 Moisture in the architectural sandstone may be detrimental to coating adhesion or (in some cases) cure. Moisture content shall be in compliance with coating manufacturer's

recommendation. See also Test Method D 4263.

4.2 Localized staining (for example efflorescence and metallic staining) and previously applied coatings or preservative treatments not compatible with the treatment may require removal by other surface preparation methods.

4.3 Water cleaning, detergent water cleaning, and chemical cleaning should not be performed at temperatures below 40°F.

4.4 Many chemical cleaning products contain acids and should be handled according to manufacturers' recommendations. Use and disposal of materials should conform to established federal, state, local, and project requirements.

4.5 If pressure washing equipment is employed for water cleaning or for flushing the surface with detergent water cleaning or chemical cleaning, the minimum effective pressure should be used. Avoid excessive pressures that could damage the sandstone substrate.

5. Procedure

5.1 Air blast, water, scrubbing, sweeping, or vacuuming are acceptable cleaning methods. Chemical cleaning agents may be used to remove surface deposits such as soot, fly ash, and hydrocarbon residues not removed by any of the above methods. Chemical cleaning should be preceded and followed by a thorough fresh water rinse.

5.2 Existing conditions of the substrate will determine the selection of appropriate procedure(s). Prior to the initiation of cleaning, small test areas should be cleaned in inconspicuous areas by the selected procedure to determine effectiveness.

5.3 The six types of cleaning procedures are described below. One or more of the procedures may be required to remove contaminants from the sandstone surface:

5.3.1 **Broom Cleaning**—Removes most loosely adherent solid contaminants.

5.3.1.1 Working from top to bottom, sweep the surface with a clean industrial stiff-bristled broom or similar device. Remove sweepings from the immediate work area.

5.3.1.2 Clean broom-cleaned surfaces again using one or more of the surface preparation procedures specified in 5.3.2 through 5.3.6.

5.3.2 **Vacuum Cleaning**—Removes surface dust and other debris.

5.3.2.1 Vacuum the surface with a heavy-duty type industrial vacuum to provide an essentially dust-free surface.

5.3.3 **Air Blast Cleaning**—Removes debris, dust, dirt, loosely adherent architectural sandstone, and laitance from walls to provide an essentially sound, dust-free surface.

5.3.3.1 Clean surface with a compressed-air stream through a blasting nozzle held at an oblique angle approximately 2 ft (0.6 m) from the surface. Air stream pressure

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.47 on Masonry Treatments.

Current edition approved Nov. 21, 1990. Published January 1991.

² Annual Book of ASTM Standards, Vol 04.08.

³ Annual Book of ASTM Standards, Vol 06.01.

should not exceed 100 psi (689 kPa).

5.3.3.2 Before initiating air-blast cleaning, verify that the air stream is free of oil in accordance with Test Method D 4285.

5.3.3.3 Surface cleanliness is dependent upon carrying off airborne dust before it is redeposited. Vacuum cleaning may be required to remove redeposited dust.

5.3.4 *Water Cleaning*—Removes dust, dirt, and water-soluble surface contaminants.

5.3.4.1 Clean the surface with a stream of clean potable water, aimed at an oblique angle approximately 2 ft (0.6 m) from the surface, having sufficient pressure to remove dust, dirt, and loose material without damaging the substrate. When necessary, hand scrub with a nonmetallic stiff-bristled fiber brush.

5.3.4.2 Prior to water cleaning, make provisions for the removal of wash water and contaminants generated.

5.3.4.3 If necessary, test the cleaned surface for moisture content in accordance with Test Method D 4263 prior to applying coatings.

5.3.5 *Detergent Water Cleaning*—Removes water-soluble surface contaminants and oils, grease, and other emulsifiable materials on the surface.

5.3.5.1 Scrape off heavy deposits of grease or oil and prewet the surface with potable water. Clean the surface with a nonmetallic stiff-bristled fiber brush, using an aqueous solution of detergent or nonsolvent emulsifier. Immediately after treatment, before the surface dries, remove residues of the cleaning agent by thoroughly flushing the surface with clean potable water. Repeat flushing until the pH of the surface water meets the acceptance criteria of Test Method D 4262.

5.3.5.2 Repeat 5.3.5.1 until water does not bead on the surfaces.

5.3.5.3 Prior to detergent water washing, make provisions for the removal of wash water and contaminants generated.

5.3.5.4 If necessary, test the cleaned surface for moisture content in accordance with Test Method D 4263.

5.3.6 *Chemical Cleaning*—This procedure is similar to detergent water washing, but involves the use of proprietary chemical cleaning compounds (generally hydrofluoric and phosphoric acids) for the removal of surface deposits such as

soot, fly ash, and hydrocarbon residues.

NOTE—In some cases, removal of heavy soiling may require use of alkaline pretreatment prior to acidic cleaning.

5.3.6.1 Chemical cleaning using acidic products is generally not recommended for calcareous sandstones. (See Definitions C 119.)

5.3.6.2 Protect all adjacent materials and surrounding areas as recommended by the manufacturer of the proprietary chemical cleaning compound.

5.3.6.3 Prior to chemical cleaning, provide for application to small test area to determine effectiveness. Make provisions for the removal of water and contaminants generated during full-scale cleaning operations.

5.3.6.4 Before applying the chemical cleaning compound, prewet the surface thoroughly with potable water to prevent absorption of the cleaning solution within the pores of the masonry.

5.3.6.5 Apply a dilute solution of a proprietary cleaning compound to the pre-wet surface as recommended by the manufacturer. Leave the cleaning solution on the surface for a prescribed dwell period (usually less than 5 min).

5.3.6.6 Immediately following the dwell period, and before the surface dries, flush thoroughly to wash chemical cleaning compounds from the surface, then rinse thoroughly from bottom to top. (Rinsing from bottom to top helps to avoid surface streaking.) Repeat flushing may be necessary to remove cleaning residues.

5.3.6.7 *Evaluation*—Visually examine the prepared surface to evaluate removal of debris, dust, dirt, oil, grease, loosely adherent sandstone building stone, and other contaminants. Test the surfaces cleaned with proprietary chemical cleaning compounds for pH in accordance with Test Method D 4262 and if necessary test for moisture content in accordance with Test Method D 4263 prior to applying coatings.

5.4 Surface cleaning is intended to provide a clean, contamination-free surface without damaging or removing architectural sandstone from intact, sound surfaces. Acceptable surfaces shall be free of oil, grease, loosely adhering sandstone, and other contamination such as dirt, soot, fly ash, and hydrocarbon residues.

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Standard Test Method for Organotin Release Rates of Antifouling Coating Systems in Sea Water¹

This standard is issued under the fixed designation D 5108; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

This test method covers the laboratory determination of the rate at which organotin expressed as tributyltin (TBT) released from an antifouling (AF) coating in synthetic sea water using graphite furnace atomic absorption spectrophotometry (GF-AAS). This does not exclude the use of other analytical methodology for measurement of organotin in sea water such as gas chromatography.

2 The values stated in SI units are to be regarded as standard. The inch-pound units given in parentheses are for information only.

3 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety practices and to determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 7.

Referenced Documents

1.1 ASTM Standards:

- D 1141 Specification for Substitute Ocean Water²
- D 1212 Methods for Measurement of Wet Film Thickness of Organic Coatings³
- D 4138 Test Method for Measurement of Dry Film Thickness of Protective Coatings by Destructive Means³

Summary of Test Method

1.1 The candidate paint system is applied to cylindrical specimens. The coated specimens are placed in a tank of synthetic sea water where the tin levels are kept low by circulating the sea water through a carbon filter. At specified intervals, each specimen is placed in 1500 mL of unused sea water and is rotated for 1 h. The rate of tributyltin release from the paint is determined by measuring tributyltin concentrations in the sea water.

1.2 Analysis of sea water for tributyltin is conducted by extracting the organotin with toluene, washing with sodium hydroxide, and measuring for total tin using (GF-AAS).

Significance and Use

1.1 This test method is designed to provide a laboratory procedure to measure changes in the release rates of solvent

soluble tin (tributyl- and triphenyltin) that occur during a period of immersion under specified conditions of constant temperature, pH, salinity, and low heavy-metal concentrations in the surrounding sea water. Quantitative measurement of the release rate is necessary to help in selection of materials, in providing quality assurance, and in understanding the performance mechanism.

4.2 This test method serves only as a guide for organotin release rates in service. Organotin release rates of antifouling (AF) paint systems in service can vary over the life of the coating system depending on the formulation and the environment. Differences in berthing locations, operating schedules, length of service, condition of paint-film surface, temperature, pH, and salinity can affect results. Results obtained may not necessarily reflect actual tributyltin release rates that will occur in service, but provide reliable comparisons of the release rate characteristics of different antifouling formulations.

4.3 This test method will serve to characterize the early release rate pattern, as well as estimate the steady state release, of tributyltin from both self-polishing copolymer and free-association antifouling paints.

5. Apparatus

5.1 *Release-Rate Measuring Container*—A 2-L (nominal ½ gal) polycarbonate container,⁴ approximately 13.5 cm (5.5 in.) in diameter and 19 cm (7.5 in.) high, fitted with three polycarbonate rods approximately 6 mm (nominal ¼ in.) in diameter to serve as baffles. Rods shall be evenly spaced on the inside circumference of the container to prevent swirling of water with the test cylinder during rotation. The rods will be secured to the container walls using acetone or methylene chloride.

5.2 *Constant Temperature Bath*—A temperature controlled water bath capable of maintaining a temperature of $25 \pm 2^\circ\text{C}$ into which one or more release rate measuring test containers can be placed.⁵

5.3 *Holding Tank*—A container of such dimensions so as to permit immersion of four or more test cylinders; must be equipped with a system to continuously circulate synthetic sea water in the tank through a carbon filter. The rate of water flow and the size of the carbon filter should be selected to maintain tributyltin concentrations below 100 µg/L. Flow

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.45 on Marine Coatings.

Current edition approved Dec. 5, 1990. Published February 1991.

² Annual Book of ASTM Standards, Vol 11.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ A Nalgene Container, available from Cole-Palmer, 7425 N. Oak Ave., Chicago, IL 60648, catalog number R-6761-20, or equivalent, has been found satisfactory for this purpose.

⁵ Boekel Water Baths, Models 148003 and 148004 available from Boekel Industries Inc., 509-T Vine St., Philadelphia, PA 19106, or equivalent, have been found satisfactory for this purpose.

rates should generally be set to obtain 2 to 8 turnovers per h. The size and geometry of the tanks as well as the positioning of the inflow and outflow ports for the water circulation system should be selected to obtain a slow, relatively uniform flow of synthetic sea water past all test cylinders in the tank. Maintain the pH of the synthetic sea water between 7.8 and 8.2, and the salinity between 30 and 35 parts per thousand (ppt). The tank shall be provided with heaters to maintain the temperature between 21 and 27°C (70 and 81°F).

5.4 Test Cylinders—Approximately 6.4 cm (nominal 2½ in.) outside diameter polycarbonate pipe coated with a 10-cm band of AF paint around the exterior circumference of the test cylinder to provide 200 cm² of paint film that can be immersed and freely rotated in the release rate measuring container. Seal the bottom of the test cylinder with a polycarbonate disc using acetone, methylene chloride, or a polycarbonate cement so as to form a watertight joint. Do not coat the bottom 1 to 2 cm of the test cylinder. The test cylinder shall be of such height so that a rotating device can be attached to rotate the cylinder and the upper open end of the cylinder is above the level of the test container immersion liquid to prevent entry of the immersion liquid into the test cylinder.

5.5 Test Cylinder Rotating Device—The device shall be capable of rotating the test cylinder in the release rate measuring container at 60 ± 5 r/min. No part of the device shall be immersed in sea water.⁶

5.6 Centrifuge Tubes, 50-mL capacity, with screw closures⁷ (or disposable bottles, culture tubes, separatory funnels, etc.) made of polycarbonate, TFE fluorocarbon, or borosilicate glass.

5.7 Mechanical Shaker, with appropriate holders.

5.8 Dispensers, automatic or repeating, for reagents.

5.9 Pipets, with disposable polypropylene tips.

5.10 Graphite Furnace, atomic absorption spectrophotometer (GF-AAS) with automatic sampler.

5.11 pH Meter, with a mercury/mercurous chloride (Hg/Hg₂Cl₂) electrode.

5.12 Appropriate Volumetric Flasks.

6. Reagents and Materials

6.1 Synthetic Sea Water—Substitute ocean water in accordance with Section 6 of Specification D 1141 or a proprietary equivalent with a salinity of 30 to 35 ppt.

6.2 Extraction Solvent—Toluene, spectrograde or equivalent.

6.3 Tributyltin Standards—Prepare standards using a stock solution of tributyltin chloride (reagent grade, minimum 96 % pure) in methanol (suggested concentration of approximately 10 mg/L). The standards are acidified with acetic acid (less than pH 4) to obtain a stable solution.

6.4 Hydrochloric Acid (HCl) (10 % aqueous solution).

6.5 Hydrochloric Acid (HCl) (0.1N).

6.6 Nitric Acid (HNO₃) (10 % aqueous solution) can be used in place of HCl to clean labware.

6.7 Sodium Hydroxide (NaOH) (3 % aqueous solution).

6.8 Sodium Hydroxide (NaOH) (0.1N).

6.9 All reagents and cleaning agents used must be tin-free.

7. Hazards

7.1 Warning—Antifouling paints contain toxic materials that could cause skin and eye irritation on contact and adverse physiological effects if ingested or inhaled. In the preparation of test specimens and the application of various types of paints, the use of appropriate protective clothing and equipment is required consistent with local, state, and federal government regulations, and recognized industrial and technical standards. Spills, overspray, and unused material should not be flushed down the drain, but should be disposed of as hazardous waste.

7.2 See antifouling paint supplier's Material Safety Data Sheet.

8. Calibration and Standardization

8.1 Prepare three standards throughout the range of the quantification limit to 100 µg of tin per litre by dilution in toluene of a stock solution of tributyltin chloride (96 % pure) in methanol. Include one standard with a concentration of approximately 50 µg of tin per litre. An alternate range of concentrations may be used when appropriate.

8.2 Prepare synthetic sea water spiked with three concentrations of TBT in the range of 10 to 50 µg of tin per litre by spiking with stock solution of tributyltin chloride in methanol. When the concentration of tin extracted in toluene exceeds 100 µg/L appropriate dilution should be employed to keep it within the limits of the calibration curve (0 to 100 µg/L).

8.3 Operate the graphite furnace in accordance with manufacturer's instructions. Optional conditions are described in Appendix X1.

8.4 Analyze the following:

8.4.1 At the beginning of each instrument run, perform analysis of the toluene blank and standards in toluene in order to establish that the response of the instrument is linear. Plot separate calibration curves for each analysis of the standards (peak height absorbance versus tin concentration), and calculate the slope, intercept, and coefficient of determination for each curve using least squares regression or another appropriate procedure.

8.4.2 Sea Water Blank—Extract and analyze as specified for test samples to establish baseline.

8.4.3 Spiked Sea Water Samples—Extract and analyze as specified for test samples to determine extraction efficiency. Recovery must be 90 to 110 %.

8.4.4 If changing the graphite tube during a run is necessary, the blank and standards in toluene should be analyzed to ensure proper response and linearity before continuing the sample analysis.

8.5 Determine the tin concentration of individual test samples with reference to the 50 µg/L calibrating standard analyzed immediately after those test samples.

9. Procedure

9.1 Organotins have a strong tendency to adsorb on certain glass or plastic surfaces. Therefore, all labware (glass or polycarbonate) used for organotin release measurements

⁶ A six-paddle stirrer, Model 300, manufactured by Whitaker Medical Mfg. Co., Phipps and Bird Div., 8741 Landmark Rd., Richmond, VA 23228, or equivalent, has been found satisfactory for this purpose.

⁷ Oak Ridge Tubes, available from Cole-Parmer, or equivalent, have been found satisfactory for this purpose.

be treated as follows: clean thoroughly by soaking in HCl or HNO₃ for a minimum of 6 h. Rinse labware thoroughly with distilled water and allow to dry. Cleaning also be accomplished by soaking in concentrated HCl for 24 h. Prepare all samples, blanks, and standards in labware prepared in this manner. Disposable materials (pipet tips, centrifuge tubes, etc.) do not have to be acid-washed before use. Prepare the exposure surfaces (200 cm²) of three replicate test cylinders to provide a suitable surface for application of the paint system to be applied. Mask the surfaces to remain uncoated (including the bottom 1 to 2 cm of the interior circumferential surface of the test cylinder).

3 Paints shall be manufactured a minimum of seven days prior to testing. Also, test paints shall not be allowed to expire beyond the manufacturer's recommended shelf life. Proper storage conditions during aging, that is, sealed in a container commonly used for sale and held at 20 to 30°C.

4 Apply one or more coats of antifouling paint to the interior circumferential surface of a test cylinder to produce a band of AF paint with an exposure surface of 200 cm² and a minimum dry film thickness of 100 µm (4 mils). Follow manufacturer's instructions with respect to mixing and drying. At a minimum, mechanically shake until the paint appears homogeneous. Apply using a sponge applicator or brush as recommended by the manufacturer. If the paint is marketed only in spray cans, then apply as a spray. After the final application allow the paint to dry for 7 ± 1 days at 23 to 25°C.

5 Estimate the initial dry film thickness using a suitable nondestructive method such as Method D 1212. If the leaching tests exceed 6 months measure the film thickness at the conclusion of the test. Methodology for the final measurement can be either that used for the initial measurement or Test Method D 4138. If a nonstandard method is used, make several film thickness measurements for each cylinder and estimate the variability of the determination. Film thickness should remain greater than 50 µm throughout the test. For tests of long duration, the initial thickness may need to be greater than 100 µm to maintain a thickness greater than 50 µm throughout the test.

6 Place all cylinders in a single batch in a holding tank after the 7-day drying period. A batch consists of one or more sets of three replicate cylinders coated with a test paint and one control (unpainted) cylinder. The painted surface on the cylinders must be completely submerged. Cylinders must be stationary and positioned so that sea water moving through the tank will flow around each cylinder.

7 Monitor the pH (using a pH meter with a calomel electrode) and the temperature of the synthetic sea water in the holding tank daily. Adjust the pH if necessary using either 0.1N NaOH or 0.1N HCl. Quantitate salinity every 14 days and adjust if necessary. Determine TBT concentrations weekly. When TBT levels increase, change the carbon filter before the TBT concentration exceeds 100 µg/L. More frequent monitoring of synthetic sea water may be necessary in some instances to maintain the specified environmental conditions.

8 After 1, 3, 7, 10, 14, 21, 24, 28, 31, 35, 38, 42, and 45 days, transfer all cylinders in given batch into individual measuring containers containing 1500 mL of fresh synthetic sea water. Randomly assign cylinders (control and painted)

to measuring containers at each leaching. When transferring cylinders, lift the cylinder out of the holding tank, allow sea water to drain off, install the cylinder into the rotating device, and submerge the painted area into the sea water. Immediately start rotation of the cylinder at 60 ± 5 r/min, and continue rotation for 60 min. When transferring the cylinders, do not touch or in any way damage the paint film, and do not allow the paint surface to dry. The transfer should be completed as quickly as possible (generally, in less than 5 min).

9.9 If testing beyond the minimum (45 days) length requirement is desired, the study may be extended to 73 days. During the extended test, remove the cylinders from the holding tank every 3 to 4 days to make a measurement of the leach rate.

9.10 At the completion of the cylinder rotation, immediately remove the cylinder from the measuring tank and return it to the holding tank. Pipet a 25-mL subsample of the sea water into a 50-mL centrifuge tube containing sufficient 10% HCl to reduce the pH to ≤4.0. If the number of samples from the leach measuring steps exceeds the daily analysis capacity, the samples may be refrigerated and stored in the acidified state in a sealed container for up to 14 days. Clean the measuring containers using appropriate procedures (distilled water or acid wash) before reuse.

9.11 Partition acidified sea water samples with 10 mL of toluene (15 min shaking on a mechanical shaker). Remove most of the toluene and wash it with 5 mL of a 3% aqueous NaOH solution (10 min shaking). Pipet off (or separate using a separatory funnel) the organic phase and analyze for total tin content by GF-AAS. The toluene extract can be stored sealed in the dark at 4°C for up to 24 h before analysis.

10. Calculation

10.1 Calculate the concentration of the TBT cation in the sea water of the measuring container as follows:

$$C_{TBT} = (C \times E \times F) / S$$

where:

C_{TBT} = concentration of TBT cation (µg/L),
 C = concentration of tin in the toluene extraction (µg/L),

E = volume of toluene = 10 mL,

F = correction factor to convert tin to TBT = 2.5, and

S = aliquot of sea water analyzed = 25 mL.

This equation can be simplified as follows if the prescribed volume of sea water and toluene are used:

$$C_{TBT} = (C_{Sn} \times 10 \times 2.5) / 25 \\ = C_{Sn}$$

10.2 Calculate the release rate by the following formula:

$$R = (C_{TBT} \times V \times D) / (T \times A)$$

where:

R = release rate (µg/cm²/day),

D = 24 h/day,

V = 1.5 L, volume of sea water in measuring container,

T = 1 h during which cylinder is in measuring container, and

A = 200 cm² surface area of paint film.

This formula can be simplified as follows:

$$R = (C_{\text{TBT}} \times 1.5 \times 24) / (1 \times 200) \\ = C_{\text{TBT}} \times 0.18$$

10.3 Calculate the 14-day cumulative release of TBT cation as follows:

$$R = R_1 + (2 \times R_3) + (4 \times R_7) + (3 \times R_{10}) + (4 \times R_{14})$$

where:

R = 14-day cumulative release, $\mu\text{g}/\text{cm}^2 \cdot 14$ days, and R_1, R_3, R_7, R_{10} , and R_{14} = release rates for sampling days 1, 3, 7, 10, and 14, respectively, $\mu\text{g}/\text{cm}^2$ per day.

10.4 Calculate the average release rate ($\mu\text{g}/\text{cm}^2$ per day) by averaging individual release rate measurements taken from day 21 through the last day of sampling (day 45 through day 73). If values at day 21 are high and it is suspected that the release had not reached pseudo steady-state conditions, then compare the release rate at day 21 to the mean for all release rates from day 21 through the termination of testing (45 to 73 days). If the release rate exceeds the mean by two or more standard deviations, then the release rate may be excluded from the average. If the day 21 release rate is excluded, the day 24 release rate may be

evaluated by the same procedure.

11. Report

11.1 Report the following information:

11.1.1 Report the concentration in micrograms per litre of TBT cation in the sea water of the measuring tank and the rate of TBT cation release (micrograms per square centimetre per day) for each sampling time (give values for individual replicates as well as the mean). Plot the rate of TBT release as a function of time (use linear axes). Also report the 14-day cumulative release and the average release rate.

12. Precision and Bias

12.1 Precision and bias for this test method have not been determined.

13. Keywords

13.1 antifouling coating system; organotin; release rate; TBT; tributyl tin

APPENDIX

(Nonmandatory Information)

X1. GRAPHITE FURNACE OPERATING INSTRUCTIONS

X1.1 Operate the graphite furnace in accordance with the manufacturer's instructions. Tables X1.1 and X1.2 provide

guidelines that have been used by two operators.

TABLE X1.1 Navy Equipment—for Toluene Extraction

NOTE—Light Source: Tin electrodeless discharge lamp (EDL)
Wavelength: 224.6 nm
Background Corrector: On

Step	Temperature, °C	Ramp Time, s	Hold Time, s	Gas Flow (mL/min)
1. Dry	100	5	10	300
2. Dry ^A	110	5	5	300
3. Char ^B	1000	10	2	300
4. Atomize	2000	0	8 ^C	0
5. Clean	2700	2	5	600
6. Cool	20	2	15	300

^A 286.3 nm tin hollow cathode.

^B Gas stop stage must be used after Step 2 to prevent the carrier gas sweeping organotin out of the tube.

^C Stop gas.

TABLE X1.2 Perkin-Elmer 4000AS^B Fitted With A Perkin-Elmer 500 Graphite Furnace With Temperature Programmer

NOTE—Light Source: Hollow cathode lamp
Wavelength: 286.3 nm

Step	Temperature, °C	Ramp Time, s	Hold Time, s
1. Chart ^A	250	7	30
2. Ash	900	7	30
3. Atomize	2600	0	5
4. Clean	2700	1	3

^A Purge gas used: argon.

X2. AUTOSAMPLER

X2.1 Perkin-Elmer AS40 Autosampler^B—This autosampler has been used with the Perkin-Elmer HGA 500 graphite

furnace. The autosampler introduces aliquots of 20 μL into the graphite tube. Each sample is analyzed three times and an average given. The machine automatically recalibrates after every 12 samples.

^B Available from Perkin-Elmer Corp., 761-T Main Ave., Norwalk, CT 06859; 0001, or equivalent, has been found suitable for this purpose.

X3. SUGGESTED TABULAR REPORT FORM

Spot	Day	Rotation Time, h	Measured Concentration of Tin, $\mu\text{g/L}$				Avg TBT Release Rate, $\mu\text{g/cm}^2$ per day	Total Release Rate, $\mu\text{g/cm}^2$ per sample interval	Cumulative Total TBT Release Rate, $\mu\text{g/cm}^2$
			Rep 1	Rep 2	Rep 3	Average			
1	1								
2	3								
3	7								
4	10								
5	14								
6	21								
7	24								
8	28								
9	31								
10	35								
11	38								
12	42								
13	45								
14	to 73 days (optional)								

erkin-Elmer
mmmer

fold Time, s

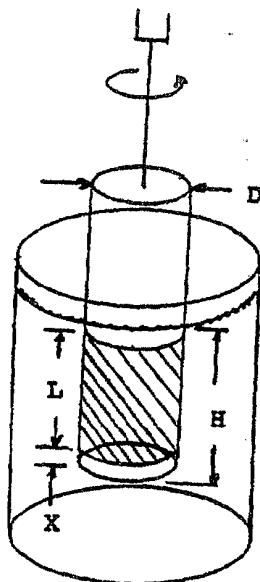
30
30
5
3

20 μL into
times and
ecalibrates

X4. DESCRIPTION OF PROPOSED TESTING APPARATUS

X4.1 A 200 cm² organotin antifouling paint film of a minimum 100 μ m thickness is applied to the curved surface of a polycarbonate cylinder closed at one end. This cylinder is suspended with its closed end immersed within and concentric with a larger polycarbonate cylinder holding

synthetic sea water. The coated internal cylinder is rotated about its axis at 60 ± 5 r/min in order to produce a peripheral speed of about 1 knot. In practice, the commercially available polycarbonate "multipurpose jars" described in this test method will hold 1500 mL of synthetic sea water.



NOTE 1—Test Container Dimensions:

Capacity, L	2
Inside diameter, cm	12.7
Outside diameter, cm	13.3
Height (without cover), cm	19

NOTE 2—Rotating Test Cylinder Dimensions:

D = approximately 7 cm (2 1/2 in. nominal),
H = 12 cm min,
L = 10-cm coated section, and
X = 1-cm uncoated band.

FIG. X4.1 Dimensions of Testing Apparatus (Container and Cylinder)

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Standard Specification for Sample Preparation for Qualification Testing of Coatings to be Used in Nuclear Power Plants¹

This standard is issued under the fixed designation D 5139; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

Scope

1 This specification defines the size composition and surface preparation requirements for test samples used to evaluate coatings according to the following ASTM test procedures.

Referenced Documents

ASTM Standards:

- A 36 Specification for Structural Steel²
- C 33 Specification for Concrete Aggregates³
- C 150 Specification for Portland Cement⁴
- C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory³
- C 260 Specification for Air-Entraining Admixtures for Concrete³
- C 494 Specification for Chemical Admixtures for Concrete³
- C 618 Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete³
- D 3911 Test Method for Evaluating Coatings Used in Light-Water Nuclear Power Plants at Simulated Design Basis Accident (DBA) Conditions⁵
- D 3912 Test Method for Chemical Resistance of Coatings Used in Light-Water Nuclear Power Plants⁵
- D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser³
- D 4082 Test Method for Effects of Radiation on Coatings Used in Light-Water Nuclear Power Plants⁵
- D 4256 Test Method for Determination of the Decontaminability of Coatings Used in Light-Water Nuclear Power Plants⁵
- D 4258 Practice for Surface Cleaning Concrete for Coating⁵
- D 4259 Practice for Abrading Concrete⁵
- D 4260 Practice for Acid Etching Concrete⁵
- D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers³

2.2 Other Standards:

American Concrete Institute, ACI 301 Specifications for Structural Concrete for Buildings⁶
SSPC-SP-1, 2, 3, 5, 6, 7, 10, or 11⁷

3. Significance and Use

3.1 This specification provides uniform requirements for the preparation of test samples used for qualification testing of coatings used in nuclear power plant construction and maintenance.

4. Steel Samples

4.1 Sample size shall be a minimum of 2 in. wide by 4 in. long by $\frac{1}{8}$ in. thick. Edges and corners may be rounded. A $\frac{1}{4}$ in. diameter hole suitably located may be in the test panel as appropriate for the test samples in Test Methods D 3911, D 3912, D 4082 and D 4256.

4.1.1 For Test Method D 4541 the minimum size shall be 3 in. wide by 5 in. long by $\frac{1}{4}$ in. thick.

4.2 All panels should be carbon steel, meeting the requirements of Specification A 36.

4.3 Surface preparation shall be in accordance with SSPC-SP 10 for qualification testing. Other surface preparation as required by the project or specific conditions may be used when testing for evaluation over surfaces other than SSPC-SP 10, such as SSPC-SP 1, 2, 3, 5, 6, 7, or 11.

5. Concrete Blocks

5.1 Applicable to Test Methods D 3911, D 3912, D 4082, D 4256 and D 4541. The minimum size shall be 2 in. by 2 in. deep by 4 in. long. The edges may be chamfered up to $\frac{1}{4}$ in. maximum.

5.2 Composition shall be as follows:

Cement	Specification C 150, Type II	7 sacks/yd ³
Gravel	Specification C 33, size $\frac{3}{4}$ in.	45 % by volume
Sand	Specification C 33	55 % by volume
Air-entraining admixture	Specification C 260	As recommended 4 to 7 %
Water-reducing admixture	Specification C 494	As recommended
Pozzolan	Specification C 618	As recommended to 15 % amount
Water, demineralized or distilled		3 in. slump

¹ This specification is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters. Current edition approved Dec. 12, 1990. Published January 1991.

² Annual Book of ASTM Standards, Vol 01.04.

³ Annual Book of ASTM Standards, Vol 04.02.

⁴ Annual Book of ASTM Standards, Vol 04.01.

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ Available from American Concrete Institute, P.O. Box 19150, Detroit, MI 48219.

⁷ Surface Preparation Standards are available from Steel Structures Painting Council, 4400 5th Ave., Pittsburgh, PA 15213-2683.

		Nominal 1 Cubic Foot Batch
Cement	Specification C 150, Type II low alkali	22.2 lb
Gravel	Specification C 33, Size $\frac{3}{4}$ in.	45.3 lb
Sand	Specification C 33	55.5 lb
Air-entraining ad- mixture	Specification C 260	31 mL
Pozzolan	Specification C 618	2.2 lb
Water-reducing admixture (Type A)	Specification C 494	As recommended by the manufac- turer
Water, demineral- ized or distilled		As required to pro- duce 3-in. slump (approximately)

5.3 Blocks shall be cast horizontally in forms using release agents that are compatible with the coatings to be used. The top surface, as cast, shall be given a broom finish, unless otherwise specified. The block surface is to be covered with plastic during the first 24 h to simulate water curing unless a curing compound is included in the testing.

5.4 The block shall be removed from the form after 24 h and wet cured in accordance with ACI 301 or Practice C 192.

5.5 If a curing compound is to be used, apply the compound to the broom finished surface immediately after finishing and to all other surfaces of the block within 2 h after its removal from the form.

5.6 Allow the block to cure for 28 days in accordance with ACI 301 before application of the coating system unless otherwise specified.

5.7 After curing, remove loose material on the broom finish surface by light wirebrushing. Remove loose material on the cast surfaces by blowing with air (80 to 100 psi) unless otherwise specified, such as Practices D 4258, D 4259, or D 4260.

5.8 All surfaces of the blocks shall be coated in accordance with Test Method D 3912. The upper and/or lower ends of the blocks shall be left uncoated for all other testing.

5.9 A suitable hanger compatible with the testing apparatus shall be affixed at the mid-point of the upper end of the block where applicable.

5.10 Large bug-holes, rock pockets and other coating defects may be simulated by drilling holes to test coating systems for patching surface defects.

6. Miscellaneous Materials

6.1 Follow 4.1 and 4.3 for metallic materials such as

aluminum, galvanized steel, and other metals.

6.2 Follow 5.1 and 5.3 for castable materials such as grout, fireproofing, and other castables.

7. Abrasion Test Samples

7.1 Substrate material for abrasion testing shall be of a size in accordance with Test Method D 4060 and shall be aluminum or steel and of a thickness to maintain a flat surface after surface preparation.

7.2 Surface preparation shall be compatible with coating to be tested.

8. Application

8.1 All test samples shall be coated with the full coating system including any applicable fillers. The manufacturer's latest published application instructions shall be followed for mixing and coating application unless otherwise specified. Any special procedures or conditions shall be noted in the documentation including thinning, mixing, drying/curing times, force curing, aging between coats/systems, intermediate surface preparations or other special procedures or conditions.

8.2 The film thickness range shall be representative of the specified work for which the testing is being conducted.

8.3 Test samples for maintenance painting test programs may include simulated aging of the existing coating, intermediate surface preparation and wide ranges of coating film thicknesses. Field conditions should be duplicated as close as possible including coating application methods that may differ from the manufacturer's published data.

9. Documentation

9.1 The procedures and conditions used for the test sample preparation shall be documented. As a minimum the documentation shall include preparation times and dates, surface preparation details, coating sequence and individual dry film thickness ranges of each coat, total thickness range, environmental conditions, and product batch identification.

9.2 Record all deviations from the procedures called for in this specification, that is, all items allowing "unless otherwise specified" that are different than those described.

10. Keywords

10.1 concrete blocks; nuclear power plants; qualification testing; sample preparation; steel samples; test samples

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Scope

1.1 This guide covers coating systems for concrete and steel in nuclear power plants.

This guide covers coating systems for concrete and steel in nuclear power plants. It is the responsibility of the user to ensure that the coating system is suitable for the intended application.

Standard Guide for Use of Protective Coating Standards in Nuclear Power Plants¹

This standard is issued under the fixed designation D 5144; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Protective coatings (paints) have been used extensively in the nuclear industry to protect the surfaces of facilities and equipment from corrosion and contamination by radioactive nuclides in accordance with ALARA. In the absence of a standard method of selecting, testing, and evaluating coatings, many sites evaluated paints by empirical tests to determine which were useful in their particular operation. Understandably, the methods of testing were not uniform throughout the industry. It has been very difficult, consequently, to compare the results obtained at one site with those obtained at another. Standard tests whereby industrial (nuclear) users of paints systematically prepare specimens and subject them to selected evaluations, thus permitting uniform comparisons, are advantageous, internationally as well as domestically.

The designer of light water-moderated nuclear reactor systems must consider the possibility of a Design Basis Accident (DBA) and the subsequent events which might lead to the release or expulsion of a fraction of the fission-product inventory of the core to the reactor containment facility. Engineered safety features, principally a reactor containment facility, are provided to prevent the release of fission products to the biological environment during and after this improbable event. The design, fabrication, quality assurance, and testing of these engineered safety features ensure reliable operation and safety under all anticipated conditions.

Large areas of the reactor-containment facility are painted with a protective coating. If severe delamination, peeling, or flaking causes significant portions of the coating to be discharged into the common water reservoir (primary containment sump), the performance of the safety systems could be seriously compromised by the plugging of strainers, flow lines, pumps, spray nozzles, and core coolant channels.

The safety requirement for protective coatings is to ensure that failure of the coatings under DBA conditions does not adversely affect the performance of post-accident fluid systems. Therefore, coatings must be qualified to the environmental exposures anticipated during normal plant operation, and during DBA and other upset conditions.

This guide is the result of a comprehensive examination of the experience and data that have been developed on protective coatings in the nuclear industry over approximately 40 years. Standards pertaining to nuclear coatings have historically been covered by ANSI N5.12, N101.2, and N101.4. Responsibility for updating, rewriting, and issuing appropriate ANSI replacement standards has been transferred to ASTM, specifically ASTM Committee D-33, on Protective Coating and Lining Work for Power Generation Facilities.

Quality assurance in the nuclear industry is a mandatory requirement for all aspects of safety related nuclear coatings work. The objective of this guide is to provide a common basis on which to define and specify the performance requirements for the coatings which will be used in Service Level I and II areas of a nuclear facility. Proposed Regulation 10CFR50.65 (Draft Rule November 28, 1988) defines the requirements for an effective program for maintenance of nuclear power plants. This guide may be used both for coatings applied to new nuclear power plants and to facilitate implementation of the coating aspects of 10CFR50.65.

Scope

1.1 This guide provides a common basis on which protective coatings for the surfaces of light water-moderated nuclear power generating facilities may be qualified and

selected by reproducible evaluation tests. This standard also provides guidance for application and maintenance of protective coatings. Under the environmental operating and accident conditions of nuclear power generation facilities, encompassing Pressurized Water Reactors (PWR's) and Boiling Water Reactors (BWR's), coating performance may be affected by exposure to any one, all, or a combination of the following conditions: ionizing radiation; contamination by radioactive nuclides and subsequent decontamination

¹ This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.02 on Service and Material Parameters. Current edition approved Jan. 8, 1991. Published March 1991.

processes; chemical and water sprays; high-temperature high-pressure steam; and abrasion or wear.

1.2 The content of this guide includes:

	Section
Referenced Documents	2
Terminology	3
Significance and Use	4
Preparation of Test Specimens	5
Radiation Resistance	6
Decontamination	7
Physical Properties	8
Chemical Resistance	9
Fire Evaluation	10
DBA Testing	11
Surface Preparation, Coating Application, and Inspection for Shop and Field Work	12
Quality Assurance	13
Keywords	14

1.2.1 In addition, this guide addresses technical topics within ANSI N5.12 and ANSI N101.2 that were covered by separate ASTM standards, for example, surface preparation, shop and field and coating application, and shop and field.

1.2.2 Applicable sections of this guide and specific acceptance criteria may be incorporated into specifications and other documents where appropriate.²

1.3 This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- D 3843 Practice for Quality Assurance for Protective Coatings Applied to Nuclear Facilities³
- D 3911 Test Method for Evaluating Coatings used in Light-Water Nuclear Power Plants at Simulated Design Basis Accident (DBA) Conditions³
- D 3912 Test Method for Chemical Resistance of Coatings Used in Light-Water Nuclear Power Plants³
- D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser³
- D 4082 Test Method for Effects of Radiation on Coatings Used in Light-Water Nuclear Power Plants³
- D 4227 Practice for Qualification of Journeyman Painters for Application of Coatings to Concrete Surfaces of Safety-Related Areas in Nuclear Facilities³
- D 4228 Practice for Qualification of Journeyman Painters for Application of Coatings to Steel Surfaces of Safety-Related Areas in Nuclear Facilities³
- D 4256 Test Method for Determination of the Decontaminability of Coatings Used in Light-Water Nuclear Power Plants³
- D 4537 Guide for Establishing Procedures to Qualify and Certify Inspection Personnel for Coating Work in Nuclear Facilities³

² Certain ASTM standards are available in compilation form (which includes this guide), as *Compilation of ASTM Standards for Use of Protective Coating Standards in Nuclear Power Plants* for expedient reference and usage by personnel involved in nuclear coating work.

³ *Annual Book of ASTM Standards*, Vol 06.01.

D 4538 Definitions of Terms Relating to Protective Coating and Lining Work for Power Generation Facilities³

D 4541 Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers³

D 5139 Specification for Sample Preparation for Qualification Testing of Coatings to be Used in Nuclear Power Plants³

E 84 Test Method for Surface Burning Characteristics of Building Materials⁴

2.2 Other Standards:

ANSI N5.12 Protective Coatings (Paints) for the Nuclear Industry⁵

ANSI N101.2 Protective Coatings (Paints) for Light Water Nuclear Reactor Containment Facilities⁵

ANSI N101.4 Quality Assurance for Protective Coatings Applied to Nuclear Facilities⁵

10CFR50.65 Draft rule "Ensuring the Effectiveness for Maintenance Programs for Nuclear Reactor Plants" November 28, 1988⁶

Regulation Guide 1.54 Quality Assurance Requirements for Protective Coatings Applied to Water-Cooled Nuclear Power Plants⁶

USNRC Review Plan 6.1.2 Protective Coating Systems (Paints). Organic Materials⁶

10CFR20.1(C) Standards for Protection Against Radiation; Purpose⁶

USNRC Regulation Guide 8.8 Information Relevant to Ensuring that Occupational Radiation Exposures at Nuclear Power Stations Will Be As Low As Is Reasonably Achievable.⁶

3. Terminology

3.1 *Definitions*—Definitions for use with this guide are shown in Definitions D 4538 or other applicable standards.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *ALARA*—the concept of reducing radiation exposure to personnel to levels "as low as is reasonably achievable," as defined in the USNRC Regulation Guide 8.8, and 10CFR20.1(C).

3.2.2 *Coating Service Level I*—areas where coating failure could adversely affect the operation of post-accident fluid systems and, thereby, impair safe shutdown. With few exceptions, *Coating Service Level I* applies to coatings inside primary containment.

3.2.3 *Coating Service Level II*—areas where coating failure could impair, but not prevent, normal operating performance. The function of *Coating Service Level II* coatings is to provide corrosion protection and decontaminability in those areas outside primary containment subject to radiation exposure and radionuclide contamination.

4. Significance and Use

4.1 This guide addresses the concerns of Regulation Guide 1.54 and Standard Review Plan 6.1.2, and the

⁴ *Annual Book of ASTM Standards*, Vol 04.07.

⁵ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁶ Available from the U.S. Government Printing Office, Washington, DC 20402.

replacement of ANSI Standards N5.12, N101.2, and N101.4. This guide covers coating work on previously coated surfaces as well as bare substrates. The entire document applies to all painting work in Service Level I areas. Applicable sections of this guide may be used to evaluate and select protective coatings for Service Level II areas.

Preparation of Test Specimens

All test samples used for qualification testing of coatings shall be prepared in accordance with Specification D 3912.

Radiation Tolerance Tests

Coating film resistance to radiation exposure shall be evaluated in accordance with Test Method D 4082.

Decontamination Test

The relative decontaminability of the coating shall be evaluated by Test Method D 4256.

Certain coatings may contaminate more readily than others, and the responses to decontamination treatments also vary. In some cases, the desired level of decontamination may be achieved merely by cleaning the coating surface; in other cases, decontamination may be achieved only by partial or complete removal of the coating. The decontamination test described gives a method of evaluating the relative ease of decontamination of a coating system. The higher the overall Decontamination Factor (DF), the easier the coating will be to decontaminate.

Physical Properties

8.1 Adhesion—Panels shall be tested for adhesion in accordance with Method D 4541. A minimum of two panels shall be tested for each coating system. If the size of the test specimen is less than 3 in. by 5 in., an annular bearing ring should be used to ensure full contact of the tester legs to the test specimen.

8.1.1 Minimum adhesion shall be 200 psi, consisting of three adhesion tests on each test panel.

8.2 Abrasion Resistance—Abrasion resistance characteristics of coating systems for floors and other surfaces where abrasion is a factor shall be determined in accordance with Test Method D 4060.

8.2.1 Weight loss shall not exceed 175 mg/1000 cycles when a CS-17 Wheel is used with a 1000-g load.

Chemical Resistance Tests

9.1 Test specimens shall be tested in accordance with Test Method D 3912.

9.2 The specific chemicals to be used should be selected to characterize the anticipated exposure; the chemicals indicated in Test Method D 3912 are shown only as examples and are not mandatory.

10. Fire Evaluation Tests

10.1 Flame Spread Tests—Flame-spread tests, when required, shall be conducted and evaluated in accordance with Test Method E 84. The permissible flame-spread and smoke generation, when tested on a noncombustible substrate, shall not exceed the limits set by the nuclear power generating facility.

10.2 The coating systems should be tested to cover the specified film thickness range (or greater) since the flame-spread and smoke density can vary with film thickness. Smoke density is significant where a coating is utilized in enclosed spaces and smoke generation can reduce visibility and prevent effective fire fighting operations. Historic test data indicates that most coatings applied at less than 25 mils dry film thickness over noncombustible substrates and tested in accordance with Test Method E 84 demonstrate flame-spread values below 25.

11. DBA Testing

11.1 The test specimen shall be tested and evaluated in accordance with Test Method D 3911.

12. Surface Preparation, Coating Application, and Inspection for Shop and Field Work

12.1 It is recommended that the *Manual of Coating Work for Light-Water Nuclear Power Plant Primary Containment and Other Safety-Related Facilities Guide*,⁷ prepared by ASTM Subcommittee D01.43, be reviewed in the course of nuclear coating work.

12.2 Surface preparation for steel, concrete, and previously coated surfaces shall be at least as good as that used in the qualification testing of the coating system intended for use.

12.3 Coating application shall be in accordance with the job specifications and the coating manufacturer's latest published instructions. Journeymen painter qualification shall meet the requirements of the applicable quality assurance (QA) program, Practices D 4227 and D 4228. Coating dry film thickness shall be in the range used in the qualification testing of the coating system.

12.4 Coatings work shall be inspected by coatings inspectors qualified and certified in accordance with the applicable QA program and Guide D 4537. Inspections shall be documented to provide a record of the coatings work.

12.5 Maintenance painting work shall follow the requirements of 12.2, 12.3, and 12.4. The maintenance painting specifications shall take into consideration the plant environment in which the coating work must be accomplished. Maintenance painting work qualification testing should be based on proposed surface preparation, coating application methods, and film thickness ranges, all of which may be different than the original design qualification work.

13. Quality Assurance

13.1 A quality assurance program for Service Level I coating work shall be established in accordance with Practice D 3843. Quality assurance requirements may also be established for Service Level II coating work based on criticality.

14. Keywords

ANSI replacement standards; Design Basis Accident (DBA); decontamination; nuclear power plants; protective coating standards; qualification testing; quality assurance Service Level I and II; radiation; safety related

⁷ ASTM, 1979.

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Standard Test Methods for Nonvolatile and Pigment Content of Electrocoat Baths¹

This standard is issued under the fixed designation D 5145; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

These test methods cover the characterization of electrocoat baths through the determination of nonvolatile content of inorganic pigment content.

This standard does not purport to address the safety hazards associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Referenced Documents

ASTM Standards:

- D 1193 Specifications for Reagent Grade Water²
- D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings³
- D 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁴

Summary of Test Method

Two specimens are accurately weighed into aluminum weighing dishes. The dishes are placed in an oven at 110°C for 1 h, reweighed to obtain the nonvolatile matter content and, if required, placed in a muffle furnace at 500°C for 2 h and weighed a third time to obtain the inorganic pigment content.

Significance and Use

4.1 The nonvolatile content and pigment content are measures of total solids and inorganic pigment solids, respectively, in electrocoat paints. In addition to production quality control, these properties are important in maintaining electrocoat baths in the optimum range.

4.2 Other test methods for determining nonvolatile content of paint and paint related materials are described in Method D 2832.

Apparatus

- 5.1 *Analytical Balance* with a sensitivity of 0.1 mg.
- 5.2 *Aluminum Weighing Dishes*, 57 mm in diameter and 7 mm deep. These commercial dishes may contain a lubricant used during their manufacture. This should be removed by heating the aluminum dishes on a hot plate at

300°C until vapors are no longer visible. Store the dishes in a desiccator until needed.

- 5.3 *Syringes*, 5-mL, disposable variety.
- 5.4 *Oven* circulating, maintained at $110 \pm 2^\circ\text{C}$.
- 5.5 *Muffle Furnace*, maintained at $500 \pm 15^\circ\text{C}$.

6. Reagents

6.1 *Purity of Water*—References to water shall be understood to mean water conforming to Type II of Specification D 1193.

7. Sampling and Sample Preparation

7.1 Obtain the sample while the electrocoat bath is under proper circulation so a uniform sample is obtained. In the case of a ultrafiltrate sample, the material should be thoroughly mixed or stirred prior to drawing the sample, thereby ensuring uniformity.

7.2 After sampling, prior to removing the test specimen, it is mandatory the sample be shaken or stirred until it is homogeneous and free of any settled material. This is particularly important if there is a delay between sampling the bath and performing this test procedure. The absence of settled material should be ascertained visually or by inserting a spatula and scraping the bottom of the container. Continue to shake or stir the sample until specimens are taken for measurement. *This Point is Very Important.*

8. Procedure

NONVOLATILE CONTENT

8.1 Weigh two aluminum dishes separately, each to 0.1 mg and record as W_1 .

8.2 Using a syringe, withdraw 1.0 to 1.5 of the well mixed sample, then quickly weigh the syringe to 0.1 mg, recording this weight as W_2 . Transfer the entire contents of the syringe into the aluminum dish. Reweigh the empty syringe to 0.1 mg and record as W_3 . In the case of ultrafiltrate clear liquids or of low solids paints, increase the specimen size to 5 mL and preheat at 60°C for 2 h. Duplicate this step with the second aluminum dish (8.1).

8.3 Add a few millilitres of water to the specimen in the aluminum dishes prior to placing them in the oven. This facilitates uniform spreading of the material. Place the dishes in the 110°C oven for 1 h.

8.4 Remove the dishes from the oven and allow to cool to room temperature in a desiccator. Reweigh them to 0.1 mg and record the weights as W_4 .

8.5 Retain the dishes for measurement of inorganic pigment content as detailed in a following section of these test methods.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coating and Materials and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials. Current edition approved Dec. 13, 1990. Published January 1991.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

9. Calculation

9.1 Calculate the percent nonvolatile content as follows:

$$\% NV = \frac{W_4 - W_1}{W_2 - W_3} \times 100$$

where:

- W_1 = weight of empty aluminum dish, g,
 W_2 = weight of syringe filled with sample, g,
 W_3 = weight of empty syringe, g, and
 W_4 = weight of dish and contents after 1 h at 110°C, g.

10. Precision and Bias

10.1 Precision is based on an interlaboratory study in which the operators in each of ten laboratories analyzed in duplicate on 2 days, four different electrocoat-bath samples with nonvolatile contents ranging from 0.30 to 25.2 %. The results were analyzed statistically in accordance with Practice E 180. The interlaboratory coefficient of variance was 1.6 % at 30 df and the interlaboratory coefficient of variation was 2.3 % at 27 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

10.1.1 *Repeatability*: Two results, each the mean of duplicate determinations obtained by the same operator on different days, should be considered suspect if they differ by more than 4.6 % relative.

10.1.2 *Reproducibility*: Two results, each the mean of duplicate determinations obtained by operators in different laboratories, should be considered suspect if they differ by more than 6.7 % relative.

10.2 No bias has been determined for this test method.

11. Procedure

INORGANIC PIGMENT CONTENT

11.1 Place the weighing dishes used for the nonvolatile content determination in the muffle furnace at 500°C for 2 h.

11.2 After 2 h, remove the dishes from the muffle furnace, transfer them to a dessicator and allow to cool to room temperature. After cooling, remove dishes and weigh to 0.1 mg, recording these weights at W_5 .

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

NOTE 1—If an organic char is still present in the aluminum dish after 2 h in the muffle furnace, continue heating in the furnace until no char is present.

12. Calculation

12.1 Calculate the percent inorganic pigment as follows:

$$\% \text{ Inorganic Pigment (EC Bath)} = \frac{W_5 - W_1}{W_2 - W_3} \times 100$$

where:

- W_1 = weight of empty aluminum dish, g,
 W_2 = weight of syringe filled with sample, g,
 W_3 = weight of empty syringe, g, and
 W_4 = weight of aluminum dish and contents after heating in muffle furnace, g.

13. Precision and Bias

13.1 Precision estimates are based on an interlaboratory study in which the operators in ten different laboratories analyzed in duplicate on 2 days, four electrocoat bath materials containing inorganic pigment content ranging from 0.17 to 4.52 weight %. The results were analyzed statistically in accordance with Practice E 180. The intralaboratory coefficient of variation was 2.2 % relative at 40 df and the interlaboratory coefficient of variation was 10.8 % at 36 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

13.1.1 *Repeatability*: Two results, each the mean of duplicate determinations, obtained by the same operator on different days, should be considered suspect if they differ by more than 6.3 % relative.

13.1.2 *Reproducibility*: Two results, each the mean of duplicate determinations, obtained by operators in different laboratories, should be considered suspect if they differ by more than 31.1 % relative.

13.2 No bias has been determined for this test method.

14. Keywords

14.1 electrocoat bath; muffle furnace; nonvolatile content; pigment content; ultrafiltrates

Scope

This test method is applicable to the determination of inorganic pigment content in electrocoat bath materials. It can be used for the determination of inorganic pigment content in electrocoat bath materials.

NOTE 1—This test method is applicable to the determination of inorganic pigment content in electrocoat bath materials. It can be used for the determination of inorganic pigment content in electrocoat bath materials.

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Standard Guide to Testing Solvent-Borne Architectural Coatings¹

This standard is issued under the fixed designation D 5146; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Scope

This guide covers the selection and use of procedures for testing solvent-borne coatings to be used on exterior, interior or both types of surfaces (see Note 1). The properties that can be examined or, in some cases, the relevant test procedures are listed in Tables 1 and 2.

NOTE 1—The term "architectural coating" as used here combines the definition in Definitions D 16 with that in the FSCT Paint/Coatings Glossary, as follows: "Organic coatings intended for on-site application to interior or exterior surfaces of residential, commercial, institutional, or industrial buildings, in contrast to industrial coatings. They are protective and decorative finishes applied at ambient temperatures. Often called Trade Sales Coatings." (See 2.3.)

NOTE 2—Architectural coatings that are designed to give better performance than most conventional coatings because they are tougher, more stain- and abrasion-resistant are covered by Guide D 3730.

2 The types of organic coatings covered by this guide are as follows:

- (1) Type 1 Interior Low-Gloss Wall Finish, partly covered by Guide D 3323,
- (2) Type 2 Interior Gloss and Semigloss Wall and Trim Enamels, partly covered by Guide D 3425,
- (3) Type 3 Exterior House and Trim Coatings, now covered by Guide D 2932, and
- (4) Type 4 Floor Enamel, Exterior and/or Interior, now covered by Guide D 3383.

2.1 Each is intended for application by brushing, rolling, spraying, or other means to the materials appropriate for its use, which may include wood, plaster, wallboard, masonry, metal, previously painted surfaces, and other architectural substrates.

Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer and Related Products^{2,3,4}
- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester⁵
- D 154 Guide for Testing Varnishes²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints^{2,3}
- D 215 Methods of Chemical Analysis of White Linseed Oil Paints²

¹ This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coating and Materials and is the direct responsibility of Subcommittee D 01.42 on Architectural Finishes.

Current edition approved Nov. 30, 1990. Published January 1991.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 06.03.

⁵ Annual Book of ASTM Standards, Vols 05.01, 06.01, and 06.03.

D 344 Test Methods for Relative Hiding Power of Paints by the Visual Evaluation of Brushouts²

D 358 Specification for Wood to be Used as Panels in Weathering Tests of Coatings²

D 522 Test Method for Mandrel Bend Test of Attached Coatings²

D 523 Test Method for Specular Gloss²

D 562 Test Method for Consistency of Paints using the Stormer Viscometer²

D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasive²

D 659 Test Method of Evaluating Degree of Chalking of Exterior Paints²

D 660 Test Method for Evaluating Degree of Checking of Exterior Paints²

D 661 Test Method for Evaluating Degree of Cracking of Exterior Paints²

D 662 Test Method for Evaluating Degree of Erosion of Exterior Paints²

D 772 Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints²

D 968 Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive²

D 1006 Practice for Conducting Exterior Exposure Tests of Paints on Wood²

D 1014 Test Method for Conducting Exterior Exposure Tests of Paints on Steel²

D 1038 Definitions of Terms Relating to Veneer and Plywood⁶

D 1208 Test Methods for Common Properties of Certain Pigments³

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems²

D 1296 Test Method for Odor of Volatile Solvents and Diluents⁴

D 1308 Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes²

D 1475 Test Method for Density of Paint, Varnish, Lacquer and Related Products²

D 1543 Test Method for Color Permanence of White Architectural Enamels²

D 1554 Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials⁶

D 1640 Test Methods for Drying, Curing or Film Formation of Organic Coatings at Room Temperature²

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials⁷

⁶ Annual Book of ASTM Standards, Vol 04.09.

⁷ Annual Book of ASTM Standards, Vol 14.02.

- D 1849 Test Method for Package Stability of Paint²
D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer²
D 2197 Test Method for Adhesion of Organic Coatings by Scrape Adhesion²
D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates²
D 2245 Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints⁴
D 2369 Test Method for Volatile Content of Coatings²
D 2370 Test Method for Tensile Properties of Organic Coatings²
D 2371 Test Method for Pigment Content of Solvent-Reducible Paints²
D 2372 Method of Separation of Vehicle from Solvent-Reducible Paints²
D 2486 Test Method for Scrub Resistance of Interior Latex Flat Wall Paints²
D 2621 Method for Infrared Identification of Vehicle Solids from Solvent-Reducible Paints²
D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings²
D 2698 Method for Determination of the Pigment Content of Solvent-Reducible Paints by High Speed Centrifuging²
D 2805 Test Method for Hiding Power of Paints by Reflectometry²
D 2932 Guide for Testing Exterior Solvent-Reducible House and Trim Coatings²
D 3278 Test Methods for Flash Point of Liquids by Setflash Closed Apparatus⁴
D 3323 Guide for Testing Interior Solvent-Reducible Flat Wall Paints²
D 3359 Test Methods for Measuring Adhesion by Tape Test²
D 3383 Guide for Testing Solvent-Reducible Floor Paints²
D 3425 Guide for Testing Solvent-Reducible Interior Semigloss Wall and Trim Enamels²
D 3450 Test Method for Washability Properties of Interior Architectural Coatings²
D 3456 Practice for Determining by Exterior Exposure Tests Susceptibility of Paint Films to Microbiological Attack²
D 3730 Practice for Testing High-Performance Interior Architectural Wall Coatings²
D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²
D 3928 Test Method for Evaluation of Gloss or Sheen Uniformity²
D 4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method²
D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser²
D 4062 Test Method for Leveling of Paints by Draw-Down Method²
D 4213 Test Method for Wet Abrasion Resistance of Interior Paints²
D 4214 Test Methods for Evaluating Degree of Chalking of Exterior Paint Films²
D 4287 Test Method for High Shear Viscosity of Paints and Varnishes by the ICI Cone/Plate Viscometer²
D 4400 Test Methods for Sag Resistance of Paints Using Multinotch Applicator²
D 4585 Practice for Testing Water Resistance of Coatings Using Controlled Condensation²
D 4707 Test Method for Measurement of Paint Spatter Resistance to Roller Application²
D 4828 Test Method for Practical Evaluation of Washability of Organic Coatings²
E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Reflectometry⁸
E 105 Practice for Probability Sampling of Materials⁷
E 313 Test Method for Indexes of Whiteness and Yellowness of Near-White Opaque Materials⁷
2.2 *U.S. Federal Standard:*
U.S. Federal Test Method Standard No. 141⁹
2112 Application by Roller
2131 Application of Sprayed Films
2141 Application of Brushed Films
3011 Condition in Container
4203 Reducibility and Dilution Stability
4401 Odor Test
4421 Absorption Test
4541 Working Properties and Appearance of Dried Film
2.3 *Other Document:*
Paint/Coatings Dictionary of the Federation of Societies for Coatings Technology¹⁰
3. **Terminology**
3.1 For definitions of terms in this guide refer to Definitions D 16, D 1554 and D 1038 and to the FSCT Paint/Coatings Dictionary.
4. **Conditions Affecting Solvent-Reducible Coatings**
4.1 *Substrate Type*—The substrate to be painted can affect not only the application properties of a coating, such as gloss and uniformity, but is also a factor in determining the type of coating to use. For instance, low-gloss wall finishes do not have the abrasion resistance required on floors, whereas finishes intended only for interior service probably do not have adequate resistance to weather factors. Other factors are the type and quality of wood or wood composite (plywood, particle board or hardboard), the type, quality and alkalinity of concrete, plaster and joint cement systems, and the type and condition of any previous coatings.
4.2 *Substrate Conditions*—Conditions such as porosity, hardness or, in the case of unpainted concrete, alkalinity determine the kind of coating that can be applied. The condition of previously painted substrates, such as degree of chalk, presence of grease, dirt, and mold, film adhesion and porosity, and the general condition of the previous coating, all influence the performance of coatings. Smoothness of the substrate affects the spreading rate, final appearance, and texture.

⁸ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

⁹ Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

¹⁰ Available from the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422.

TABLE 1 List of Standards in Sectional Order

Property (or related test)	Section	ASTM Standard	Federal Test Method Standard 141
Sample	6.2	D 3925	1022
Paint Properties			
Skinning	7.1	D 154	3021
Condition in container	7.2		3011
Coarse particles and foreign matter	7.3	D 185	
Density or Weight per gallon	7.4	D 1475	
Fineness of dispersion	7.5	D 1210	
Consistency	7.6	D 562	
Rheological properties of non-Newtonian liquids	7.7	D 2196, D 4287	
Flash point	7.8	D 93, D 3278	
Color	7.9	D 1295	4401
Absorption	7.10		4421
Colorant acceptance	7.11		
Dilution stability	7.12		4203
Package stability	7.13	D 1849	
Paint Application and Film Formation			
Application properties	8.1		4541
Brush application	8.1.1		2141
Roller application	8.1.2		2112
Roller spatter	8.1.2.1	D 4707	
Spray application	8.1.3		2131
Drying properties	8.2	D 1640	4061
Levelling properties	8.3	D 4062	
Sag resistance	8.4	D 4400	4494
Touch up uniformity	8.5	D 3928	
Appearance of Dry Film			
Color difference by visual comparison	9.1	D 1729	
Color difference by instrumental measurement	9.2	D 2244	
Directional reflectance	9.3	E 97	
Gloss	9.4		
Gloss, 60°	9.4.1	D 523	
Sheen (85° gloss)	9.4.2	D 523	
Hiding power	9.5	D 344, D 2805	
Yellowness index	9.6	E 313	6131
Properties of Dry Film			
Interior and Exterior Coatings	10.1		
Abrasion resistance	10.1.1	D 658, D 968, D 4080	6192
Adhesion	10.1.2	D 2197, D 3359	
Flexibility	10.1.3	D 522, D 2370	6221 ^A
Interior Coatings	10.2		
Color change of white enamels	10.2.1	D 1543	6192
Washability and cleansability	10.2.2		
Washability	10.2.2.1	D 2486, D 4213	
Cleansability	10.2.2.2	D 3450, D 4828	6141 ^B
Resistance to household chemicals	10.2.3	D 1308	
Exterior Coatings	10.3		
Blister resistance	10.3.1	D 4585	
Exposure resistance	10.3.2	D 1066, D 1014	
Chalking	10.3.2.1	D 658, D 4214	
Checking	10.3.2.2	D 660	
Cracking	10.3.2.3	D 661	
Erosion	10.3.2.4	D 662	
Flaking	10.3.2.5	D 772	
Mildew resistance	10.3.3	D 3456	
Fume resistance	10.3.4		
Coating Analysis			
Coating analysis	11.1	D 215	
Volatile content	11.2	D 2369	
Nonvolatile volume content	11.3	D 2697	
Water content	11.4	D 1208, D 4017	4081
Pigment content	11.5	D 2371	4021
Pigment analysis	11.6	D 215	7261
Nonvolatile vehicle content	11.7	D 215	4053
Vehicle separation	11.8	D 2372	
Nonvolatile vehicle identification	11.9	D 2621, D 2245	

^A Equivalent only to Method B of Test Method D 522.

^B Except for scrub medium.

^C 6271 is not equivalent.

4.3 *Substrate Aspects of the Building*—If construction defects or defects due to age are such that excessive moisture from the inside or the outside makes its way through the

substrate or if the substrate is in direct contact with damp ground, blistering, flaking or peeling may result.

4.4 Preparation of previously painted substrates including

TABLE 2 Alphabetical List of Properties

Property (or related test)	Section	ASTM Standard	Federal Test Method Standard 141
Abrasion Resistance	10.1.1	D 658, D 968, D 4060	6192
Absorption	7.10		4421
Adhesion	10.1.2	D 2197, D 3359	
Analysis	11.1	D 215	
Application properties	8.1		4541
Brush application	8.1.1		2141
Chalking	10.3.2.1	D 659, D 4214	
Checking	10.3.2.2	D 660	
Cleansability	10.2.2.2	D 3450, D 4828	6141 ^A
Coarse particles and foreign matter	7.3	D 185	
Colorant acceptance	7.11		
Color change	10.2.1	D 1543	6192
Color difference by instrumental measurement	9.2	D 2244	
Color difference by visual comparison	9.1	D 1729	
Condition in container	7.2		3021
Consistency	7.6	D 562	
Cracking	10.3.2.3	D 661	
Density or weight per gal	7.4	D 1475	
Dilution stability	7.12		4203
Drying properties	8.2	D 1640	4061
Erosion	10.3.2.4	D 662	
Fineness of Dispersion	7.5	D 1210	
Flaking	10.3.2.5	D 772	
Flash point	7.8	D 93, D 3278	
Flexibility	10.1.3	D 522, D 2370	6221 ^B
Fume resistance	10.3.4		
Gloss	9.4		
Gloss, 60°	9.4.1	D 523	
Hiding power	9.5	D 344, D 2805	
Levelling properties	8.3	D 4062	
Mildew resistance	10.3.3	D 3456	^C
Nonvolatile vehicle content	11.7	D 215	4053
Nonvolatile vehicle identification	11.9	D 2621, D 2245	
Nonvolatile volume content	11.3	D 2697	
Odor	7.9	D 1296	4401
Package stability	7.13	D 1849	
Pigment analysis	11.6	D 215	7261
Pigment content	11.5	D 2371	4021
Resistance to household chemicals	10.2.3	D 1308	
Rheological properties of non-Newtonian liquids	7.7	D 2196, D 4287	
Roller application	8.1.2		2112
Roller spatter	8.1.2.1	D 4707	
Sag resistance	8.4	D 4400	4494
Sampling	6.2	D 3925	1022
Sheen (85° gloss)	9.4.2	D 523	
Skimming	7.1	D 154	3021
Spray application	8.1.3		2131
Touch up uniformity	8.5	D 3928	
Vehicle separation	11.8	D 2372	
Volatile content	11.2	D 2369	
Washability	10.2.2.1	D 2486, D 4213	
Water content	11.4	D 1208, D 4017	4061
Yellowness index	9.6	E 313	6131

^A Except for scrub medium.

^B Equivalent only to Method B of D 522.

^C 6271 is not equivalent.

detergent cleaning, solvent cleaning, and sanding.

4.5 Type and quality of primer or undercoat and time of drying before topcoating.

4.6 Environmental conditions such as temperature and humidity at the time of coating application and during drying.

4.7 Environmental conditions after application, both general for the area and specific, such as under eaves, behind shrubbery, northside and southside exposure.

5. Selection of Tests

5.1 Because the conditions to which a coating is subjected vary with (a) the surface type: wall, floor, ceiling, and (b) the

service environment: exterior or interior, specialized types of solvent-borne coatings have been developed for the different locations. The recommended test methods presented in Tables 1 and 2 cover practically all the properties of solvent-reducible coatings but all of them are not required with each type. Coatings intended for exterior use only or both exterior and interior use require certain properties not relevant to those for interior use only. Selection of the methods to be followed must be governed by experience and the requirements in each individual case, together with agreement between the purchaser and the seller.

5.2 The purchaser should first determine the properties a coating should have and then select only those test methods

Method
141

measure or evaluate those properties. After selecting the tests, the purchaser should then decide which properties are the most important and establish the requirements accordingly. Since coating properties frequently tend to oppose each other, such as low sheen versus cleanability, some properties may need to be less emphasized if others are to be accentuated. This balance of properties must be considered when selecting the tests and establishing the requirements. The significance of the tests in the normal range of values are presented in the different sections, in most cases.

This guide does not indicate relative importance of the tests nor does it recommend specific test values. These properties very important to one purchaser may be less so to another.

Sampling

Prior to sampling, the condition of the container should be checked since damage to it may cause evaporation, leaking, or other undesirable effects on the coating.

2. Sample in accordance with Practice D 3925. Determine the density in pounds per gallon (kilograms/litre) in accordance with Test Method D 1475. Continue sampling until determining density until successive results agree within 0.1 lb (45 g) or as agreed upon between the purchaser and supplier. Then take samples for testing.

3. Specify the amount required for a representative sample, the package sizes, and an identification code. A 1 U.S. gal (or 4-L) sample is usually sufficient for the recommended tests, but for guidance in selecting a sampling method consult Practice E 105.

Liquid Coating Properties

1. *Skinning*—Coatings that contain a binder that dries by oxidation may be subject to skin formation in a partially-filled can. Since skins are insoluble in the material they must be removed before use. The referenced test in a partially-filled container indicates the tendency of the material to skin. A typical minimum time for skinning in accordance with this method is 48 h. Examine the original sample for skins, both on and below the surface. Using a well-mixed, skin-free portion of the sample, perform a skinning test in accordance with Guide D 154.

2. *Condition in Container*—Thickening, settling, and separation are undesirable and objectionable if material that has been stored cannot be readily reconditioned and made suitable for application with a reasonable amount of stirring. The referenced method covers procedures for determining changes in properties after storage and lists characteristics that are undesirable and objectionable in a stored paint. Determine condition in the container in accordance with Test Method 3011 of Federal Test Method Standard No. 141. See also 7.13, Package Stability.)

3. *Coarse Particles and Foreign Matter*—Liquid coatings must be free of coarse particles and foreign matter to be able to form uniform films of good appearance, a typical maximum being 0.5 weight % of the total material. The referenced method with a 325-mesh (45- μ m) screen gives the percent of these particles. Determine content of coarse particles and foreign matter in accordance with Test Methods D 185.

7.4 *Density or Weight per Gallon*—The density measured in pounds per gallon (kilograms per litre = g/mL) is used to ensure product uniformity from batch to batch, provides a check against the theoretical weight calculated from the formula, and is useful for determining the similarity of two samples. The referenced method gives a procedure for measuring the density of the coating at a specified temperature. Most interior paints have densities of about 10 to 12 lb/gal (1.2 to 1.4 kg/L). Determine density in accordance with Test Method D 1475, using a calibrated weight per gallon cup.

7.5 *Fineness of Dispersion*—Generally, the more finely a pigment is dispersed, the more efficiently it is being utilized. One method for measuring the degree of dispersion (commonly referred to as "fineness of grind") is to draw the liquid coating down a calibrated tapered groove varying in depth from 4 to 0 mils (100 to 0 μ m) (0–8 Hegman units). The depth at which continuous groupings of particles or agglomerates, or both, protrude through the surface of the wet film is taken as the fineness of dispersion value. Higher readings in Hegman units or lower readings in mils or micrometres indicate finer dispersion. Low sheen finishes may have a dispersion value of 2 mils (50 μ m or 4 Hegman) while gloss enamels might be near zero (8 Hegman) indicating that the pigment agglomerates are too small to be detected by the referenced method. Determine fineness of dispersion in accordance with Test Method D 1210.

7.6 *Consistency (Low-Shear Viscosity)*—Consistency is important, relating to application and flow, and should fall within a stated range for satisfactory reproduction of a specific formula. While consistency is an important property it does not determine the quality of a coating and should be used mainly to ensure product uniformity. In the referenced method, consistency is defined as the load in grams to produce a specified rate of shear. The load value is frequently converted to Krebs units (KU) and the Stormer Consistency reported on that basis. Although the consistency of most solvent-borne house and trim coatings is about 150 to 300 g/100 revolutions (72 to 95 KU), a much wider range is possible because of the great variation that may occur in the rheological properties of these paints. Enamels for professional painters are usually formulated at a higher consistency range than consumer enamels. Typical ranges are 75 to 90 KU for consumer enamels and 90 to 100 for professional painter enamels. Two paints of the same consistency may have quite different rheological properties during application. Determine the consistency in accordance with Test Method D 562.

7.7 *Rheological Properties of Non-Newtonian Materials*—Rheological properties are related to application and flow characteristics of the liquid coating. The referenced methods cover the determination of rheological properties and are particularly suited for coatings that display thixotropic characteristics. They actually measure viscosity under different shear rates. In Test Method D 4287 the rate is similar to that occurring during brush application so that the measured viscosity is related to brush drag, spreading rate and film build. Determine rheological properties in accordance with Test Methods D 2196 or D 4287, or both.

7.8 *Flash Point*—Organic solvents used in these coatings have characteristic temperatures at which they support

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combustion. This temperature is known as the flash point and is often used for danger classification in shipping by common carrier. It is also used to determine conditions of storage to meet fire regulations and the safety requirements of the U.S. Occupational Safety and Health Act (OSHA). Determine flash point in accordance with Test Methods D 93, Part B, or D 3278.

7.9 Odor—Some solvent combinations produce obnoxious odors, particularly when painting indoors with inadequate ventilation and at elevated temperatures. Interior solvent-borne coatings usually contain low-odor or odorless mineral spirits. Nevertheless they should be evaluated to ensure that they are acceptable. Test for odor in accordance with Method 4401 of Federal Test Method Standard No. 141. Although not specifically designed for liquid coatings Test Method D 1296 may be used with the solvent-reducible type.

7.10 Absorption—On porous surfaces, binder penetration can result in pigment volume concentration changes as the film dries. This may cause appearance to vary. The referenced method provides a rough measure of the wetting and penetrating properties of the binder on a porous surface. Determine the absorption in accordance with Method 4421 of Federal Test Method Standard No. 141.

7.11 Colorant Acceptance—Tintability of white bases with colorants of standardized tinting strength is a trade requirement. If tinting colors are not adequately compatible with tint bases, lighter, darker, or nonuniform shades of colors are produced. There is no accepted ASTM test method at present. Test methods may be agreed upon between the purchaser and seller.

7.12 Dilution Stability—Dilution with a specified thinner shows whether the materials are compatible and whether the reduced coating is stable. Consequently the suggested diluent should be readily incorporated into the coating without excessive stirring or shaking. The referenced method evaluates the stability of the material that has been reduced by a given amount or to a specified viscosity. Determine dilution stability in accordance with Method 4203 of Federal Test Method Standard No. 141.

7.13 Package Stability—Since paints cannot normally be used immediately after manufacture, they must remain stable in the can for some time. At normal temperatures most paints can be stored for over a year with little change in properties. Although indications of long term package stability can usually be obtained in several days or weeks at an elevated temperature, such as 122°F (50°C) or 140°F (60°C), occasionally the results of the accelerated test do not agree with those at prolonged normal storage conditions. The referenced method predicts the change in consistency and certain other properties of packaged paint when stored at temperatures above freezing. Determine package stability in accordance with Test Method D 1849.

8. Coating Application and Film Formation

8.1 Application Properties—Application or working properties of a paint are generally compared to a standard or described by requirements in the product specification. Determine working properties in accordance with Method 4541 of Federal Test Method Standard No. 141.

8.1.1 Brush Application—Brushed films should be smooth and free of seeds and on vertical surfaces should

show no sagging, color streaking, or excessive brush marks. Brush drag should not be excessive although some degree of drag may be desirable for adequate film thickness application. Wall finishes are tested on vertical surfaces and floor enamels on horizontal surfaces, although evaluation of the latter on vertical surfaces may be necessary to determine performance on stair risers, railings, posts, etc. The referenced method covers a means for the determination of the brushing properties of a coating. Even though the test is subjective someone experienced in the art can produce quite consistent results, particularly in the evaluation of drag qualities. Determine the brushing properties in accordance with Method 2141 of Federal Test Method Standard No. 141.

8.1.2 Roller Application—Both wall and floor coatings are frequently applied by roller. This type of application tends to produce some stipple pattern. The referenced method covers the evaluation of a material's characteristics when applied by roller. Determine roller coating properties in accordance with Method 2112 of Federal Test Method Standard No. 141.

8.1.2.1 Some coatings spatter more than others when applied by roller. The degree to which a paint spatters when roller applied can be determined by the density of the spatter. In the referenced method a specially designed notched spool is rolled through a film of the test material that has been applied to a plastic panel. Any spatter generated falls upon a catch paper and after drying is rated against photographic standards. This procedure eliminates the influence of the roller cover, thus determining the spattering characteristics of the paint alone. Determine spatter resistance in accordance with Test Method D 4707.

8.1.3 Spray Application—Architectural coatings are sometimes applied by spray. Both air and airless spray are used on commercial work. Determine the spray application properties in accordance with Method 2131 of Federal Test Method Standard No. 141. Manual application is very subjective and should be performed only by an individual skilled in the art of using spray equipment.

8.2 Drying Properties—The drying time of a coating is important in determining when a freshly painted room, floor or stair may be put back in use. Slow drying may result in dirt or insect pickup resulting in a poor appearance or, if on an exterior surface, rain or dew may cause a nonuniform appearance. The drying time of a coating is determined by its composition and by atmospheric conditions during drying. Typical drying times for enamels are ½ to 2 h set-to-touch and 18 h dry hard under normal temperature and humidity conditions. Some coatings lose drying speed during storage in the container. Any of the several methods for determining the various stages of film formation in the drying or curing of organic coatings may be used. For example, if two coats are specified the determination of "dry-to-recoat" time is important. Determine appropriate drying time(s) in accordance with Test Methods D 1640.

8.3 Levelling Properties—Levelling is an important property when smooth, uniform surfaces are to be produced, as it affects hiding and appearance. The referenced method covers the relative levelling characteristics of liquid coatings. Determine levelling in accordance with Test Method D 4062.

8.4 Sag Resistance—Some coatings sag and form curtains

before the film sets. This is an important property particularly for semigloss and gloss enamels because sagging results in unsightly film appearance. Measure sag resistance in accordance with Test Methods D 4400.

8.5 Touch-Up Uniformity—After a paint has dried, areas where less material was applied sometimes become noticeable. If the paint has suitable touch-up properties, additional material can be applied to these areas only, instead of finishing the complete wall. The color, gloss, and levelling of the touched-up areas and the previously coated area should be uniform. Differences in these properties are often caused by short wet-edge time, poor levelling on recoat, and pigment orientation or flotation during and after application. Determine touch-up properties in accordance with Test Method D 3928.

9. Appearance of Dry Film

9.1 Color Differences by Visual Comparison—Visual comparison of colors is fast and often acceptable although numerical values are not obtained. The referenced method covers the spectral, photometric, and geometric characteristics of light source, illuminating and viewing conditions, sizes of specimens, and general procedures to be used in the visual evaluation of color differences of opaque materials relative to their standards. Determine color difference in accordance with Practice D 1729.

9.2 Color Differences Using Instrumental Measurements—The difference in color between a product and its standard can be measured by instrument. Generally the tolerance is agreed upon by the purchaser and seller and may also be required if a product specification is involved. Color measuring instruments provide numerical values that can be compared to subsequent measurements. The referenced method covers the calculation of instrumental determinations of small color differences observable in daylight illumination between nonfluorescent, nonmetameric, opaque surfaces such as coated specimens. If metamerism is suspected, visual evaluation (9.1) should be used to verify the results. Calculate in accordance with Test Method D 2244 the color differences that have been measured instrumentally.

9.3 Directional Reflectance—This property is a measure of the appearance of lightness of a coating. It is usually assigned a value in specifications for white and pastel shades, a typical minimum reflectance value being 86 % for white wall paint. In the referenced method the directions of illumination and viewing are specified so as to eliminate the effect of gloss. Determine daylight directional reflectance in accordance with Test Method E 97.

9.4 Gloss—This property is a measure of the capability of a coating surface to reflect light in a mirror-like (specular) manner, that is, light strikes the surface and is reflected at the equal but opposite angle. In the referenced method the numerical gloss units are the ratio of light reflected by a specimen to that reflected by the primary standard black glass that is assigned a gloss value of 100. The gloss of some coatings varies greatly with the angle of incidence so that a complete description of their gloss would require measurements over a wide range of angle. In practice, the gloss of architectural finishes is adequately characterized by measurements at 60° or 85°, or both, from a line perpendicular (normal) to the surface. The 85° angle is a very low

("grazing") angle (5°) of illuminating and viewing the surface and the gloss at this angle is called "sheen". Attempts to standardize the levels of gloss associated with the several descriptive terms have not been very successful since the gloss scale is continuous with no distinct boundaries. Hence, there is some overlap at the ends of some gloss classifications in common usage.

9.4.1 Gloss, 60°—Semigloss enamels are particularly sensitive to enamel hold-out of primers and undercoats. Low or uneven gloss readings are indicative of this defect. Oil house paints are typically in a 60° gloss range from 30 to 70 while trim enamels are from 70 to 90. Floor enamels generally have a high (90+) gloss reading when first applied but this decreases with time and traffic. Interior semigloss enamels after drying 48 h are typically in the range from 40 to 70 but measurements taken shortly after drying should be repeated after one week because the gloss can drop considerably in the first few days of drying. Determine the 60° gloss in accordance with Test Method D 523.

9.4.2 Sheen (85° Gloss)—Although low-gloss paints with good uniformity of appearance at low angles of viewing often have little sheen while those with good cleansability usually have moderate sheen, this is not always the case so that sheen should not be used as a measure of other paint properties. Most flat wall paints have a sheen of about 1 to 10. The referenced method, using the 85° geometry, is useful in characterizing the low-angle appearance of low-gloss coatings. Determine the sheen (85° gloss) in accordance with Test Method D 523.

NOTE 3—This property is of particular interest with flat to low-gloss finishes.

9.5 Hiding Power—Hiding power is a measure of the ability of a coating to obscure the substrate and is usually expressed as the spreading rate for a specified level of opacity. It is, however, dependent on uniformity of film thickness, which in practical applications is influenced by flow, levelling and application properties of the coating. Test Method D 2805 is precise and gives an absolute rather than a comparative result. Paint is applied with an applicator bar to minimize the effects of flow and levelling, film thickness is rigorously measured, and film opacity is determined instrumentally. Test Method D 344 is a practical test in which paint is applied with a brush, wet-film thickness is approximately controlled by spreading rate, and hiding power is evaluated visually by comparison with a standard paint, but results are affected by flow and levelling of the materials. Determine hiding power in accordance with Test Methods D 344 or D 2805.

9.6 Yellowness Index—The referenced method is used for white or near white specimens to determine color departure from white toward yellow when first applied. Determine the yellowness index in accordance with Method E 313. (See also 10.2.1.)

10. Properties of the Dry Film

10.1 Interior and Exterior Coatings:

10.1.1 Abrasion Resistance—Abrasion resistance is a measure of the ability of a dried film to withstand wear from foot traffic and marring from objects rolled or pulled across

the surface. In the referenced methods, dry abrasive is applied to a coated panel using the force of gravity or a jet blast for free-flowing abrasive or a weighted wheel for abrasive embedded in a resilient rubber matrix. Determine dry abrasion resistance in accordance with Test Methods D 658, D 968 or D 4060. (See 10.2.2.1 for wet abrasion resistance.)

NOTE 4—Because of the poor reproducibility of abrasion test methods, testing should be restricted to only one laboratory when numerical abrasion resistance values are to be used. Interlaboratory agreement is improved significantly when rankings are used in place of numerical values.

10.1.2 *Adhesion*—Adhesion is the property of a film that resists removal from the substrate. It is an important property in a paint. Determine adhesion in accordance with Test Method D 2197 or D 3359, or both.

10.1.3 *Flexibility*—Elongation is a measure of the flexibility of a coating film. Generally, gloss house paints and trim enamels have no problems in passing a mandrel bend test at $\frac{1}{8}$ in. (3.2 mm). However, interior flat and eggshell finishes may pass only a $\frac{1}{2}$ -in. (12.7-mm) bend. For exterior coatings Test Method D 2370 is a much more discriminating method.¹¹ Determine flexibility in accordance with Methods D 522 or elongation with D 2370.

10.2 Interior Finishes:

10.2.1 *Color Change of White Architectural Enamels*—Color permanence is an important characteristic for interior white enamels. Lack of permanence is usually caused by after-yellowing. The referenced method attempts to accelerate color change by exposing coatings to dry sulfur dioxide. Determine resistance to color change in accordance Test Method D 1543.

10.2.2 *Washability and Cleansability*—The capability of satisfactorily removing marks without damaging the film is essential for good performance of interior finishes. A coating may be washable, that is, unaffected by the detergent solution, but may not have good cleansability. Frequently the difference between the two terms "cleansability" and "washability", is not clearly understood so that there is confusion as to what is really being tested; for example, the title of Test Method D 3450. Cleansability is evaluated by applying one or more stains and soils and determining how readily they are removed. Washability is evaluated by determining the resistance of the film to wet erosion either by visual assessment or measured film loss. In general, the precision of both types of test is poor because several properties, such as hardness, water and detergent resistance, cohesion and adhesion, are involved and the end point, except for the wet abrasion method, is rather indefinite.

10.2.2.1 *Washability (Also referred to as Scrubbing or Wet Abrasion Resistance)*—The scrubbing method, Test Method D 2486, developed for interior latex flat wall paints can be applied to coatings of almost any type. In it the coating is applied to a black plastic panel that, during scrubbing with a nylon brush and abrasive cleaning agent, is raised by a narrow shim to concentrate the test area. The number of back-and-forth strokes (cycles) required to remove the film over the shim is determined. The wet-abrasion method, Test

Method D 4213, is similar except that a sponge is used in place of the bristle brush while the shim is not used. This method also provides for the use of a nonabrasive medium with paints having very low abrasion resistance. The weight or volume loss per 100 cycles to erode the film almost to exposure of the black substrate is the measure of scrub resistance. Evaluate washability, as described just, in accordance with Test Methods D 2486 or D 4213.

10.2.2.2 *Cleansability*—The older referenced method, Test Method D 3450, is similar to the wet-abrasion method, Test Method D 4213, except that the sponge is used with either the nonabrasive or abrasive cleaning agent to remove a carbon black-oil stain. The ability to remove the stain is expressed as the ratio (in percent relative) of the reflectance of the cleaned area to that of the area before application of the stain. In Test Method D 4828, referred to as a "practical" test, numerous staining and soiling agents found in service and commercial abrasive or nonabrasive cleaners as well as the standardized cleaning agents can be used. In the current edition the films are cleansed only manually with the result that the method is not suitable for interlaboratory testing. Evaluate ease of removability in accordance with Test Methods D 3450 or D 4828.

10.2.3 *Resistance to Household Chemicals*—An important property of interior coatings is the ability to resist spotting, softening or removal when subjected to household chemicals or strong cleaners. Determine resistance to these chemicals in accordance with Test Method D 1308.

10.3 Exterior Coatings:

10.3.1 *Blister Resistance*—Blister resistance is the ability of a dry film on wood to resist the formation of blisters caused by water from the wood substrate. In practice water can come from either the interior of a home or from structural defects that permit entry of exterior water behind the wood. Moisture blister resistance can be qualitatively evaluated in a laboratory test. Determine resistance to moisture blistering in accordance with Practice D 4585.

10.3.2 *Exposure Resistance*—If the coating is intended for exterior use, evaluation of the resistance to weathering may be required. In conducting exterior exposures follow Practice D 1006 for wood substrates or Test Method D 1014 for steel.

10.3.2.1 In establishing exterior performance on wood, use the panels described in Specification D 358.

10.3.2.2 *Degree of Chalking*—Determine the rating using Methods D 659 or D 4214.

10.3.2.3 *Degree of Checking*—Determine the rating using Test Method D 660.

10.3.2.4 *Degree of Cracking*—Determine the rating using Test Method D 661.

10.3.2.5 *Degree of Erosion*—Determine the rating using Test Method D 662.

10.3.2.6 *Degree of Flaking*—Determine the rating using Test Method D 772.

10.3.3 *Mildew Resistance*—Virtually all exterior paints are subject to microbiological discoloration on the surface with time. This is especially true in warm, moist climates. Determine mildew resistance in accordance with Practice D 3456.

10.3.4 *Fume Resistance*—Some paints exhibit a change in appearance (usually color) when exposed to air containing certain sulfur compounds, notably hydrogen sulfide and

¹¹ H. E. Ashton, *Journal of Coatings Technology*, Vol 51, June 1979, p. 41.

dioxide. This type of atmosphere may be present near material or other polluted areas and can cause paint to discolor or darken in as little time as overnight. There are no Federal test methods for evaluating this color change, but one procedure used by the industry is as follows:

11.4.1 Apply a sufficient number of coats of the paint to glass plates to hide the surface completely, allow to dry, and expose one in a moist atmosphere of hydrogen peroxide for 18 h. Compare the color with the unexposed plate. The exposed plate should be practically no darker than the unexposed one. The color difference should not exceed that of plates that have been coated with a paint made with titanium dioxide pigment, lead-free zinc oxide, raw or refined linseed oil, and sufficient cobalt added for drying, similarly treated.

Coating Analysis

11.1 *Coating Analysis*—If a specification requires certain materials or certain components in a given amount then analysis is needed to determine whether the specified components are present and in what amounts. Analysis is primarily a measure of uniformity and does not necessarily establish quality that can also be greatly affected by manufacturing techniques. No single schematic analysis is comprehensive enough to cover the wide variety of paint compositions. Select test procedures from Methods D 215 and other ASTM methods that are pertinent to the components of solvent-borne coatings.

11.2 *Volatile Content*—The percent of volatile matter is a measure of the amount of a film lost as it dries. This quantity is not necessarily indicative of the quality of the coating. It is useful, however, for determining the similarity of two coatings. The referenced method covers the determination of volatile content of solvent- and water-reducible coatings. The quantity determined subtracted from 100 % gives the volatile content of the coating. Determine the volatile content in accordance with Test Method D 2369.

11.3 *Nonvolatile Content (Volume Percent)*—The nonvolatile content by volume is a useful figure in calculating coverage or spreading capacity per gallon (or litre) at a specified dry-film thickness. Determine nonvolatile content by volume percent in accordance with Test Method D 2697.

11.4 *Water Content*—If too much water is incorporated in a solvent-reducible coating it may retard the drying at high relative humidities. The amount of water in a coating is required in the calculation of the volatile organic content (VOC) of coatings. One referenced method covers the determination of water in paint and related materials by distilling with a volatile solvent. The newer method utilizes the Karl Fischer reaction. Determine water in accordance with Test Methods D 1208 or D 4017.

11.5 *Pigment Content*—Pigment provides the hiding and color and influences many other properties of a coating. The referenced method describes the procedure for the quantitative separation of the vehicle from the pigment in solvent-reducible coatings. It is used to measure the weight percent pigment in the paint. Determine the percent pigment content in accordance with Test Method D 2371.

11.6 *Pigment Analysis*—The analysis of pigment may be required if the product is covered by a specification or upon agreement between the purchaser and seller. Analyze the pigment in accordance with selected test procedures from Methods D 215 and other appropriate ASTM methods.

11.7 *Nonvolatile Vehicle Content*—The nonvolatile vehicle is the film-forming portion of a coating excluding the pigment. Water, volatile thinner, and pigment are determined and their sum subtracted from 100 % to give the binder content in accordance with Methods D 215. The vehicle may be separated for further analysis.

11.8 *Separation of Vehicle*—The recommended procedure describes this separation for certain solvent-borne coatings. If desired or required, separate the vehicle in accordance with Method D 2372.

11.9 *Identification of Nonvolatile Vehicle*—The type of binder used in a coating has a great influence on its properties. The referenced method covers the qualitative characterization or identification by infra-red spectroscopy of separated nonvolatile vehicle. It is useful in detecting uniformity, batch to batch, and the presence of adulterants. Identify the nonvolatile vehicle in accordance with Method D 2621. The composition of this vehicle can be further broken down into the types of oils present. If desired determine the identity of oils in accordance with Method D 2245.

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tion was found to be 0.5 rating units with 36 df. Based on these values the following criterion should be used for judging the acceptability of results at the 95 % confidence level:

10.1.1 *Reproducibility*—Two single results obtained by operators in different laboratories should be considered

suspect if they differ by more than 1.5 rating units.

11. Keywords

11.1 architectural paints and coatings; hiding power; opacity; roller application of paints

APPENDIX

Nonmandatory Information

X1. MANUFACTURER'S DESCRIPTION AND SPECIFICATIONS FOR THE PRACTICAL OPACITY CHARTS USED IN DEVELOPING THIS TEST METHOD⁵

X1.1 Description and Specifications are as follows:

X1.1.1 *Overall Size*—24 by 37½ in. (610 by 953 mm),

X1.1.2 *Test Area*—Size 24 by 36 in. (610 by 914 mm), 6 ft² (5575 cm²),

X1.1.3 *Design*—Alternate stripes of white and neutral grey,

X1.1.4 *Stripe Dimensions*—1½ by 36 in. (36 by 914 mm);

X1.1.5 *Stripe Numbers*—The stripes are numbered 1 through 6 in order of increasing contrast, the numbers being printed on the two 24-in. borders. These borders are ¾-in. (19-mm) wide and are not normally considered a part of the test area.

X1.1.6 *Unnumbered Stripes*—The unnumbered stripes shown in Figure 1 are an integral part of the test area. Their purpose is to favor application uniformity on the numbered stripes by preempting the boundary locations that tend to be less uniformly coated than more centrally located areas.

X1.1.7 *Grey Stripe Reflectances*⁵—The reflectance values of the numbered stripes are selected with the objective that their color differences with respect to the white surround will be in the geometric sequence: 2, 4, 8, 16, 32, and 64 CIELAB units. The defining variable for the grey stripes is not the

reflectance as such, but the ratio of the grey reflectance *G* to the white reflectance *W*. Table X1.1 shows the relevant relationships and production tolerances.

NOTE X1.1—The term "reflectance" as used here refers to the luminous reflectance factor as defined in 4.2 of Method E 97, which may be expressed as a decimal fraction or as a percentage. The latter is numerically the same as the CIE Y-tristimulus value and is the scale reading on most if not all commercial colorimeters.

TABLE X1.1 Relevant Relationships and Production Tolerances

Stripe Number	Color Difference, ^a %	Ratio <i>G/W</i> ^b	Reflectance <i>G</i> , ^c %
1	2 ± 12.5	0.945 ± 0.007	75.6 ± 0.6
2	4 ± 12.5	0.893 ± 0.013	71.4 ± 1.0
3	8 ± 12.5	0.793 ± 0.024	63.5 ± 1.9
4	16 ± 12.5	0.617 ± 0.042	49.4 ± 3.4
5	32 ± 12.5	0.347 ± 0.055	27.8 ± 4.4
6	64 ± 12.5	0.067 ± 0.037	5.3 ± 3.0

^a This is ΔE^*_{ab} , the color difference with respect to the white stripes, expressed in CIELAB units as defined in 6.2 of Method D 2244.

^b $G/W = (1 - \Delta E^*_{ab}/107.7)^2$. This equation is derived from 6.2.1 of Method D 2244 and assumes that for neutral grey versus white, the values ΔE^*_{ab} and ΔL^* are sufficiently close to be considered equal for the purpose of this test method.

^c Values shown here are with a white stripe reflectance *W* of 80 %. These values will vary somewhat with *W*, tolerances for the latter being ± 2 % inter-batch and ± 0.5 % intra-batch.

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Standard Guide for Specifying Inspection Requirements for Coating and Lining Work (Metal Substrates)¹

This standard is issued under the fixed designation D 5161; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This guide is intended to aid the coating specification writer in selecting and specifying the appropriate inspection requirements. It indicates the inspection requirements that may be employed for each of four service environments including mild, moderate, severe, and immersion (see Table

1.2 In order to aid the user in determining when to specify inspection requirements, a relationship between the consequence of failure and the suggested level of inspection is demonstrated (see Fig. 1).

1.3 It is not the intent of this guide to address the selection of protective coating systems, to specify surface preparation and application requirements, or to be a referenced document in a specification.

Referenced Documents

- 2.1 *ASTM Standards:*
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Metal Base²
 - D 1400 Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
 - D 3276 Guide for Painting Inspectors (Metal Substrates)²
 - D 4285 Test Method for Indicating Oil or Water in Compressed Air²
 - D 4414 Practice for Measurement of Wet Film Thickness by Notch Gages²
 - D 4417 Test Method for Field Measurement of Surface Profile of Blast Cleaned Steel²
 - E 337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet and Dry-Bulb Temperatures)³
- 2.2 *NACE Standard:*
- T6A-37 Recommended Practice for Discontinuity, (Holiday Testing of Protective Coating)⁴
- 2.3 *SSPC Standard:*

SSPC-Visl-89 Visual Standard for Abrasive Blast Cleaned Steel⁵

3. Terminology

3.1 *Service Environments Terms are as follows:*

- 3.1.1 *mild service*—indoor or protected outdoor areas not subject to rain, dew or corrosive elements, or both.
- 3.1.2 *moderate service*—areas subject to weather away from coastal or corrosive industrial environments, or both.
- 3.1.3 *severe service*—corrosive/erosive environments including coastal salt laden atmosphere, industrial atmosphere, and high intensity sunlight.
- 3.1.4 *immersion service*—wetted surfaces of tanks, containers, pits, etc and surfaces that are normally wet with condensation or exposed to other corrosive environments.

4. Significance and Use

- 4.1 The requirements for inspection should be addressed in all protective coating and lining work specifications.
- 4.2 This guide may be used by specification writers when selecting and establishing the inspection requirements for coating and lining specifications.
- 4.3 The instructions for using this guide are as follows:
 - 4.3.1 Identify the service environment for the coating or lining system being specified. Read down the column under the appropriate service environment in Table I. Suggested levels of inspection listed on the left are identified with x's.

5. Inspection Requirements

5.1 *Pre-Surface Preparation:*

- 5.1.1 *Contaminants*—Visually verify that oil and grease are removed from the surface prior to surface preparation operations (see Guide D 3276).
- 5.1.2 *Surface Anomalies*—Visually verify that welds and sharp or jagged edges have been suitably prepared for the coating system being used (see Guide D 3276).
- 5.2 *Surface Preparation:*
 - 5.2.1 *Air Supply*—Verify that air supply is clean and dry (see Test Method D 4285).
 - 5.2.2 *Abrasives*—Verify cleanliness and proper size and type to achieve specified profile.
 - 5.2.3 *Ambient Conditions*—Verify that air temperature, humidity, surface temperature, and dew point spread are appropriate (see Test Method E 337).
 - 5.2.4 *Surface Anomalies*—Identify any burrs, slivers, scabs, and weld spatter visible after blasting.

¹ This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

Current edition approved Aug. 15, 1991. Published October 1991.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 11.03.

⁴ Available from National Association of Corrosion Engineers, P.O. Box 18340, Houston, TX 77218.

⁵ Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

TABLE 1 Inspection Requirements

Suggested Inspection Requirements	Service Environments			
	Mild	Moderate	Severe	Immersion
Pre-Surface Preparation:				
Visual for contaminants	X	X	X	X
Visual for surface anomalies			X	X
Surface Preparation:				
Air supply for contaminants			X	X
Abrasives for type and cleanliness			X	X
Ambient conditions and dew point			X	X
Surface anomalies			X	X
Degree of cleanliness	X	X	X	X
Profile		X	X	X
Visual final for contamination			X	X
Coating Application:				
Materials		X	X	X
Mixing			X	X
Equipment		X	X	X
Air supply for contaminants		X	X	X
Ambient conditions and dew point			X	X
Wet film thickness			X	X
Intercoat parameters		X	X	X
Appearance between coats			X	X
Dry film thickness between coats			X	X
Final Acceptance:				
Appearance	X	X		
Visual runs and sags		X	X	X
Visual holidays and pinholes			X	X
Dry film thickness		X	X	X
Holiday tests				X
Repairs			X	X
Final cure			X	X

5.2.5 *Surface Cleanliness*—Verify degree of surface cleanliness (see SSPC-Visl-89).

5.2.6 *Profile*—Verify profile is within specified tolerance using appropriate instruments (see Test Method D 4417).

5.2.7 *Surface Contamination*—Verify that all visible surface contaminants including oil, grease, dust, etc have been removed.

5.3 Coating Application:

5.3.1 *Materials*—Verify that the coating materials and thinners are as specified, properly labeled and stored, and are within their shelf life.

5.3.2 *Mixing*—Verify that the coating materials are mixed in accordance with the manufacturer's instructions.

5.3.3 *Equipment*—Verify that proper application equipment is being utilized.

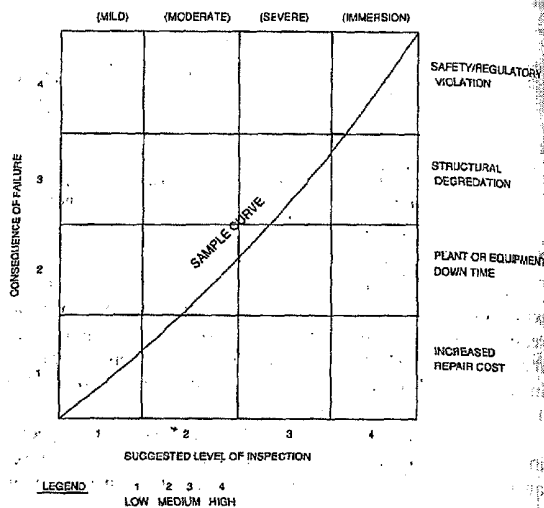
5.3.4 *Air Supply*—Verify that air supply is clean and dry (see Test Method D 4285).

5.3.5 *Ambient Conditions*—Verify that air temperature, humidity, surface temperature, and dew point spread are appropriate (see Test Method E 337).

5.3.6 *Wet Film Thickness*—Verify that the applicator is checking wet film thickness during the application process when required (see Practice D 4414).

5.3.7 *Intercoat Parameters*—Verify that parameters during recoat are observed and that no surface contaminants are present.

SERVICE ENVIRONMENT



NOTE—This figure is provided as a visual aid to represent graphically the general relationship between the consequence of failure and the suggested level of inspection. The slope and configuration of the curve will vary due to the many factors affecting the consequence of failure, however, the suggested level of inspection should increase when the service environment is more severe.

FIG. 1 Relationship Between the Consequence of Failure and the Suggested Level of Inspection

5.3.8 *Appearance*—Visually inspect each coat for defects and uniform appearance (see Guide D 3276).

5.3.9 *Dry Film Thickness*—Verify that the dry film thickness is within the specified range after each coat (see Test Methods D 1186 and D 1400).

5.4 Final Acceptance:

5.4.1 *Appearance*—Visually inspect the surface for defects and uniform appearance (see Guide D 3276).

5.4.2 *Dry Film Thickness*—Verify that the total coating system's dry film thickness is within the specified range (see Test Methods D 1186 and D 1400).

5.4.3 *Holiday Test*—Verify that the surface meets the specified holiday/pinhole criteria (see NACE T6A-37).

5.4.4 *Repairs*—Verify that all identified repairs have been properly made. Repeat inspection requirements and testing as appropriate.

5.4.5 *Final Cure*—Verify that the coating is cured in accordance with the specified requirements of Guide D 3276.

6. Precautions and Limits

6.1 Specialty lining systems such as reinforced laminates may have additional inspection requirements to address hardness, strength, etc that are not addressed by this guide.

6.2 The quality of a project cannot be guaranteed by merely specifying inspection requirements, however, when the recommended inspections are properly implemented and documented, failures are minimized or eliminated.

7. Keywords

7.1 coating, inspection, specifications; consequence of failure; level of inspection; linings

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Discontinuity (Holiday) Testing of Nonconductive Protective Coating on Metallic Substrates¹

This standard is issued under the fixed designation D 5162; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers procedures for determining discontinuities using two types of test equipment:

- 1.1.1 *Test Method A—Low Voltage Wet Sponge, and*
- 1.1.2 *Test Method B—High Voltage Spark Testers.*

1.2 This practice addresses metallic substrates. For concrete surfaces, refer to Practice D 4787.²

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 4787 Practice for Continuity Verification of Liquid or Sheet Linings Applied to Concrete Substrates²

3. Terminology

3.1 *Description of Terms Specific to This Standard:*

3.1.1 *discontinuity, as used in this standard*—a void, crack, thin spot, foreign inclusion, or contamination in the coating film that significantly lowers the dielectric strength of the coating film. May also be identified as a holiday or pinhole.

3.1.2 *holiday, as used in this standard*—a term that identifies a discontinuity.

3.1.3 *holiday detector, as used in this standard*—a device that locates discontinuities in a nonconductive coating film applied to a conductive surface.

3.1.4 *pinhole, as used in this standard*—a film defect characterized by small porelike flaws in the coating which, when extended entirely through the film, will appear as a discontinuity. A pinhole in the finish coat may not appear as a discontinuity.

4. Significance and Use

4.1 A coating is applied to a metallic substrate to prevent corrosion, reduce abrasion or reduce product contamination, or both. The degree of coating continuity required is dictated by service conditions. Discontinuities in a coating are frequently very minute and not readily visible. This practice provides a procedure for electrical detection of minute

discontinuities in nonconductive coating systems.

4.2 Electrical testing to determine the presence and number of discontinuities in a coating film is performed on a nonconductive coating applied to a conductive surface. The allowable number of discontinuities should be determined prior to conducting this test since the acceptable quantity of discontinuities will vary depending on coating film thickness, design, and service conditions.

4.3 The low voltage wet sponge test equipment is generally used for determining the existence of discontinuities in coating films having a total thickness of 20 mils (0.5 mm) or less. High voltage spark test equipment is generally used for determining the existences of discontinuities in coating films having a total thickness of greater than 20 mils (0.5 mm).

4.4 Coatings that are applied at a thickness of less than 20 mils (0.5 mm) may be susceptible to damage if tested with high voltage spark testing equipment. Consult the coating manufacturer for proper test equipment and inspection voltages.

4.5 To prevent damage to a coating film when using high voltage test instrumentation, total film thickness and dielectric strength in a coating system shall be considered in selecting the appropriate voltage for detection of discontinuities. Atmospheric conditions shall also be considered since the voltage required for the spark to gap a given distance in air varies with the conductivity of the air at the time the test is conducted. Suggested starting voltages are provided in Table 1.

4.6 The coating manufacturer shall be consulted to obtain the following information, which would affect the accuracy of this test to determine discontinuities:

4.6.1 Establish the length of time required to adequately dry or cure the applied coating film prior to testing. Solvents retained in an uncured coating film may form an electrically conductive path through the film to the substrate.

4.6.2 Determine whether the coating contains electrically

TABLE 1 Suggested Voltages for High Voltage Spark Testing

Total Dry Film Thickness		Suggested Inspection, V
mils	mm	
8-12	0.20-0.31	1 500
13-18	0.32-0.46	2 000
19-30	0.47-0.77	2 500
31-40	0.78-1.03	4 000
41-60	1.04-1.54	5 000
61-80	1.55-2.04	7 500
81-100	2.05-2.55	10 000
101-125	2.56-3.19	12 000
126-160	3.20-4.07	15 000
161-200	4.08-5.09	20 000
201-250	5.10-6.35	25 000

¹ This practice is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

Current edition approved Sept. 23, 1991. Published November 1991.

² *Annual Book of ASTM Standards*, Vol 06.01.

ductive fillers or pigments that may affect the normal electric properties.

5.1.7 This practice is intended for use with new coatings. It may also be applicable for coatings previously placed in service. However, considerations must be given to potential changes in the electrical properties of the coating due to service exposure.

Test Methods

TEST METHOD A—LOW VOLTAGE WET SPONGE TESTING

1 Apparatus

5.1.1 *Low Voltage Holiday Detector*—an electronic device powered by a self-contained battery with voltages ranging from 5 to 90 V dc, depending on the equipment manufacturer's circuit design. It is used to locate discontinuities in a nonconductive coating applied to a conductive substrate. Operation includes the use of an open-cell sponge electrode wetted with a solution for exploring the coating surface, a ground connection, and an audible or visual indicator, or both, for signaling a point of coating discontinuity.

5.1.2 *Low Voltage Wet Sponge Tester*—a sensitivity device with the operating voltage being of little importance other than being part of the particular electronic circuit design.

5.1.3 *Wet Sponge Type Instrument*—a number of commercially available, industry-accepted, instruments are available. The following electronic principle describes two types of devices generally used; others may be available but are not described in this practice.

5.1.3.1 *Lightweight, Self-Contained, Portable Devices*—based on the electrical principle of an electromagnetic sensitive relay or a solid-state electronic relay circuit that energizes an audible or visual indicator when a coating discontinuity is detected. Generally this equipment is capable of being recalibrated in the field by the user.

5.1.3.2 *Lightweight, Self-Contained, Portable Devices*—also based on the principle of an electronic relaxation oscillator circuit that reacts significantly to the abrupt drop in electrical resistance between the high dielectric value of the coating film and the conductive substrate at the point of coating film discontinuity. This results in a rise in oscillator frequency as well as in the audible signal from the device. Generally, this equipment is incapable of being recalibrated in the field by the user.

2 Procedure

5.2.1 Sufficient drying or curing of the coating shall be allowed prior to conducting a test. The length of time required shall be obtained from the coating manufacturer. Solvents retained in the coating film could produce erroneous indicators.

5.2.2 The surface shall be clean, dry, and free of oil, dirt and other contaminants. Measure the film thickness of the coating with a nondestructive dry film thickness gage. If the coating film exceeds 20 mils (0.5 mm), use the procedures for high voltage spark testing described in Test Method B, High Voltage Spark Testing.

5.2.3 Test the instrument for sensitivity in accordance with 5.3.

5.2.4 Attach the ground wire from the instrument ground output terminal to the metallic substrate and ensure positive electrical contact.

5.2.5 Attach the exploring sponge lead to the other output terminal.

5.2.6 Wet the sponge with a solution consisting of tap water and a low sudsing wetting agent, combined at a ratio of not more than 1/2 fluid oz of wetting agent to 1 gal water. An example of a low sudsing wetting agent is one used in photographic development. The sponge shall be wetted sufficiently to barely avoid dripping of the solution while the sponge is moved over the coating. The wetting agent residue must be removed prior to executing repairs.

5.2.7 Sodium chloride (salt) shall not be added to the wetting solution because of the potential erroneous indications of discontinuities. The salt, after drying on the coated surface, may form a continuous path of conductivity. It will also interfere with intercoat adhesion of additional coats.

5.2.8 Contact a bare spot on the conductive substrate with the wetted sponge to verify that the instrument is properly grounded. This procedure shall be repeated periodically during the test.

5.2.9 Move the sponge over the surface of the coating at a moderate rate approximately 1 ft/s (0.3 m/s), using a double pass over each area. Apply sufficient pressure to maintain a wet surface. If a discontinuity is detected, turn the sponge on end to determine the exact location of the discontinuity.

5.2.10 Discontinuities that require repair shall be identified with a marker that is compatible with the repair coating or one that is easily removed.

5.2.11 To prevent telegraphing (current traveling through a moisture path to a discontinuity, giving an erroneous indication), take care to ensure that the solution is wiped dry from a previously detected discontinuity before continuing the test.

5.2.12 The wetting agent must be completely removed by rinsing the holiday area prior to repair.

5.2.13 Wet sponge holiday detection is not recommended between coats of a multicoat system. However, when a test is conducted between coats of a multicoat system, a wetting agent shall not be used and all residue left by the test water must be completely removed prior to applying additional coats.

5.3. Verifying Operation of Equipment

5.3.1 The instrument shall be tested for sensitivity prior to initial use and periodically thereafter, in accordance with the equipment manufacturer's instructions.

5.3.2 Test the battery for proper voltage output. Refer to the manufacturer's instructions.

5.3.3 Switch the instrument to the "on position," if applicable.

5.3.4 Wet the sponge with a wetting solution consisting of tap water and a wetting agent (see 5.2.6).

5.3.5 Connect the ground cable to the instrument ground output terminal.

5.3.6 Touch the ground cable alligator clip to the wetted sponge. The instrument signal should actuate in accordance with the instrument manufacturer's instructions.

5.3.7 If the instrument should fail to signal, it shall be considered defective.

5.4 Verifying Instrument Calibration

5.4.1 Verify instrument calibration in accordance with the manufacturer's latest published instructions. If out of calibration, the instrument shall be calibrated in accordance with the instrument manufacturer's latest published instructions, or returned for calibration.

TEST METHOD B—HIGH VOLTAGE SPARK TESTING

5.5 Apparatus

5.5.1 *High Voltage Detector (in excess of 800 V)*—An electronic device used to locate discontinuities in a nonconductive protective coating applied to a conductive substrate. It consists of an electrical energy source, an exploring electrode, and a ground wire connection from the indicator signaling current flow through a coating film discontinuity to the substrate. The detector shall be equipped with a visual or audible indicator, or both.

5.5.2 *Exploring Electrode*, shall be of the type capable of maintaining continuous contact with the surface being inspected, such as bolts, raised areas, etc. It shall be kept clean and free of coating material.

5.5.3 *High Voltage Electrical Detector*, can be identified as either a pulse or direct current type. A pulse type detector discharges a cycling, high voltage pulse, while a direct current type discharges continuous voltage.

5.6 Procedure

5.6.1 Sufficient drying or curing of the coating shall be allowed prior to conducting a holiday test. The length of time required shall be obtained from the coating manufacturer. Solvents retained in the coating film could produce erroneous results, as well as a fire hazard.

5.6.2 The surface shall be clean, dry, and free of oil, dirt and other contaminants. Measure thickness of the coating with a nondestructive dry film thickness gage. If the coating film is less than 20 mils (0.5 mm), consider using procedures for low voltage testing (see Test Method A, Low Voltage Wet Sponge Testing). Although the high voltage spark tester is suitable for determining discontinuities in coating films of less than 20 mils (0.5 mm), it is recommended that the coating manufacturer be consulted before using this test. Certain coatings may be damaged if tested with this equipment.

5.6.3 Verify test instrument operation in accordance with 5.7.

5.6.4 Adjust the test instrument to the proper voltage for the coating thickness being tested. In selecting the inspection voltage, it is important to provide sufficient voltage to break the air gap that exists at the holiday. The air gap will vary depending on the total applied film thickness. The voltage required to break a given air gap may also vary due to atmospheric conditions such as relative humidity. Ensure that the voltage is high enough to break the air gap equivalent to the highest coating film thickness by separating the exploring electrode from the bare metal substrate using a nonconductive spacer equal to the maximum coating thickness. The voltage is set high enough to conduct the holiday test only if the spark will jump the gap formed by the spacer. Excessive voltage may produce a holiday in the coating film. The maximum voltage for the applied coating shall be

obtained from the coating manufacturer. Table 1 contains suggested voltages that can be used as guides.

5.6.5 Attach ground wire from the instrument ground output terminal to the metal substrate and ensure positive electrical contact.

5.6.6 Make contact with the exploring electrode on the conductive substrate to verify that the instrument is properly grounded. This test shall be conducted periodically during the test. The spacer test described in 5.6.4 shall also be repeated if significant atmospheric changes take place during testing.

5.6.7 Move the exploring electrode over the surface of the dry coating at a rate of approximately 1 ft/s (0.3 m/s) using a single pass. Moisture on the coating surface may cause erroneous indications. If moisture exists, remove or allow to dry before conducting test.

5.6.8 Discontinuities that require repair shall be identified with a marker that is compatible with the repair coating or one that is easily removed.

5.7 Verifying Operation of Equipment

5.7.1 Test the energy source (battery) for proper voltage output. Refer to the manufacturer's instructions.

5.7.2 Connect the exploring electrode and grounding cable to the terminals of the detectors.

5.7.3 Switch the instrument to the "on" position.

5.7.4 Touch the exploring electrode to the ground cable alligator clip. The instrument signal should actuate in accordance with the instrument manufacturer's operating instructions.

5.7.5 If the instrument fails to signal, it shall be considered defective.

5.8 Verifying Instrument Calibration

5.8.1 Verify instrument calibration in accordance with the manufacturer's latest published instructions. If out of calibration, the instrument shall be calibrated in accordance with the instrument manufacturer's latest published instructions, or returned for calibration.

5.8.2 Perform field checking of the test voltage with the electrode placed against the surface of the lining since the exploring electrode voltage may be reduced by the slight current flow of the lining.

5.8.3 If required, compare measured voltage with the selected test voltage. Depending on the type of tester, adjust the selected voltage $\pm 5\%$.

6. Testing of Repaired Area

6.1 Sufficient drying or curing of the repair coating shall be allowed prior to retesting. The length of time required shall be obtained from the coating manufacturer.

6.2 Conduct the test following the procedures as previously outlined in this practice for the test instrument selected.

6.3 Retest only those areas that have been repaired, unless otherwise specified.

7. Keywords

7.1 discontinuity; holiday; holiday detectors; spark testers

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Standard Guide for Establishing Procedures to Monitor the Performance of Safety Related Coatings in an Operating Nuclear Power Plant¹

This standard is issued under the fixed designation D 5163; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers procedures for establishing a program to monitor Service Level I coating systems in operating nuclear power plants. Monitoring is an on going process of evaluating the condition of the in-service coating systems.

1.2 It is the intent of this guide to provide a recommended basis for establishing a coatings monitoring program, not to mandate a singular basis for all programs. Variations or simplifications of the program described in this guide may be appropriate for each operating nuclear power plant depending on their licensing commitments. Similar guidelines may be applicable for Service Level II and other areas outside containment.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 610 Test Methods for Evaluating Degree of Rusting on Painted Steel Surfaces²
- D 714 Test Method for Evaluating Degree of Blistering of Paints²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 3359 Test Methods for Measuring Adhesion by Tape Test²
- D 4121 Practice for Photographic Documentation of Coating and Lining Failures and Defects²
- D 4537 Guide for Establishing Procedures to Qualify and Certify Inspection Personnel for Coating Work in Nuclear Facilities²
- D 4541 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers²
- F 1130 Practice for Inspecting the Coating System of a Ship's Topside and Superstructure³
- F 1131 Practice for Inspecting the Coating System of a Ship's Tanks and Voids³
- F 1132 Practice for Inspecting the Coating System of a Ship's Decks and Deck Machinery³

¹ This guide is under the jurisdiction of ASTM Committee D-33 on Protective Coating and Lining Work for Power Generation Facilities and is the direct responsibility of Subcommittee D33.04 on Inspection.

Current edition approved Oct. 3, 1991. Published December 1991.

² Annual Book of ASTM Standards, Vol 06.01.

³ Annual Book of ASTM Standards, Vol 01.07.

F 1133 Practice for Inspecting the Coating System of a Ship's Underwater Hull and Boottop During Drydocking³

2.2 ANSI Standard:

N45.2.6—Qualification of Inspection, Examination, and Testing Personnel for Nuclear Power Plants⁴

2.3 SSPC Standard:

SSPC-PA2—Measurement of Dry Paint Thickness With Magnetic Gages⁵

2.4 NACE Standard:

RP0188-88—Standard Practice Discontinuity (Holiday) Testing of Protective Coatings⁶

3. Significance and Use

3.1 Establishment of an in-service coatings monitoring program permits planning and prioritization of coatings maintenance work as needed to maintain coat integrity and performance in nuclear Service Level I coating systems. For additional information on nuclear maintenance coating work, refer to ASTM Manual on Maintenance Coatings for Nuclear Power Plants.⁷

3.2 A coatings monitoring program enables early identification and detection of potential problems in coating systems. Some Service Level I coating systems may be known in advance to be suspect, deficient, or unqualified. Monitoring coating performance will assist in developing follow-up procedures to resolve any significant deficiency relative to coating work.

3.3 Degraded coatings may generate debris under design basis accident conditions that could adversely affect the performance of the post-accident safety systems. A coatings monitoring program may be required to fulfill safety analysis report commitments for Service Level I coating work in a nuclear power plant facility.

4. Responsibility

4.1 The owner/operator shall identify the department or group within the organization to be responsible for establishing the applicable requirements for activities or procedures covered by this guide and shall document the scope of their responsibility. Owner/operator delegation of this responsibility to other outside qualified organizations is per-

⁴ Available from American National Standards Institute, 11 W. 42nd Street, 13th Floor, New York, NY 10036.

⁵ Available from Steel Structures Painting Council, 4400 Fifth Ave., Pittsburgh, PA 15213.

⁶ Available from National Association of Corrosion Engineers, P. O. Box 218340, Houston, TX 77218.

⁷ Available from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103.

mitted and shall be documented.

4.2 It is the responsibility of the owner/operator or his designee performing these activities to specify the detailed methods and procedures for meeting the applicable requirements of this guide.

4.3 The owner/operator or his designee shall assign a coordinator to be responsible for supervising coating inspection activities, data collection and documentation, and for ensuring that inspection personnel are adequately trained and qualified.

4.4 The owner/operator shall assign responsibility for evaluating the results of inspection activities carried out under the coatings performance monitoring program.

Frequency

5.1 Frequency of in-service coating inspection monitoring shall be determined by the owner/operator. In operating nuclear power plants certain monitoring activities may be restricted to major maintenance outages or refueling outages. It is a good practice to perform inspections during each refueling outage or during other major maintenance outages as needed.

Records and Past History

6.1 Coating performance will depend on the operating conditions experienced by the coating systems. Records of these conditions shall be obtained for each operating unit. These may include, but not be limited to, ambient conditions, temperatures, humidity, immersion, splash and spillage, chemical exposures, radiation exposures, previous decontamination procedures, abrasion and physical abuse, and start-up/shutdown frequency. Any change in service criteria or modifications of the physical design must be identified and dated.

6.2 The last two performance monitoring reports pertaining to the coating systems shall be reviewed prior to the monitoring process. Other past coatings history data to be reviewed may include:

6.2.1 Copies of coating specifications, manufacturer's product data sheets, and application procedures for in-place coatings.

6.2.2 Quality control documentation for the existing in-place coating systems and their application.

6.2.3 Copies of previous inspection or monitoring reports.

6.2.4 Documentation pertaining to any maintenance work performed on existing coating systems.

Premonitoring Procedure

7.1 Prior to conducting an inspection of the coating systems, the responsible organization shall ensure that the necessary services and equipment required for inspection are provided. Factors that must be considered while planning the inspection activities include, but are not limited to, lighting, access to coated surfaces, cleaning surfaces of any deposit or build up, ventilation and, where necessary, special underwater inspection requirements.

7.2 Station access procedures for Service Level I coating systems monitoring shall be followed. While access procedures may vary from plant to plant, specific station access procedures may include:

7.2.1 Security clearance for protected, radiation con-

trolled, and vital areas, and escorted or unescorted clearance as required.

7.2.2 Radiological history including prior radiation exposure for all personnel involved.

7.2.3 Health physics classroom training in the use of radiation detection and monitoring devices and procedures for wearing anticontamination clothing.

7.2.4 A radiation work permit based on health physics radiological survey of the work location.

7.2.5 Compliance with radiation work permit requirements and other special radiation controls unique to each work location, and

7.2.6 Issuance of dosimetry.

7.3 The safety requirements of the facility owner/operator must be met when performing all inspection operations.

8. Personnel Requirements, Qualifications, and Training

8.1 The facility owner/operator shall specify the requirements and guidelines for qualification and training of inspection personnel involved in the program. However, inspectors and the coordinator shall be knowledgeable coatings personnel meeting the requirements of ASTM Guide D 4537 or ANSI N45.2.6. The coordinator shall, as a minimum, be a Level II Coatings Inspector.

8.2 The evaluator shall be a person knowledgeable and experienced in nuclear coatings work.

9. Inspection Plan

9.1 The owner/operator shall develop a plant specific inspection plan to accomplish the objectives of the monitoring program. A general visual inspection shall be conducted on all readily accessible coated surfaces during a walk-through. After the walk-through, thorough visual inspections shall be carried out on previously designated areas and on areas noted as deficient during the walk-through. A thorough visual inspection shall also be carried out on all coatings near sumps or screens associated with the Emergency Core Cooling System (ECCS). The inspection plan shall address the following based on specific owner/operator requirements:

9.1.1 A pre-inspection briefing to familiarize all inspection personnel with objectives of the inspections, procedures to be followed, and precautions to be taken.

9.1.2 Monitoring team(s) assignments to specific areas for inspection(s), and

9.1.3 Location maps dividing the plant into identifiable areas for inspection activities to be issued to inspection teams. The maps shall also identify items/areas requiring special testing, if any. The locations of all defects and of all tests performed shall be recorded on the maps so that additional testing, recoating, and further monitoring may be performed.

9.2 During walk-through, visually examine coated surfaces for any visible defects, such as blistering, cracking, flaking/peeling, rusting, and physical damage.

9.2.1 *Blistering*—Compare any blistering found to the blistering pictorial standards of coatings defects (refer to Test Method D 714) and record size and frequency. If the blisters are larger than those on the comparison photographs, measure, record size and extent, and photograph. Report if blistered portions are intact.

9.2.2 *Cracking*—Cracking can be limited to the one layer of coating or extend through to the substrate. Measure the length of the crack or if extensive cracking has occurred, measure the size of the area affected. Determine if the cracking is isolated or is part of a pattern. Record measurements and describe crack depth and pattern on the inspection report. Photograph the area affected.

9.2.3 *Flaking/Peeling/Delamination*—Measure the approximate size of the peels and note the pattern formed. Carefully test to see if lifting can be easily achieved beyond the obvious peeled area. Note all observations on the inspection report and photograph the area affected.

9.2.4 Rusting—Compare with the pictorial standards of Test Method D 610 to determine the degree of rusting. Try to determine the source of rusting (that is, is it surface stain caused by rusting elsewhere, or is it a failure of the coating allowing the substrate to rust). Photograph the affected area and record observations on the inspection report.

9.2.5 If no defects are found, mark "Coating Intact, No Defects" on the inspection report.

9.2.6 If portions of the coating cannot be inspected, note the specific areas on the location map-inspection report, along with the reason why the inspection cannot be conducted.

9.3 Written or photographic documentation, or both of coating inspection areas, failures, and defects shall be made and the process of documentation standardized by the facility owner/operator. Written documentation practice for inspection of coating system as given in Practices F 1130, F 1131, F 1132, and F 1133 may be adopted as a guideline. Practice D 4121 provides one method to obtain consistent, comparable close-up photographs.

9.4 For coating surfaces determined to be suspect, defective or deficient, one or more physical tests, such as dry film thickness (Test Methods D 1186 and SSPC-PA-2), adhesion (Test Methods D 3359 and D 4541), and continuity (NACE-RP0188-88), may be performed when directed by the evaluator. Samples may be gathered, and the size and extent of defective patterns may be described.

9.5 Instruments and equipment needed for inspection may include, but not be limited to, flashlights, spotlights, marker pen, mirror, measuring tape, magnifier, binoculars, 35-mm camera with or without wide angle lens, and polyethylene zip lock sample bags.

10. Reporting and Documentation

10.1 Inspection reports for submittal to responsible evaluation personnel based on specific owner/operator requirements should be prepared by a qualified Level II or Level III Coatings Inspector and should include at least the following information:

10.1.1 A list and location of all areas evidencing deterioration.

10.1.2 Prioritize the repair areas into areas that must be repaired during the same outage and areas where repair can be postponed to future outages, keeping the coating under surveillance in the interim period.

10.1.3 *Inspection Data Sheets*—A typical data sheet is given in Fig. 1, and

10.1.4 *Photographic Documentation*—A typical location map for photographic identification is given in Fig. 2. The

[illegible]

FIG. 1 Inspection Data Sheet

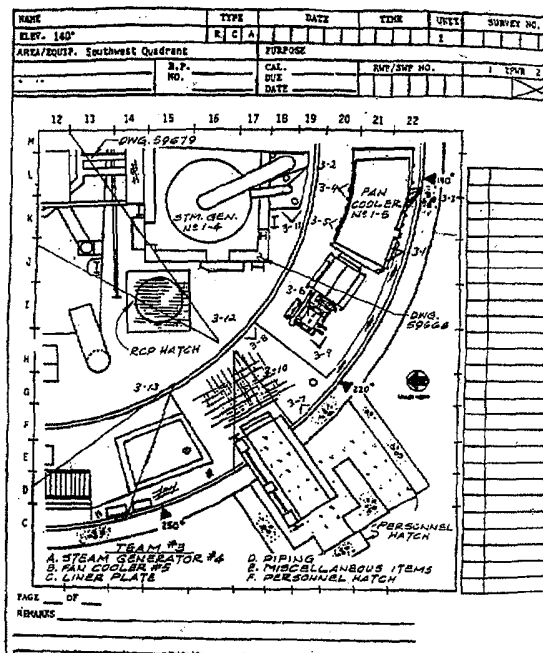


FIG. 2 Photographic Documentation Location Map

tion and direction of all photographs shall be recorded on the maps for comparison with past and future photographs and to record existing conditions. Numbers 3-1 through 3-13 in Fig. 2 depict the location of the photographs and the directions of the view.

Evaluation

11 The inspection report shall be evaluated by the responsible evaluation personnel. The evaluator shall prepare a report that includes a summary of findings and recommendations for future surveillance or repair; this

would include an analysis of the reasons or suspected reasons for failure. The repair work should be prioritized into major and minor defective areas. A recommended corrective plan of action must be provided for the major defective areas so that the plant can repair these areas, if appropriate, during the same outage.

12. Keywords

12.1 coatings monitoring program; coating performance; inspection; maintenance; nuclear power plant; safety related coatings; Service Level I; surveillance

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Mar Resistance of Organic Coatings¹

This standard is issued under the fixed designation D 5178; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the mar resistance on smooth, flat surfaces. Results are expressed in terms of force-to-mar films of organic coatings such as paint, varnish, and lacquer when applied to smooth, flat planar panel surfaces.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 609 Methods for Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products²
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels²
- D 1005 Test Method for Measuring of Dry-Film Thickness of Organic Coatings Using Micrometers²
- D 1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base²
- D 1400 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base²
- D 2691 Test Methods for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products²

3. Terminology

3.1 *mar resistance*—the ability of a coating to resist damage caused by light abrasion. As just defined, it is a resistance of the surface of the coating to permanent deformation, resulting from the application of a dynamic mechanical force.

4. Summary of Test Method

4.1 The materials under test are applied at uniform thickness to flat panels of uniform surface texture. After drying/curing, the mar resistance is determined by pushing the panels beneath a rounded stylus or loop that is loaded in increasing amounts until the coating is marred.

5. Significance and Use

5.1 In some situations, marring of coatings applied to substrates under typical use conditions is unacceptable. This test method has been found useful in differentiating the degree of marring of coatings on substrates. It is most useful in providing relative ratings for a series of coated panels exhibiting significant differences in marring.

6. Apparatus

6.1 *Application Equipment*, as described in Test Methods D 823.

6.2 *Film-Thickness Measuring Apparatus*, as described in Test Methods D 1005, D 1186, D 1400, or D 2691.

6.3 *Balanced Beam Scrape Adhesion and Mar Tester* (Figs. 1 and 2),³ consisting of a balanced beam to which is secured a platform for supporting weights, and a rod at an angle of 45° that holds the scraping loop, a polished, chromium plated steel rod hardened to Rockwell 56 to 58. The loop shall be a $\frac{1}{16}$ -in. (1.6-mm) diameter rod bent into a "U" shape with an outside radius of 0.128 ± 0.002 in. (3.25 ± 0.05 mm). These testers are adjustable to accommodate flat, metallic, and nonmetallic specimens to 0.5 in. (12 mm) thick and 4 to 16 in. (100 to 400 mm) wide and long; the specimen should be at least $\frac{1}{2}$ in. (12 mm) wide.

7. Preparation of Specimens

7.1 Apply the materials under test to panels of the composition and surface condition on which it is desired to determine mar resistance of the coating. The panel material (7.1.1), surface preparation, thickness, and number of coats shall be specified or agreed upon between the seller and the purchaser. Apply coatings and air dry or bake under conditions of humidity and temperature mutually agreeable to the seller and the purchaser.

7.1.1 The surface of the coating must be hard enough to resist damage by the scraping loop at a load agreed upon between the producer and the user. If no panel material is specified, use 0.032-in. (0.8-mm) cold-rolled carbon steel prepared in accordance with Methods B or C of Methods D 609.

8. Conditioning and Number of Tests

8.1 Condition the test panels for at least 48 h at $23 \pm 2^\circ\text{C}$ ($73.5 \pm 3.5^\circ\text{F}$) and $50 \pm 5\%$ relative humidity, and test in the same environment, or immediately on removal therefrom, unless otherwise specified or agreed upon by the seller

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films. Current edition approved Oct. 15, 1991. Published December 1991.

² Annual Book of ASTM Standards, Vol 06.01.

³ The tester is available from BYK-Gardner, Inc., 2435 Linden Lane, Silver Spring, MD 20910 or the Paul N. Gardner Company, Inc., 316 N.E. First St., Pompano Beach, FL 33060.

and the purchaser. Test at least two replicate specimens of each material.

Procedure

9.1 In preparation for using the apparatus, ensure that it is reasonably level and place it so that the weight holder is toward the operator. This places the beam release on the operator's right and allows freedom to move the test specimen manually under the weighted scraping element (loop). Insert the loop into the holder as far as it will go and tighten the clamping screw. Adjust the main bearing support so that the beam is balanced in the horizontal plane when the loop is just touching the specimen surface. The alignment of the beam should be such that the end of the loop is over the midline of the movable table.

9.2 Raise the beam and lock it in the raised position. Wipe the loop with a clean cloth or chamois. Locate the specimen on the sliding platform against the stop so that the specimen can be moved away from the operator and there is an area at least 3 in. (75 mm) long by 1/2 in. (12 mm) wide on the sample parallel to the horizontal plane through the beam. Place weights on the weight support using an initial amount that is estimated to be appropriate for the particular coating. Release the beam and carefully lower it until the loop rests on the coated test specimen and the full load is applied, then slowly push the sliding platform away from the operator at a rate of 1/4 in. (6 mm)/s for a distance of at least 3 in. (75 mm). If the coating is marred, continue the testing using successively smaller loads (0.5-kg increments) until the coating is not marred. If the coating is not marred by the initial scrape, continue the test using successively larger loads



FIG. 2 Balanced Beam Scrape Adhesion and Mar Tester

(0.5-kg increments) until the coating is marred or until the maximum load of 10 kg has been applied. Use a new area of the test surface each time a scrape is made.

9.3 When the critical load has been approximately located, repeat the test five times at each of three loadings: above, below and at the load determined in the first trial. Apply different loads in random fashion so that all scrapes at one load are not made in succession or on one panel.

9.3.1 Periodically examine the loop to ensure that the original smooth surface is intact. If the contacting surface is worn, reverse the loop. When both sides are worn, replace with a new loop.

9.4 For each applied load, tabulate the number of times the coating was marred.

10. Report

10.1 Report the following information:

- 10.1.1 Load at the marring failure point, kg,
- 10.1.2 Panel material and surface preparation,
- 10.1.3 Dry-film thickness, and
- 10.1.4 Any deviation from the specified procedure.

11. Precision

11.1 *Precision*—If sufficient cooperators can be obtained, an interlaboratory study will be conducted to establish precision.

12. Keywords

12.1 balanced beam scrape adhesion and mar tester; balanced beam tester; coatings, mar or organic mar resistance

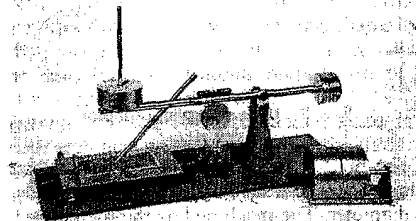


FIG. 1 Balanced Beam Scrape Adhesion and Mar Tester

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Standard Test Method for Measuring Adhesion of Organic Coatings to Plastic Substrates by Direct Tensile Testing¹

This standard is issued under the fixed designation D 5179; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the laboratory determination of organic coating adhesion to plastic substrates by mounting and removing an aluminum stud from the surface of the coating and measuring the force required to break the coating/substrate bond with a tensile tester.

1.2 This test method requires that the aluminum stud be glued directly to the surface of a coated, cured panel (Fig. 1).

1.3 This test method is used to compare the adhesion of coatings to various plastic substrates, thus allowing for a quantitative comparison of various coating/substrate combinations.

1.4 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Summary of Test Method

2.1 An aluminum stud is bonded directly to a coated cured panel. The adhesive is allowed to cure for 2 h at room temperature. The specimen is then subjected to test on a tensile tester equipped with an upper coupling adaptor (Fig. 2), and a restraining device (Fig. 3).

3. Significance and Use

3.1 The pull-off strength (commonly referred to as adhesion) of a coating is an important performance property that has been used in specifications. This test method serves as a means for uniformly preparing and testing organic coatings on plastic substrates.²

4. Apparatus and Materials

4.1 *Tensile Tester*, commercially available.³

4.2 *Aluminum Stud*—The shape and dimensions of the stud are shown in Fig. 1.

4.3 *Upper Coupling Adaptor*—The apparatus is shown in Fig. 2. The adaptor is 3 3/4 in. (94 mm) long, 1 1/4 in. (31 mm) in diameter at the top, and 1 in. (25 mm) in diameter at the

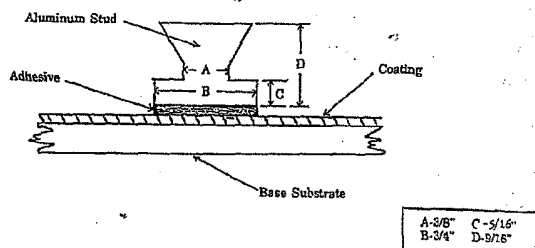


FIG. 1 Direct Tensile Model

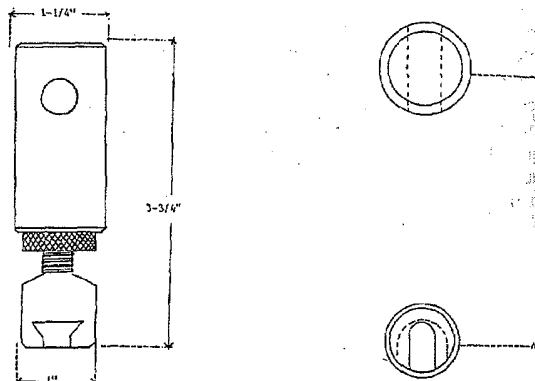


FIG. 2 Upper Coupling Adaptor

bottom. The hole, indicated by "B," is used to attach the adaptor to the tensile tester load cell. The hole has a 1/2 in. (13 mm) diameter. The machined opening indicated by "A," is to receive the head of the aluminum stud.

4.4 *Restraining Device*—The apparatus is shown in Fig. 3 A and 3 B. The 1/2-in. (13-mm) diameter hole, marked "A," in the 2 1/2-in. (63.5-mm) long, 1 1/4-in. (31-mm) diameter lower coupling adapter, is used to mount the device in the tensile tester. The top portion is 4 in. (100 mm) in diameter, 3/4 in. (19 mm) high, and is fitted with a hole slightly larger than 3/4 in. (19 mm) in diameter to allow stud clearance. The bottom screw portion is machined to fit with the top portion and is 1/2 in. (13 mm) thick. Fig. 3 C illustrates the final appearance of the test assembly before it is tested.

4.5 *Wooden Applicators*,⁴ used to clean the adhesive from around the aluminum stud.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

Current edition approved Nov. 18, 1991. Published December 1991.

² Gray, K. N., Buckley, S. E., and Nelson, G. L., "Assessing Measurement Standards for Coating Adhesion to Plastics" *Modern Paint and Coatings Journal*, Vol 75, No. 10, Oct. 1985, p. 160.

³ Tensile tester available from Instron Co., 100 Royall St., Canton, MA 02021, or equivalent, has been found satisfactory for this purpose.

⁴ Applicators, such as Puritan available from 6 in. (150 mm) in length and 0.07 in. (2 mm) in diameter, have been found to be satisfactory for this purpose.

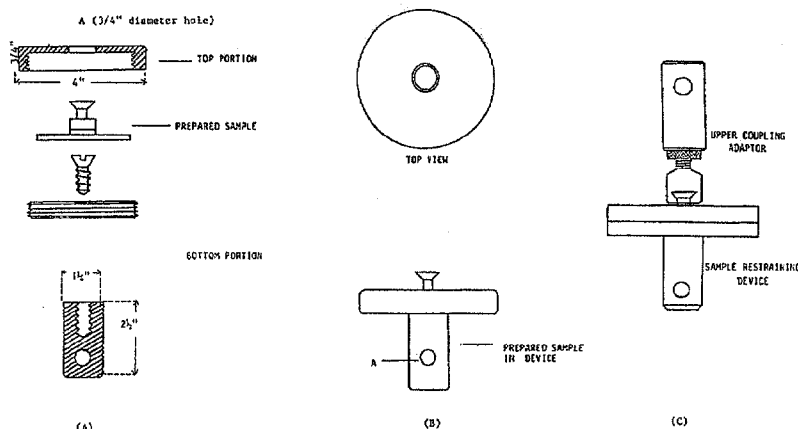


FIG. 3 Direct Tensile Restraining Device

6. Adhesive, cyanoacrylate adhesive.⁵ Since cyanoacrylate adhesives lose adhesive bond strength with time, do not use previously opened containers or lots of adhesive known to be old.

7. Two-Kilogram Weight, approximately 2 in. (50 mm) diameter and 5 in. (130 mm) in height.

Preparation and Conditioning of Specimen

1. Sanding Procedure—Sand large face of stud with 60-grit sandpaper, making certain that the surface is uniformly roughened. When sanding studs, sand straight up and down; rotate stud one quarter of a turn and continue sanding. Make certain surface of stud is flat but rough. While sanding, keep stud face parallel to sandpaper. Non-planar faces cannot be used in testing because they lead to non-uniform bonding of the stud to the coated surface. Only (planar) studs should be used in testing. Planarity may be checked by placing a stud on a flat surface and checking to see if the stud wobbles. Careful preparation of studs is essential for good adhesive adhesion.

2. Cleaning Procedure—Place sanded studs in a large beaker. Pour technical grade acetone over the studs and swirl beaker to completely wash studs. Pour out acetone and repeat the cleaning procedure. Soak the stud for at least 15 min, pour out acetone, and allow studs to dry.

3. Place washed and dried studs and beaker in an oven at 100°C and allow to heat for 1 h. Remove the beaker and studs from the oven and allow to cool. DO NOT TOUCH FACE OF STUDS.

4. Spread four drops of cyanoacrylate adhesive on the large face of an aluminum stud. Next, quickly press the stud into the coated test substrate. Place a 2-kg weight on the stud to ensure good contact between the stud, adhesive, and surface of the coating. Clean the excess adhesive from the edge of the stud with a wooden applicator. Carefully remove the weight after 2 min.

NOTE 1—Substrate panels may be cut to any size that fits the restraining device.

NOTE 2—When the stud is pressed, excess adhesive should escape from under the stud. Excess adhesive buildup at the edge of the stud is a major source of error if allowed to dry. This excess must be removed from around the stud. The adhesive may be removed using a wooden applicator and an absorbent, creped, low-lint material.⁶ Some workers have found cotton swabs⁷ also work well when removing the excessive adhesive.

5.5 Allow the sample to cure for 2 h at room temperature.

NOTE 2—When reusing aluminum studs, the studs must be soaked in acetone to remove coating and resanded. It has been found that soaking the studs in two separate acetone baths thoroughly removes the adhesive and coating. The first acetone bath is used to dissolve the cyanoacrylate adhesive, and the second is used to further clean and dissolve any coating or adhesive left on the stud. Before the studs are used, they should be resanded and cleaned with acetone as specified in 5.2, 5.3, and 5.4.

5.6 Conditioning—Condition the coated substrates for at least 24 h at $23 \pm 2^\circ\text{C}$ ($73.5 \pm 3.5^\circ\text{F}$) and $50 \pm 5\%$ relative humidity, and test in the same environment or immediately on removal therefrom, unless otherwise specified by the purchaser and seller.

6. Procedure

6.1 Install the restraining device and upper adaptor in the tensile tester.

6.2 Calibrate the tensile tester. Make sure that the chart speed is set at 8 to 20 in./min (20 to 50 cm/min). Make sure that the crosshead speed is set at 2 in./min (5 cm/min). Make sure that the chart full scale is set at 100 kg.

6.3 Place the specimen to be tested in the restraining device (Fig. 3 A and 3 B). Pre-position the crosshead and then slowly lower it so the upper coupling adaptor can be attached to the test specimen. Take care to prevent the crosshead from impacting into the top of the specimen. Carefully attach the upper coupling adaptor to the stud (Fig. 3 C).

NOTE 3—When testing thin substrates, a piece of plastic may be placed in the restraining device behind the test specimen to prevent the

⁵ Cyanoacrylate adhesive such as Elmer's Wonder Bond Plus, or equivalent, has been found to be satisfactory for this purpose.

⁶ Kimwipes or equivalents, have been found satisfactory for this purpose.

⁷ Q-Tips or equivalents, have been found satisfactory for this purpose.

TESTING PROGRAM DATA SHEET

NAME: _____ ADDRESS: _____	
COMPANY: _____	
DAY ONE: Date _____	
Material:	Run A B C D E F
Tensile Str:	_____
Type Failure:	_____
% Adhesive Fail:	_____
DAY TWO: Date _____	
Material:	Run A B C D E F
Tensile Str:	_____
Type Failure:	_____
% Adhesive Fail:	_____
Material:	Run A B C D E F
Tensile Str:	_____
Type Failure:	_____
% Adhesive Fail:	_____

FIG. 4 Coating Adhesion Testing by Direct Tensile Methods

substrate from flexing when the stud is pulled from it.

6.4 Turn on the chart recorder and pen. Start the tensile test. Stop the test when the stress returns to zero on the chart.

6.5 Examine test area on each specimen to determine the type of coating failure, rating it according to the following:

- 6.5.1 Adhesive failure of the coating at the substrate, *A*,
- 6.5.2 Cohesive failure in the coating, *C*,
- 6.5.3 Combination of adhesive failure at the coating/substrate interface and cohesive failure in the coating, *AC*,
- 6.5.4 Adhesive failure at the stud, *S*, and
- 6.5.5 Combination of adhesive failure at the stud and cohesive failure in the coating, *CS*.

For multilayer coatings, note if the failure is between the layers. If so, label as *CM*.

6.6 Number and retain all test specimens for adhesion failure calculations. Test five specimens of each coated substrate one day and five on a second day. If one specimen differs significantly from the other four at the same time, fails because of an uneven (non-planar) stud, or for any other reason performs unlike the other four, test a replacement specimen.

NOTE 4—Examine the stud and specimen carefully. Adhesive should have been applied uniformly to the entire stud surface. Coating should have pulled off uniformly over the entire stud surface either with adhesive failure from the substrate (*A*) or cohesive failure in the coating (*C*). If failure is less than 90 % *A* or *C* or (*CM*), if the adhesive has failed at the stud, retest exercising particular care in the specimen and stud preparation.

NOTE 5—The percentage of adhesive failure at the coating/substrate interface is determined by inspecting the tested area on the substrate. This may be assessed by overlaying a transparent sheet grid marked in 0.10-in. (2.54 mm) squares and estimating the percentage of adhesive

failure in each square that lies over the tested area. These percentages may then be averaged to obtain failure for each specimen.

NOTE 6—Sample conditioning, if any, and humidity and temperature of test room may affect results. Notation of these conditions should be made on the test report.

7. Report:

7.1 Report the number of tests, the maximum stress obtained in each test, and the type of failure. Test data sheet is shown in Fig. 4. Fill in data sheet with results obtained from each test. Attach tensile test traces to the data sheets. Calculate and report mean and standard deviation for tensile strength for each coating/substrate combination tested.

8. Precision and Bias⁸

8.1 The precision and bias are primarily dependent upon the accuracy of the force measurements, the alignment of the device, the care exercised in stud and specimen preparation, and the care in testing.

8.2 A round robin involving five different types of coatings and seven different plastic substrates, and ten different laboratories yielded interlaboratory reproducibility data as shown in Table 1. Within laboratory repeatability data is shown in Tables 2 and 3.

9. Keywords

9.1 adhesion; bond strength; cyanoacrylate adhesive; plastic substrate; pull-off strength; pull testing; tensile tester

⁸ A complete report with additional data is available. See Nelson, G. L. "Testing of EMI/RFI Coating Adhesion to Plastics by a Tensile Test Method," Final Report for Computer and Business Manufacturers Association and Society of the Plastics Industry, University of Southern Mississippi, June, 1989.

TABLE 1 Overall Test Reproducibility

Note—This data was generated with lesser cautionary language than found in Note 2 and includes laboratories who had not removed excess adhesive. With experienced operators, reproducibility data can be anticipated to be better than shown in Table 1.

Ten Laboratories		
Sample	Kilogram Force	Standard Deviation
1	34.6	6.7
2	36.2	9.3
3	19.8	4.5
4	26.7	8.7
5	31.7	9.9
6	26.0	7.2
7	41.6	9.2
8	16.6	6.5
9	60.5	20.5
10	28.3	9.8

TABLE 2 Average Tensile Strength Measurements^A

Note—Units for tensile strength and standard deviation are kilograms of force. Multiply by 4.99 to convert to pounds per square inch.

Sample Number		1	2	3	4	5	6	7	8	9	10
Laboratory 1	$\bar{X}$	33.2	32.2	12.9	23.5	25.3	20.8	38.8	5.8	63.9	21.8
	SD ^B	4.3	4.6	2.2	3.5	10.6	3.8	6.3	2.1	12.2	2.5
Laboratory 2	$\bar{X}$	29.2	30.9	19.9	28.0	33.9	22.3	36.8	9.1	80.3	26.0
	SD	5.5	5.8	2.8	3.7	9.2	6.3	7.5	4.0	10.7	5.7
Laboratory 3	$\bar{X}$	33.5	35.7	24.2	32.2	33.8	31.2	49.8	18.6	79.1	39.7
	SD	7.0	4.4	3.9	11.8	5.4	6.4	9.5	4.5	9.6	7.2
Laboratory 4	$\bar{X}$	41.9	45.1	26.2	31.8	43.2	37.2	50.5	17.2	>72.7	33.0
	SD	10.4	4.8	6.1	7.6	6.4	7.7	8.8	5.8	18.7	9.4
Laboratory 5	$\bar{X}$	27.5	23.0	16.7	19.3	24.3	21.1	32.1	26.4	44.6	19.9
	SD	1.1	2.0	3.7	2.2	4.0	2.5	5.3	14.3	11.1	3.1
Laboratory 6	$\bar{X}$	42.4	>41.9	26.2	31.6	48.3	34.1	53.1	23.9	>85.2	33.7
	SD	6.7	5.2	3.0	4.8	6.7	3.5	11.7	6.5	4.8	2.2
Laboratory 7	$\bar{X}$	42.6	42.0	19.0	29.4	31.9	28.9	45.8	16.8	72.4	37.3
	SD	4.1	10.0	2.4	3.7	5.0	4.7	9.9	9.5	26.1	6.3
Laboratory 8	$\bar{X}$	23.2	30.9	18.6	8.6	20.6	21.8	32.8	19.8	32.1	11.2
	SD	5.6	5.1	4.8	1.8	4.7	7.7	9.7	7.5	6.2	2.6
Laboratory 9	$\bar{X}$	34.0	26.4	16.2	25.6	17.4	13.9	27.1	10.4	36.6	20.8
	SD	8.4	8.2	8.8	7.6	2.5	5.3	8.8	5.5	12.4	5.8
Laboratory 10	$\bar{X}$	38.9	53.4	17.6	41.8	38.5	28.5	49.3	18.1	38.4	40.0
	SD	9.9	6.5	2.8	8.7	10.2	9.2	7.4	8.5	14.9	9.0

^A Each is average tensile strength using ten samples.

^B Standard deviation.

TABLE 3 Average Tensile Strengths, Standard Deviations (SD) and Ranks

Note—Units for tensile strength and standard deviation are kilograms of force. Multiply by 4.99 to convert to pounds per square inch.

Sample Number	Coating	Substrate	Failure Mode	Collaborator A			Collaborator B		
				Average	SD	Rank	Average	SD	Rank
1	Lacquer	Xenoy ^A	C	10.7	3.8	12	12.5	2.5	13
2	Lacquer	Lexan ^B	A	5.8	1.0	14	8.8	2.4	15
3	Lacquer	ABS ^C	C	10.2	0.8	13	22.8	3.8	12
4	Lacquer	polycarbonate	A	5.4	2.7	15	9.6	1.9	14
5	Lacquer	ABS	C	12.6	3.4	11	24.8	4.5	11
6	Lacquer	PVC ^D	AC	15.4	3.3	10	25.9	3.1	10
7	Enamel	polycarbonate	A	55.6	29.1	5	67.7	9.5	6
8	Urethane	ABS	A	133.5	23.6	2	122.5	24.9	2
9	Urethane	ABS	A	98.9	12.4	3	91.7	16.9	3
10	Urethane	metal	S	133.7	46.1	1	133.4	51.5	1
11	Enamel	metal	A ^E	61.6	14.8	4	75.4	14.3	4
12	Lacquer	GTX 901	C	27.8	4.9	8	62.9	6.8	7
13	Enamel	GTX 901	C	24.7	6.8	9	66.0	17.9	5
14	Enamel	GTX 901	A	40.9	14.1	6	56.0	14.6	8
15	Enamel	GTX 901	A	34.6	10.4	7	50.6	19.1	9

^A Polycarbonate-based polymer alloy is a trademark of the General Electric Co.

^B Polycarbonate resin is a trademark of the General Electric Co.

^C ABS = acrylonitrile butadiene styrene.

^D PVC = poly(vinyl chloride).

^E Failure rate is 75 %.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Abrasion Resistance of Printed Matter by the GA-CAT Comprehensive Abrasion Tester¹

This standard is issued under the fixed designation D 5181; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method covers the procedure for determining abrasion resistance of printed matter using a GA-CAT Comprehensive Abrasion Tester.

1.2 This test method is applicable to packaging labels, book, catalog, and magazine covers, bar codes, corrugated boxes, and other containers having applied graphics on any substrate.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 ASTM Standards:

E 171 Specification for Standard Atmospheres for Conditioning and Testing Materials²

Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *abrasion resistance*—resistance against the act of scraping, smudging, or rubbing off.

3.1.2 *abrasiveness*—the degree to which a product tends to cause abrasion by the act of rubbing or scraping.

3.1.3 *receptor*—film or paper of standard abrasiveness onto which material removed from the specimen is deposited during the abrasion testing process. Alternatively, printed paper from which material is removed onto the specimen that has a higher degree of abrasiveness than the receptor (in case of testing abrasiveness).

4. Summary of Test Method

4.1 The test print and a receptor are sandwiched in the panel holder of the GA-CAT Comprehensive Abrasion Tester, clamped together with a known force, and made to slide over each other at a known frequency and over a known distance for a predetermined time period.

4.2 The test specimen is examined for degree of print degradation and the receptor for amount of ink or other material transferred from the specimen surface. Results may be rated relative to a comparative control run in the identical manner, or they may be quantified by comparison to a

ranking scale numbered from zero to ten (zero being the most abrasion resistant and ten the least abrasion resistant).

5. Significance and Use

5.1 Abrasion resistance during transport and storage is essential to prevent marring of type matter, designs, or protective coatings on the exterior of labels and other printed materials. Recognizing that the actual amount of abrasion occurring in the field depends on relative humidity, temperature, tightness of packing, and a host of other variables, this test method provides a rapid means for comparing the abrasion resistance of test surfaces under laboratory conditions. It is useful for specification acceptance between the supplier and the customer.

5.2 This test method can also be used to evaluate the relative abrasion resistance of printed inks, varnishes, laminates and substrates, and the abrasiveness of inks.

6. Apparatus

6.1 *GA-CAT Comprehensive Abrasion Tester*.³

6.2 *Ranking Book*,³ containing specimens whose degree of ruboff is ranked from 0 (no ruboff) to +10 (most ruboff).

7. Materials

7.1 *Standard Receptors*, approximately 4 by 4½ in. (102 by 114 mm), of an appropriate grade, as follows:

7.1.1 *C-1³ (least abrasive)*—Glossy coated paper suitable for use with samples of low abrasion resistance.

7.1.2 *A-1, A-3, and A-4^{3,4} (intermediate abrasiveness)*—Imperial lapping film with aluminum oxide abrasive particles of different sizes (9, 12, and 30 μ m, respectively).

7.1.3 *A-6^{3,4} (most abrasive)*, wet or dry, tri-um-it[®] 600.

7.1.4 *B-2³ (intermediate abrasion resistance)*—Printed single color paper suitable for measuring abrasiveness of samples of average abrasiveness (corrugated).

NOTE 1—Other receptors may be substituted provided they have equal abrasiveness on the same test specimens. Further research and experience may indicate a need for additional grades of standard receptors.

7.2 *Foam Sheet*,³ 2 pieces, each approximately 2 mm in thickness and cut to 4 by 4½ in. (102 by 114 mm).

NOTE 2—Use of foam sheeting as a backing for the test specimen and the receptor is recommended to provide uniform pressure over the test surfaces and to prevent ink, varnish, or other particles from becoming imbedded in the sensitive surface of the panel holders.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.56 on Printing Inks.

Current edition approved Oct. 15, 1991. Published December 1991.

² *Annual Book of ASTM Standards*, Vols 08.03 and 15.09.

³ Available from Gavarti Assoc., Ltd., 9240 N. Sleepy Hollow Lane, Milwaukee, WI 53217.

⁴ Available from 3M Co., St. Paul, MN 55119.

7.3 *Comparative Control*, a production or laboratory print preferably having known abrasion resistance. The comparative control must have the same substrate, color(s), and subject matter as the test print.

8. Test Specimen and Conditioning

8.1 The specimen size in the grain direction (or flute in the case of corrugated board) must not exceed 4½ in. (114 mm). The specimen size in the cross-grain direction is less critical; 4 in. (102 mm) is recommended. Care should be taken to avoid contaminating the test surface with fingerprints during specimen cutting and handling.

8.2 Condition the specimens at $73.5 \pm 3.6^\circ\text{F}$ ($23 \pm 2^\circ\text{C}$) and $50 \pm 5\%$ relative humidity in accordance with Specification E 171.

9. Preparation of Apparatus

9.1 Set the abrasion tester on a sturdy bench, preferably in a room conditioned at the temperature and relative humidity prescribed in 8.2. Make sure that the three-prong power cord is correctly connected.

9.2 Periodically, check the liquid level in the hydraulic pressure system. If not full, follow the instructions in the manufacturer's manual.

9.3 If test conditions have not been specified, select an appropriate receptor by running the reference standard for the length of time required to achieve a visible level of degradation. Start with receptor C-1 (7.1.1). If the test time exceeds 2 min, use a receptor with a higher level of abrasiveness.

NOTE 3—Excessively long rubbing times are to be avoided because they introduce uncontrollable heat development that can alter the results.

10. Procedure for Abrasion Testing

10.1 Loading the Instrument:

10.1.1 Move the tray containing the four panel holders to the front black support shelf. Make sure that the inscribed "L" appears on the upper left side of the left panel holder and the "R" in the upper right side of the right panel holder (see Fig. 1). There should be space of at least ½ in. between the two center panel holders.

10.1.2 Face the test specimen on the receptor (selected in 9.3) and sandwich between the foam sheets. Place the sandwich between the two center panel holders so that the

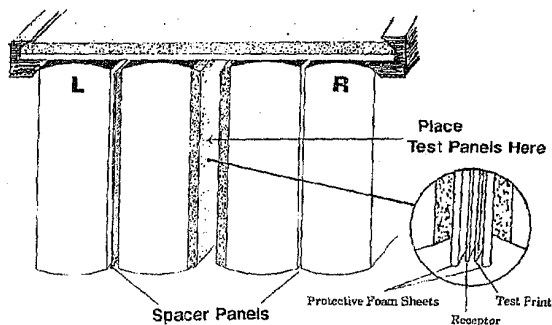


FIG. 1 Panel Holders

grain direction of the specimen is vertical. Take care that the sandwich does not extend below the bottom of the panel holders.

10.1.3 Push the four panel holders together into an upright parallel position. Make sure that the sandwich is not pinched at the bottom of the panel holders; otherwise, it will tend to slide down during the test. Grab the pins and move the tray inside the instrument so that the two retaining pins drop into the carriage holes. If the pins do not drop into their respective holes, recenter the panel holders on the sample tray.

10.1.4 Apply side pressure by turning the lower black spindle until the corresponding dial reads 20 pounds per square inch (psi). Do not apply top pressure at this time.

10.1.5 The carriage inside the instrument is meant to move between two limit switches (used for centering) approximately 2 in. (50.8 mm) apart. If the carriage is not situated between these limits, manually center the carriage.

10.2 Testing Under the Preset Mode:

10.2.1 Turn the power switch on by pressing the upper side of the flip switch at the right side of the instrument.

10.2.2 After the control box window (LCD) displays "GA-CAT ready," press CNTR; the LCD will read "check limits" to make sure the carriage is within the range of the limit switches. Press CNTR again and the LCD will read "release top pressure." Press CNTR once more and the carriage will move to the extreme right (limit switch) and then back to stop exactly in the center. At this point, the LCD will show that the carriage has been centered by showing a flashing square after "GA-CAT READY."

10.2.3 After the instrument is centered, apply the top pressure of 40 psi.

10.2.4 Push the "STRT" button. The LCD will read "computing" and after 3 s, the instrument will run under the following default values:

Time	5.0 s
Frequency	2.0 Hz
Span	1.0 in.
Offset	0.0 in.

10.2.5 When the instrument stops, release the top pressure, making sure that the top pressure plate is high enough to clear the panel holders. Push CNTR to re-center the carriage. Release the side pressure and pull the panel holders out of the carriage. Remove the sandwich and examine the test specimen and the receptor.

10.2.6 To continue running the test with the same settings, reload the instrument as in 10.1, apply the top pressure as in 10.2.3, and push STRT. When the instrument stops, follow the procedure in 10.2.5. Turn the switch off only at the conclusion of testing.

10.3 Changing the Default Values:

10.3.1 The default values may be changed to any of the following settings:

Time	Continuous from 1 to 3600 s
Frequency	0.2 to 3.0 Hz (cycles per s) with a resolution of 0.1 Hz
Span	0.1 to 1.9 in. with a resolution of 0.1 in.
Offset	0.1 to 0.4 in. (increments of 0.1 in.)

10.3.2 In order to change a setting, press the "mode" button until the LCD displays the default value to be altered. Enter the new value on the number buttons and then press "enter." The LCD will read "new value?". If the value is correct, press "enter" a second time. If incorrect, enter

other value or press "clear" and start over again.

NOTE 4—The instrument reverts to the original default values by pressing "reset" and any time the instrument is turned off.

10.3.3 When running thick samples such as corrugated material, follow the procedure in 10.1 through 10.2.1. If the center of the panel holders is not aligned with the center mark of the carriage, use the "offset" mode. Press the "mode" button until "offset" appears and then enter the new setting. A setting of 0.1 or 0.2 is recommended. An additional adjustment for overall width of the panel holders may also be necessary by removing one or two black separators.

10.3.4 Proceed further as in 10.2.2 until 10.2.6.

11. Interpretation of Results

11.1 Run the test material and the comparative control in the identical manner. Examine each print for degree of degradation and each receptor for amount of material transferred from the print. Report results for abrasion relative to the comparative control as equal, slightly or appreciably less, or slightly or appreciably greater.

11.2 A numerical record of degree of abrasion ranging from zero to ten can be obtained from a comparison of the test specimen with examples in the Ranking Book (6.2).

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12. Precision and Bias

12.1 *Precision*—An interlaboratory study of this test method was conducted in which operators in 17 laboratories tested six prints in triplicate on each of two days. The prints consisted of three density levels of two different colors and were ranked for abrasion resistance from 1.0 to 4.0. The within-laboratory pooled standard deviation was 0.54 at 15 df and the between-laboratories pooled standard deviation was 0.58 at 18 df. Based on statistical analysis of the results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

12.1.1 *Repeatability*—Two results, each the mean of two determinations obtained by the same operator on different days, should be considered suspect if they differ by more than 1.5 ranking units.

12.1.2 *Reproducibility*—Two results, each the mean of results obtained on different days by operators in different laboratories, should be considered suspect if they differ by more than 2.2 ranking units.

12.2 *Bias*—Bias cannot be determined as there are no standard materials.

13. Keywords

13.1 abrasion; abrasiveness; printed matter; printing inks; smudging



Standard Test Method for Determination of Volatile Organic Compounds (VOC) of Solvent Reducible Paints in Aerosol Cans¹

This standard is issued under the fixed designation D 5200; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is for the determination of the volatile organic compounds of solvent-based paints in aerosol cans. It offers a unique way to obtain paint specimens from aerosol cans.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific hazard statement is given in Note 1.*

2. Referenced Documents

2.1 ASTM Standards:

- E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens²
- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals³

3. Summary of Test Method

3.1 A designated quantity from an aerosol coating is sprayed into an adapter glass tube assembly and heated in an oven at $110 \pm 5^\circ\text{C}$ for 60 min. The percent volatile is calculated from the loss in weight.

4. Significance and Use

4.1 This test method is the procedure of choice for determining volatiles in coatings for the purpose of calculating the volatile organic content in aerosols under specified test conditions. The inverse value, nonvolatile, is used to determine the weight percent solids content. This information is useful to the paint producer, user, and to environmental interests for determining the grams of volatile organic compounds per gram of solids emitted from aerosol cans.

5. Apparatus

5.1 Adapter Glass Tube Assembly, (Fig. 1).

5.1.1 *Sample Adapter Tube*, straight connecting with 35/25 spherical joints. Loosely fill with glass wool and

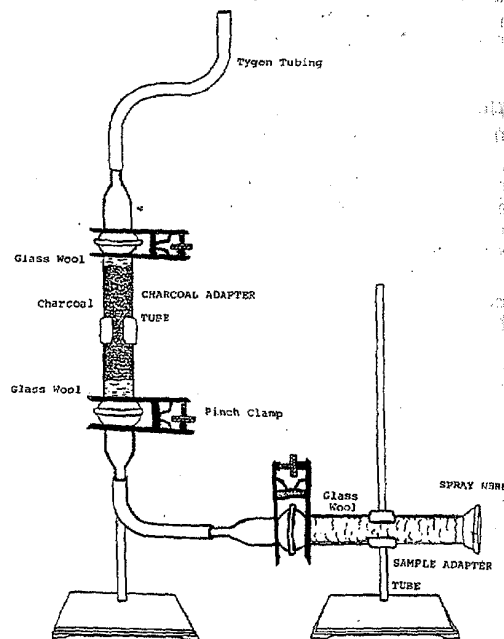


FIG. 1 Adapter Glass Tube Assembly

precondition for 30 min in an oven at $110 \pm 5^\circ\text{C}$ and store in a dessicator prior to use.⁴

5.1.2 *Charcoal Adapter Tube*, straight connecting with 35/25 spherical joints. Fill with activated charcoal and plug both ends with glass wool. This tube is used to prevent the solvent vapors from contaminating the vacuum pump.⁵

5.1.3 *Adapters*, connecting hose with 35/25 socket joints.⁶

5.1.4 *Adapter*,⁷ connecting hose with 35/25 ball joint.

5.1.5 *Clamps*, pinch type, with screw locking device.⁸

¹ This test method is under the jurisdiction of ASTM Committee D-01 on Paints and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Nov. 18, 1991. Published December 1991.

² *Annual Book of ASTM Standards*, Vol 14.02.

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ Sample adapter tube, Catalog No. 5035-35, available from Ace Glass Inc., P.O. Box 688, 1430 Northwest Boulevard, Vineland, NJ 08360, has been found suitable for this purpose.

⁵ Charcoal adapter tube, Catalog No. 5035-3, available from Ace Glass Inc. has been found suitable for this purpose.

⁶ Adapters (socket joints), Catalog No. 5217-35, available from Ace Glass Inc. have been found suitable for this purpose.

⁷ Adapter (ball joint), Catalog No. 5216-35, available from Ace Glass Inc. has been found suitable for this purpose.

⁸ Clamps, Catalog No. 7669-14, available from Ace Glass Inc. have been found suitable for this purpose.

- 6 Glass Wool, medium fine silk.
- 7 Activated Charcoal, coconut, 8 to 12 mesh.
- 8 Tygon Tubing.
- 9 Iron Stands.
- 10 Utility Clamps.
- Vacuum Pump.
- Forced Draft Oven, Type II A or Type II B as specified in Specification E 145.
- Actuators (Valves), with extension tubes.
- Top Loading Balance, capable of weighing to 0.01 g.
- Shaker, similar to the Eberbach shaker in Fig. 2.

Procedure

- 1 Mix the aerosol can thoroughly using a shaker, similar to the Eberbach shaker in Fig. 2, for 15 min at the low speed setting. It is essential that the samples be well mixed to obtain valid results.
- 2 Weigh accurately to 0.01 g, a preconditioned sample adapter tube. Use a pair of gloves at all times when handling the adapter glass tube.
- 3 Remove the cap and actuator from the mixed can. Replace the actuator with one having an extension tube.
- 4 Test actuator and extension tube fit by spraying some contents out for about 5 s. This step also clears the dip tube to ensure a separation has occurred. If a leak is observed, replace with a better fitting actuator or extension tube.
- 5 Weigh the aerosol can with the actuator to the nearest 0.01 g. Spray 3 to 5 g of aerosol into the adapter tube assembly, spreading out the coating by moving the extension tube around the wall of the adapter tube. The spraying is done with the vacuum on.
- 6 Obtain the specimen weight by difference by weighing the aerosol can again to 0.01 g after spraying out the specimen.

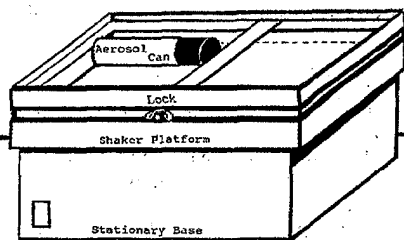


FIG. 2 Aerosol Can on Eberbach Shaker

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6.7 Place the sample adapter tube in the drying oven for 60 min at $110 \pm 5^\circ\text{C}$.

NOTE 1: Warning—Provide adequate ventilation, consistent with accepted laboratory practice, to prevent solvent vapors from accumulating to a dangerous level.

6.8 Remove the adapter tubes from the oven, place immediately in a desiccator, cool to ambient temperature and weigh to 0.01 g.

7. Calculations

7.1 Calculate the percent nonvolatile NV, in the aerosol can as follows:

$$\text{NV, \%} = (W_2/W_1) \times 100 \quad (1)$$

where:

W_1 = weight of aerosol can before spraying sample minus weight of aerosol can after spraying sample, g, and
 W_2 = weight of sample adapter tube with solids minus weight of sample adapter tube, g.

7.2 The percent volatile organic compounds, VOC in the aerosol can may be calculated by the difference as follows:

$$\text{VOC, \%} = 100 - \text{NV} \quad (2)$$

where grams of VOC/grams of solid equal VOC/NV.

8. Precision and Bias

8.1 *Precision*—Estimates are based on an interlaboratory study in which 1 operator in each of 3 laboratories analyzed in duplicate on two different days 3 samples of solvent-based aerosol coatings containing 63.04 % to 77.53 % VOC. The coatings were commercially supplied. The results were analyzed statistically in accordance with Practice E 180. The within laboratory coefficient of variation was found to be 0.51 % relative at 9 df and the between laboratory coefficient of variation was 1.04 % relative at 6 df. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

8.1.1 *Repeatability*—Two results, each the mean of duplicate determinations obtained by the same operator on different days, should be considered suspect if they differ by more than 1.62 % relative.

8.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations obtained by operators in different laboratories should be considered suspect if they differ by more than 3.59 %.

8.2 *Bias*—Bias has not been determined.



Standard Practice for Calculating Formulation Physical Constants of Paints and Coatings¹

This standard is issued under the fixed designation D 5201; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice describes procedures for calculating weight solids, volume solids, solvent content, and density of liquid paints and coatings based on formulation data (not from analytical determinations). These calculations may be related to either as-supplied (unreduced) or as-applied (reduced) coating materials, including multicomponent types.

1.2 These values may not be acceptable for regulatory purposes.

1.3 Calculated values for these physical constants are used in the coatings industry for developing lower volatile organic compound (VOC) paints, making comparisons, etc.

1.4 The calculations described in this practice are based on the following assumptions: (1) Solids (nonvolatile) content and density of the individual ingredients (raw materials or intermediates, or both) have been established by appropriate analytical methods (see Test Methods D 153, D 1475, D 2369, D 4558, and Guide D 2832), and (2) The blended formulation behaves as an ideal solution with no volume change on mixing (see 6.2).

1.5 In calculations involving the liquids in a formulation, those volatile liquids that are incorporated primarily for vehicle solvency and control of the application characteristics should be classified as *solvents*. This would include solvents normally used to adjust viscosity for application and appearance of the coating. Liquids that are expected to be retained in the dried film to affect the final performance properties should be classified as part of the coating solids.

NOTE 1—For regulatory VOC purposes, use EPA Reference Method 24 and ASTM standards listed, or state regulatory requirements (see EPA 450/3-88-018).

1.6 Volatile by-products of cross-linking reactions (cure volatiles) are not considered in these calculations since the object of this practice is to define paint physical constants based on formulation information.

2. Referenced Documents

2.1 ASTM Standards:

- D 153 Test Methods for Specific Gravity of Pigments²
- D 1475 Test Method For Density of Paint, Varnish, Lacquer, and Related Products³
- D 2369 Test Methods for Volatile Content of Coatings³

D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings³

D 3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings³

D 4558 Test Method for Nonvolatile Content of Latexes³

2.2 U. S. Environmental Protection Agency Standards: EPA 450/3-88-018, U.S. Environmental Protection Agency Protocol for Determining the Daily Volatile Organic Compound Emission Rate of Automobile and Light Duty Truck Topcoat Operations⁴

EPA Federal Reference Method 24—Determination of Volatile Matter Content, Water Content, Density Volume Solids, and Weight Solids, of Surface Coatings⁵

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *formula density*—(see Test Method D 1475), the mass (weight in vacuum) of a unit volume of material at any given temperature.

3.1.1.1 DISCUSSION—In this practice, density is expressed in pounds per U.S. gallon (lb/gal) since this is commonly used in the coatings industry. Where dry materials are concerned, actual density (not bulk density) should be determined analytically or obtained from supplier information. Use Test Methods D 153 where applicable.

3.1.2 *formula percent volume solids content*—the calculated volume of nonvolatile material in a formula divided by the total volume of the paint material, times 100 %.

3.1.3 *formula percent weight solids content*—the calculated weight of nonvolatile material in a formula divided by the total weight of the coating material, times 100 %.

3.1.4 *formula solvent content*—the calculated weight of the solvents in a specific volume of paint (such as pounds of solvent per gallon of paint) based on formulation, which is determined by totalling all solvents present.

3.1.4.1 DISCUSSION—Volatile by-products of cross-linking reactions (cure volatiles) are not included in the formula solvent content. See Note 1 and applicable government regulations for definition of the term VOC.

3.1.5 *formula solvent density*—the calculated density of the combined solvent composition.

3.1.6 *formula VOC content*—calculated amount based

¹ This practice is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved Nov. 15, 1991. Published January 1992.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Refer to EPA 450/3-88-018 dated December 1988. This protocol makes reference to the paint formulation physical constants for VOC and volume solids content.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

on total formula solvent content, (such as pounds of solvent per gallon of paint) exclusive of water or solvents that are exempted by regulations. This is a theoretical value.

1.6.1 DISCUSSION—Solvent and VOC are not equivalent terms.

Significance and Use

5.1 Physical constants of paints and coatings are required for all aspects of their formulation, manufacture and use. This practice demonstrates standard methods agreed upon for calculating formulation values for some of these physical constants. The calculations are the same for either metric or U.S. customary units.

5.2 These formula values may not be used to replace measured values required by government regulations unless specifically stated in the governing documents.

Calculations

5.1 Formula Density (weight per unit volume):

5.1.1 The formula density (D_f) can be calculated from the total weight (W_f) and total volume (V_f) of the formulation. The formula volume can be calculated from the weight and density of each ingredient as given by the following equation:

$$D_f = \frac{W_f}{V_f} = \frac{W_1 + W_2 + \dots + W_n}{W_1/D_1 + W_2/D_2 + \dots + W_n/D_n} = \sum \frac{W_i}{W_i/D_i} \quad (1)$$

where:

- n = number of items in the formulation,
- D_f = formula density, lb/gal (g/L),
- W_f = total weight of formula, lb (g),
- V_f = total volume of formula, gal (L),
- W_i = weight of ingredient, lb (g), and
- D_i = density of ingredient.

5.1.1.1 An example would be as follows:

Ingredient	Weight W_i (lb)	Density D_i (lb/gal)	Volume V_i (gal)
	81.50	7.74	10.530
	6.10	7.90	0.772
	0.40	8.72	0.046
	<u>12.00</u>	<u>7.65</u>	<u>1.569</u>
Formula	100.00	D_f	12.917

Therefore, formula density, $D_f = \frac{100}{12.917} = 7.74$ lb/gal.

5.1.2 The density of any one of the ingredients in a product can be calculated as long as the density of the paint formulation and the other ingredients in that formulation are known ($D = W/V$), as in the following example:

Ingredient	Weight W_i (lb)	Density D_i (lb/gal)	Volume V_i (gal)
Polymer solids	50.00	D_{ps}	V_{ps}
Solvent A	25.00	6.95	3.60
Solvent B	<u>25.00</u>	<u>7.18</u>	<u>3.48</u>
Formula	100.00	7.50	V_f

where:

$$V_f = \text{volume of total formula, gal} = \frac{100.00}{7.50} = 13.3 \text{ gal,}$$

$$V_{ps} = \text{volume of polymer solids, gal} = 13.33 - (3.60 + 3.48) = 6.25 \text{ gal, and}$$

$$D_{ps} = \text{density of polymer solids, lb/gal} = \frac{50.00}{6.25} = 8.00 \text{ lb/gal.}$$

5.2 Formula Solvent (Volatile) Density:

5.2.1 The density of the solvent (volatile) portion can be calculated using the following equation:

$$D_s = \frac{V_1 D_1 + V_2 D_2 + V_3 D_3 + \dots + V_n D_n}{V_1 + V_2 + V_3 + \dots + V_n} = \sum \frac{V_i D_i}{V_i} \quad (2)$$

where:

- D_s = density of solid formula, lb/gal (g/L),
- V_i = volume of solvent, gal (L),
- D_i = density of solvent, lb/gal (g/L), and
- n = number of items in the formulation.

5.3 Formula Weight Percent Solids (Nonvolatile):

5.3.1 Calculate percent of solids by weight as follows:

$$S_w = \frac{\text{weight of solids}}{\text{total weight of coating}} \times 100 \quad (3)$$

where S_w = weight percent of solids (nonvolatile), %.

5.4 Formula Volume Percent Solids (Nonvolatile):

5.4.1 Calculate percent of solids by volume using either of the following equations depending on available information:

$$S_v = \frac{\text{volume of solids}}{\text{total volume of paint}} \times 100 \quad (4)$$

where S_v = volume percent of solids (nonvolatile), %.

or,

5.4.1.1 Generally the volume solids content is calculated by subtracting the volume of all solvent from the total volume since the volume of the solvent portion is usually more readily available than the volume of solid materials

$$S_v = \frac{\text{total volume of paint} - \text{volume of solvents}}{\text{total volume of paint}} \times 100 \quad (5)$$

where S_v = total volume percent of paint minus volume of solids.

5.4.2 When the volume solids of each ingredient in a formulation is known, the volume solids of the formulation can be calculated.

5.4.2.1 The volume (V_i) of each ingredient is calculated from the formula weight (W_i) of each ingredient divided by its density (D_i).

$$V_i = W_i/D_i \quad (6)$$

5.4.2.2 Total volume of the formula is determined by the sum of the volumes of the individual ingredients

$$V_f = \sum V_i \quad (7)$$

5.4.2.3 Formula volume solids (S_{vf}) is calculated in the following manner. The volume of each ingredient (V_i) is multiplied by the volume percent solids of that ingredient (S_{vi}) and the sum of these volume solids is divided by the total volume of the formula to give formula volume solids. This is shown symbolically as follows:

$$S_{vf} = \frac{\sum (S_{vi} \times V_i)}{V_f} \quad (8)$$

5.4.2.4 An example would be as follows:

Ingredient	Weight W, (lb)	Density D, (lb/gal)	Total Volume	Volume Solids, %	Volume of Solids
1	7.35	8.00	0.92	31.0	0.29
2	22.41	7.96	2.82	21.5	0.61
3	52.85	8.24	6.41	23.8	1.53
4	5.98	7.16	0.84	0.0	...
5	6.13	9.27	0.66	100.00	0.66
6	0.28	7.17	0.04	0.0	...
7	5.00	8.14	0.61	33.6	0.20
	100.00		12.30		3.29

$$V_s \% = \frac{\text{volume of solids, gal}}{\text{total volume, gal}} \times 100 \quad (9)$$

$$= \frac{3.29}{12.30} \times 100 = 26.7 \%$$

5.5 Formula Total Solvent Content and VOC Content (see 3.6):

5.5.1 If all of the solvent in the formulation is considered to be VOC for regulatory purposes, the VOC content may be expressed as pounds of VOC per gallon (grams per litre) of paint material, calculated as follows:

$$\text{VOC (lb/gal)} = \frac{(100 - \text{weight \% solids}) \times \text{density of coating}}{100} \quad (10)$$

5.5.2 If water is present in the paint material formulation, it must be subtracted from the total volatile portion in determining the formulation VOC content.

$$\text{VOC (lbs/gal) (less water)} = \frac{\text{weight of volatiles} - \text{weight of water}}{\text{volume of paint} - \text{volume of water}}$$

or,

$$= \frac{(100 - \text{weight percent of solids} - \text{weight percent of water}) \text{ formula density}}{100 - \frac{(\text{weight percent of water}) \text{ formula density}}{\text{water density}}} \quad (11)$$

where density equals pounds/gallon (grams per litre).

NOTE 2—Deduction for other non-VOC volatile ingredients as defined in local ordinances may be treated in a manner similar to water as just illustrated and in Practice D 3960.

NOTE 3—Solvent content and VOC content values may be converted from pounds per (U.S.) gallon (lb/gal) to grams per litre (g/L) by multiplying by 119.84.

5.6 Paints Reduced for Application:

5.6.1 The calculations and examples shown in 5.1 through 5.5 are for as-supplied materials intended for use without further reduction. The same calculations can be used for determining the formulation density, percent weight solids content, percent volume solids content, and solvent (VOC) content of materials that have been reduced for application. It is only necessary to know the amount of reduction (volume or weight) and the density of the reducing solvent. The reducing solvent then becomes an additional ingredient in the paint formulation.

5.6.2 Each of the following examples is based on the equation:

$$D = \frac{W}{V}$$

where:

D = density,

W = weight, and

V = volume.

5.6.2.1 Density of Reduced Material Knowing Percent Reduction by Volume:

(1) The paint material described in the example in 5.1.1.1 is reduced 20 % by volume with reducing thinner having a density of 7.20 lb/gal as follows:

	Volume, gal	Density, lb/gal	Weight, lb
Unreduced material	1.00	7.74	7.74
Reducing thinner	0.20	7.20	1.44

where (reducing thinner), weight = volume × density, or

$$W = 0.20 \times 7.20 = 1.44 \text{ lb.}$$

(2) The density of the reduced material is obtained by adding the volume and weight of each component, and dividing the total weight by the total volume:

	Volume, gal	Weight, lb
Unreduced material	1.00	7.74
Reducing thinner	0.20	1.44
	1.20	9.18

where density of reduced paint = $\frac{9.18}{1.20} = 7.65 \text{ lb/gal.}$

5.6.2.2 Formula Volume Solids of Reduced Paint Knowing Percent Reduction and Unreduced Volume Solids:

Using the information from the previous example and assuming an unreduced volume solids of 50 % and a volume reduction of 20 % thinner, the reduced formula volume solids content (S_{vr}) is calculated as follows:

	Volume, gal	Solids, gal
Unreduced material	1.00	0.50
Reducing thinner	0.20	0.00
	1.20	0.50

where formula volume solids, reduced = $\frac{0.50}{1.20} = 0.42 \text{ gal, or}$
 $S_{vr} = 42.0 \%$

6. Precision and Bias

6.1 No statement is made about either the precision or bias of this practice for calculating formulation physical constants since the results are obtained strictly by mathematical calculations and will be related to the accuracy of the data used and conformance to the prescribed calculations.

6.2 A bias toward slightly smaller volumes and higher densities may result from non-ideal solution behavior (see 1.3, (2)).

7. Keywords

7.1 density; formulation; physical constants; solids content; volatile organic compound, (VOC)

based on the

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Standard Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry¹

This standard is issued under the fixed designation E 97; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This method covers the determination of the 45-deg, 0-deg directional reflectance factor of nonfluorescent opaque specimens by means of filter photometers. To obtain similar results from spectrophotometers, see Method E 308.

NOTE 1—The values stated in inch-pound units are to be regarded as the standard.

NOTE 2—This method has been developed for determining (1) the daylight luminous reflectance factor of paint, opaque white porcelain enamels, and ceramic whitewares; and (2) the blue-light reflectance factor (sometimes called "brightness") of uncolored papers and pulps in sheet form. The method may also be used for determining the reflectance factor of other opaque specimens.

NOTE 3—The blue-light reflectance factor of paper provides a measure of freedom from yellowness such as results in pulp and paper from the presence of lignin and other so-called impurities left by incomplete bleaching.

NOTE 4—Blue-light reflectance factor by this method differs slightly, both spectrally and geometrically from the TAPPI Standards T 217 and T 452. The TAPPI methods are considered to be standard for the pulp and paper industry when brightness measurements are to be made.

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- C 347 Test Method for Reflectance, Reflectivity, and Coefficient of Scatter of White Porcelain Enamels²
- D 523 Test Method for Specular Gloss³
- D 585 Method for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Product⁴
- D 823 Test Methods for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels³

¹ This method is under the jurisdiction of ASTM Committee E-12 on Appearance of Materials and is the direct responsibility of Subcommittee E12.03 on Geometric Practices.

Current edition approved Oct. 29, 1982. Published December 1982. Originally published as E 97 - 53 T. Last previous edition E 97 - 77.

² Annual Book of ASTM Standards, Vol 02.05.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vol 15.09.

E 179 Practice for Selection of Geometric Conditions for Measurement of Reflectance and Transmission Properties of Materials⁵

E 284 Terminology Relating to Appearance of Materials⁵

E 308 Method for Computing the Colors of Objects By Using the CIE System³

2.2 TAPPI Standards:⁶

T 217 Brightness of Pulp

T 452 Brightness of Paper and Paper Board

3. Significance and Use

3.1 Measurements of reflectance factor provide a measure of the amount of incident radiation reflecting diffusely under the geometric conditions of this method. High values indicate low absorption. When the response of the detector conforms to the sensitivity of the average human eye (Y-tristimulus filter), the result is designated luminous reflectance. For near white specimens, the reflectance value for blue light (Z-tristimulus filter), provides a relative index of freedom from yellowness for a particular type of material.

3.2 This method is frequently used to evaluate near-white, diffusely reflecting materials such as paint, paper, and powders.

4. Terminology

4.1 *reflectance factor*—ratio of flux reflected from a specimen to the luminous flux reflected from a standard surface, under the same geometric conditions of measurement. Unless otherwise indicated, the standard surface is assumed to be a perfect reflecting diffuser. If some real surface is used, the quantity measured is called "relative reflectance factor."

4.2 *daylight 45-deg, 0-deg luminous directional reflectance factor*—the ratio of the luminous flux from a specimen illuminated at an angle of 45 deg and viewed perpendicularly by the equivalent of CIE standard source C and the 1931 CIE standard observer, to the luminous flux from the perfect reflecting diffuser, similarly illuminated and viewed (see Definitions E 284).

NOTE 5—The combination of illumination at 45-deg and viewing at 0-deg (perpendicularly), designated 45/0, or the converse designated 0/45, has been selected as being representative of average conditions of illuminating and viewing. This reflectance factor determines which of

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Available from Technical Association of the Pulp and Paper Industry, P.O. Box 105113 Atlanta, GA 30348.

specimens will appear lighter when viewed in daylight at an angle at which the observation of highlights is avoided.

5.3 blue-light 45-deg, 0-deg directional reflectance factor—the ratio of the light flux from the specimen illuminated at an angle of 45 deg by CIE standard source C, and viewed perpendicularly by a receptor whose response is equivalent to the $\bar{z}$ -function of the CIE standard observer, to the light flux from the perfect reflecting diffuser similarly illuminated and viewed (see Definitions E 284).

NOTE 6—CIE standard sources and functions are defined in Method D 585.

Apparatus

5.1 The apparatus shall consist of a reflectometer, either visual or photoelectric type, having source, filter, receptor, and design characteristics such that it will measure reflectance factors accurately to within 1.0 % of full-scale reading. The apparatus shall have the following characteristics:

5.1.1 Spectral Characteristics:

5.1.1.1 For Measurement of Daylight Luminous Reflectance Factor—The spectral energy distribution of the illuminator and the spectral sensitivity of the receptor, in combination, shall provide the equivalent of illumination by CIE standard source C and observation by the luminosity (y) function of the 1931 CIE standard observer.

NOTE 7—Spectral characteristics based on CIE standard illuminant D₆₅, or any other CIE daylight standard illuminant, or on the $\bar{y}$ function of the CIE 1964 supplementary standard observer (which is not the luminosity function) may be used as an alternative.

5.1.1.2 For Measurement of Blue-Light Reflectance Factor—The product of spectral energy of source, spectral transmission of filters, and spectral response of receptor shall be equivalent to the product of the $\bar{z}$ -function of the CIE standard observer multiplied by the energy distribution of CIE standard source C.

5.1.2 Geometric Characteristics—Illumination shall be within 4 deg of, and centered about, a single direction of 45 deg from the perpendicular to the test surface. It shall be effectively nonpolarized. Viewing shall be within 15 deg of, and centered about, the perpendicular.

NOTE 8—Any instrument that meets these apparatus specifications may be used. Instruments should also meet the precision requirements prescribed in Section 11. In general, commercial instruments do not conform exactly to the apparatus requirements. The suitability of a given instrument depends in large measure on the spectral selectivity of the specimens being measured, and on the availability of standards of similar reflectance and spectral character.⁷

6. Standards

6.1 Primary Standard—The primary standard for reflectance is the perfect reflecting diffuser as described in Definitions E 284. It is assigned a value of 100 for the conditions of 45-deg illumination and perpendicular view.

⁷ The names and addresses of manufacturers producing instruments for the measurement of reflectance are listed in *Optical Industry and Systems Directory*, available from Optical Publishing Co., Inc., P.O. Box 1146, Pittsfield, Mass. 01201.

6.2 Secondary Standards⁸—Porcelain-enameled metal plaques, or other materials known to be reasonably permanent in reflectance and uniform over the surface, may be calibrated and used as secondary reflectance standards.

7. Preparation of Test Specimens

7.1 Paint—Unless otherwise specified, prepare panels for the determination of reflectance factor by applying the paint to a suitable flat background with a doctor blade in accordance with Test Methods D 823. Use sufficient thickness so that additional coats produce no measurable change in reflectance factor. Allow 72 h for the paint to dry. It is recommended that 60-deg gloss as described in Test Method D 523 and unusual surface structure be noted.

7.2 Porcelain Enamel—Laboratory-prepared specimens, articles of commerce, or sections cut from articles of commerce may be tested. The area to be tested shall be flat and reasonably free of surface defects. Laboratory specimens shall be at least 60 by 90 mm (2½ by 3½ in.) in size; however, specimens 100 by 100 mm (4 by 4 in.) are preferred.

NOTE 9—The method of preparation of the specimen, such as weight of application or firing treatment, may affect the reflectance factor but is not a part of this test method; however, each manufacturer should investigate the effect of such variables on the reflectance factor of his products (see Test Method C 347).

7.3 Paper—Sample in accordance with Test Methods D 585. Handle the test specimen carefully to avoid soiling, and take care not to touch the areas to be tested. The test specimen shall consist of a pad of sheets sufficiently thick that doubling the number of sheets causes no measurable change in reflectance factor.

7.4 Other Materials—Prepare test specimens of materials other than those listed in 7.2 and 7.3 in accordance with accepted practice, and record the method of preparation in the report.

NOTE 10—All high-reflectance diffusely reflecting materials are somewhat translucent since most of their reflectance is due to internal scattering. Roughly 95 % of the incident light is transmitted through the surface, and partially absorbed. What is not absorbed is multiply scattered back to the surface, at a point different from the point of incidence, where it is retransmitted as reflected light. The maximum distance between the points of incidence and exitance is primarily a function of the extinction coefficient of the material. For materials such as most white paints and porcelain enamels, the extinction coefficient is large, and the maximum distance of travel is small, and errors due to this internal travel may be small enough to be neglected. Other high-reflectance materials, such as opal glass and white plastics, may have smaller extinction coefficients, and the maximum distance of internal travel may be large enough (several millimetres is not uncommon) that significant light losses can occur unless the reflectometer has been designed to eliminate such losses. The maximum errors occur in measurements of reflection factor when comparing sample and standard of markedly different extinction coefficients. See NBS Technical Note 594-12 (Oct. 1976) for a detailed discussion of such losses.

⁸ Secondary standards of porcelain enamel may be obtained from the Hunter Associates Laboratory, Inc., 11495 Sunset Hills Rd., Reston, Va. 22090, or the Gardner Laboratory Division of Pacific Scientific, P.O. Box 5728, Bethesda, Md. 20014. Other sources of standards may be obtained by writing to ASTM Headquarters.

8. Procedure

8.1 Operate the reflectometer in accordance with the instructions supplied by the manufacturer, including line voltage, warm-up time, and adjustment of the scale.

8.2 Use a standard having a reflectance factor close to that of the test specimen or, if several specimens having a small range of reflectance factor values are being tested, use at least two standards, preferably at the extremes of the range being measured. Use a green filter for luminous reflectance factor; a blue filter for blue-light reflectance factor of paper or other materials; and, when specified, other filters for reflectance factor for other kinds of light, such as an amber filter for amber-light reflectance factor, etc.

NOTE 11—In general, instrumental errors are related to the differences in reflectance factors and spectral characteristics between specimen and standard. For greatest accuracy, standards close in reflectance factor and similar in spectral characteristics to the specimens should be used. The same area of standard should be measured as that used for its calibration.

8.3 Obtain the instrument readings for the standards and then for the specimens, in turn; read the specimens in reverse order, and finally read the standards again.

8.3.1 *Paper Specimens*—Make readings with the blue filter on at least five separate sheets, and equal numbers of readings parallel to and at right angles to the machine direction of the paper on both sides of the sheets.

8.3.2 *Porcelain-Enameled Specimens*—Make reflectance factor readings with the green filter on an area not less than that of a circle 70 mm (2 7/8 in.) in diameter. A determination shall comprise a sufficient number of readings so that the average of successive determinations can be reproduced with a difference between the highest and lowest determinations, not exceeding 0.5 % on the scale for which the perfect reflecting diffuser is assigned a value of 100 (see 6.1). Instruments covering areas smaller than that prescribed may be used, provided a sufficient number of well-distributed readings is taken so that the average reflectance factor is determined within the spread prescribed for repeated determinations on a given specimen.

9. Calculation

9.1 Calculate the mean value for the readings on each standard and specimen.

9.2 Calculate the reflectance factor of each specimen according to the directions of the instrument manufacturer. Apply corrections for scale nonlinearity, if available. In the

absence of more explicit instructions, calculate the specimen reflectance factor as follows:

$$R = c \times (R_s/b)$$

where:

R = reflectance factor of the specimen for the filter used,
 c = mean instrument reading for the specimen,
 b = mean instrument reading for the standard used, and
 R_s = assigned reflectance factor of the standard for the filter used.

NOTE 12—The scale linearity of an instrument may be checked by reading the reflectance factors of a series of ten or more nonselective standards ranging from 85 to 0.5 %.⁸

10. Report

10.1 Report values of reflectance factor in percent relative to the perfect reflecting diffuser as 100 (as defined by U.S. National Bureau of Standards). The result is described as "45-deg, 0-deg directional reflectance factor." When the green filter is used, the words "daylight luminous" shall be inserted before "directional." When the blue or amber filters are used, the phrase "for blue light" or "for amber light," respectively, shall be added after "reflectance factor." Report reflectance factor values to the nearest 0.1 %. For paper specimens, report values for the wire and felt sides separately.

10.2 Describe or otherwise identify the method of preparing laboratory specimens.

10.3 Identify the reflectometer used by the manufacturer's name, the model, and the serial number.

11. Precision

11.1 *Precision (Repeatability and Reproducibility)*—Repeatability is the deviation from mean experienced when measuring a single specimen on a single instrument. Note that with many samples measured, excessive nonuniformity by area, or by method of presentation to the instrument, is the chief factor limiting repeatability. Reproducibility is the deviation from mean experienced between different instruments employing calibrated standards of nearly the same reflectance factor. Refer to Table 1.

TABLE 1 Precision Data^a

NOTE—All values refer to standard deviations.

Measurement	Reproducibility	Repeatability
Directional blue reflectance	0.4	0.2
Y Reflectance factor	0.7	not available
Z Reflectance factor	0.3	not available

^a TAPPI Collaborative Reference Program for Paper; NBS; Reports 1 to 10 and 28 to 33; MCCA Collaborative Reference Program for Color and Appearance.

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Standard Practice for Preparation of Pressed Powder White Reflectance Factor Transfer Standards for Hemispherical Geometry¹

This standard is issued under the fixed designation E 259; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

The internationally accepted standard of reflectance is the perfect reflecting diffuser. This ideal reflecting surface reflects 100 % of the radiant power incident on it such that the radiance is the same for all directions within the hemisphere of solid angles. No physical realization of this standard exists. Optical properties of standards prepared from pressed plaques of magnesium carbonate (MgCO_3), barium sulfate (BaSO_4), or polytetrafluoroethylene (PTFE) can approximate those of this ideal standard. For further information see CIE Publication No. 46 (1).² The principal use of a white reflectance factor standard is to transfer an absolute scale of reflectance to a more durable material or from one instrument to another. In theory, this transfer, done from first principles, should be quite easy. In practice, values are likely to be required for parameters that are unknown, proprietary, or require a highly sophisticated level of skill. Some, but not all, of those parameters are discussed in this practice.

Scope

1.1 This practice covers procedures for preparing pressed powder transfer standards of reflectance factor. These standards can be used in the near-ultraviolet, visible and near-infrared region of the spectrum. Procedures for calibrating the reflectance factor of materials on an absolute basis are contained in CIE Publication No. 44 (2). Pressed powder standards are used as transfer standards for such calibrations because they have a high reflectance factor which is nearly constant with wavelength and because the distribution of reflected flux resembles that from the perfect reflecting diffuser.

1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

Referenced Documents

- 2.1 *ASTM Standards*:
E 284 Terminology of Appearance³
D 1457 Specification for Polytetrafluoroethylene (PTFE) Molding and Extrusion Materials⁴

¹ This practice is under the jurisdiction of ASTM Committee E-12 on Appearance of Materials and is the direct responsibility of Subcommittee E12.02 on Spectrophotometry and Colorimetry.

Current edition approved Aug. 15, 1991. Published October 1991. Originally published as E 259 - 65. Last previous edition E 259 - 66 (1987).

² The boldface numbers in parentheses refer to the list of references at the end of this practice.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 08.01.

D 4894 Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials⁵

3. Terminology

3.1 *Definitions*—All terms used in this practice are consistent with Terminology E 284. The following three definitions are particularly important to this practice:

3.1.1 *reflectance*—the ratio of the flux reflected from a specimen to the flux incident on the specimen.

3.1.2 *perfect reflecting diffuser*—ideal reflecting surface that would neither absorb nor transmit light, but would reflect diffusely, with the radiance of the reflecting surface the same for all reflecting angles, regardless of the angular distribution of the incident light.

3.1.3 *reflectance factor*—the ratio of flux reflected from a specimen to the flux reflected from a standard surface, under the same geometric conditions of measurement. Unless otherwise indicated, the standard surface is assumed to be the perfect reflecting diffuser.

4. Summary of Practice

4.1 Procedures are given for the preparation of white reference standards of diffuse reflectance factor. The recommended materials are white powders that are pressed into plaques. These plaques provide close approximations to the optical properties of the perfect reflecting diffuser, and may be used to transfer a scale of absolute reflectance to another material or to an instrument.

4.2 Previous editions of this practice included guidelines for the preparation of standards from magnesium oxide smoke. Preparation of this type of standard is no longer recommended. The ease of preparation, physical ruggedness,

⁵ *Annual Book of ASTM Standards*, Vol 08.03.

and photometric stability of pressed powder plaques make them far superior to smoked magnesium oxide plaques as reflectance factor standards.

5. Significance and Use

5.1 All commercial reflectometers measure relative reflectance. The instrument reading is the reflectance factor, the ratio of the light reflected from a reference specimen to that reflected from a test specimen. That ratio is dependent on specific instrument parameters.

5.2 National standardizing laboratories and some research laboratories measure reflectance on instruments calibrated from basic principles, thereby establishing a scale of absolute reflectance as described in CIE Publication No. 44 (2). These measurements are sufficiently difficult that they are usually left to laboratories that specialize in them.

5.3 A standard that has been measured on an absolute scale could be used to transfer that scale to a reflectometer. While such procedures exist, the constraints placed on the mechanical properties restrict the suitability of some of the optical properties, especially those properties related to the geometric distribution of the reflected light. Thus, reflectance factor standards which are sufficiently rugged and cleanable to use as permanent transfer standards depart considerably from the perfect diffuser in the geometric distribution of reflected radiance.

5.4 The geometric distribution of reflected radiance from a pressed powder plaque is sufficiently diffuse so that such a standard can provide a dependable calibration of a directional-hemispherical reflectometer. Although pressed powder standards are subject to contamination and breakage, the directional-hemispherical reflectance factor of pressed powder standards can be sufficiently reproducible from specimen to specimen made from a given lot of powder, so as to allow one to assign absolute reflectance factor values to all the powder in a lot.

5.5 This practice describes how to prepare white reflectance factor standards from a powder in a manner that allows a standardizing laboratory to assign the absolute scale of reflectance to the powder.

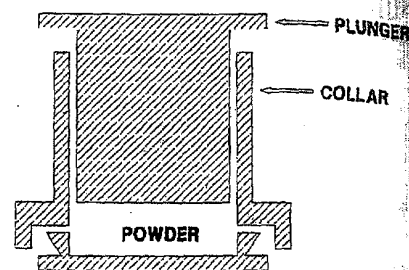
6. Apparatus

6.1 The basic apparatus for producing a pressed powder standard includes a powder press, powder containers and a balance. There are presently two commercial suppliers of powder presses.⁶ The press and receptacles can also be made in a local machine shop. A suggested configuration is shown in Fig. 1. The optical surface of the plaque should be pressed against a surface of ground glass or poly(methyl methacrylate) to provide a matte finish on the pressed plaque. Powder receptacles should be at least 5 mm deep for BaSO₄ and at least 10 mm deep for PTFE.

8. Reagents and Materials

8.1 *Barium Sulfate*—the barium sulfate should be of the

⁶ Powder press conforming to ISO 2469 and DIN 5033 is available from Carl Zeiss Canada, Ltd., 45 Valleybrook Drive, Don Mills, Ontario M3B-2S6, Canada, Part Number 505866; and Technidyne Corporation, 100 Quality Avenue, New Albany, IN 47150-2272, Part Number 176601.



RECEPTACLE

NOTE—The collar and receptacle should be securely held in place before pressing the powder.

FIG. 1 Example Powder Press

highest purity. It should be specially refined for optical and spectroscopic use (3).⁷

8.2 *Polytetrafluoroethylene*—the PTFE (4)^{8,9,10} should also be specially refined for optical and spectroscopic use but some commercial grades have been found to be acceptable substitutes. There is currently no commercial source for small quantities of optical grade PTFE powder. Large quantities (drums) of commercial grade PTFE can be obtained from the manufacturers.

9. Procedure

9.1 All powdered reflectance standards should be stored in tightly capped glass containers. If the powder is purchased in plastic containers it should be transferred to a glass container as soon as possible. Before using the powder, it should be placed in a glass blender equipped with stainless-steel or PTFE-coated blades and pulverized to a uniform consistency. The quantity of powder to be used should be transferred with stainless steel or PTFE-coated spoons. The whole operation should be performed in a draft-free location, away from sources of small particulate contamination; filters, sweaters, windows, ovens, etc. All measurements (weight, height, width, depth, volume, area, etc.) should be performed with adequate precision to ensure that the final density is within 5 % of the specified value. The most reproducible standards are made by pressing the powder to a specific density. Thus, the mass of the powder to be used should be determined from the volume of the receptacle.

9.2 *Barium Sulfate*—BaSO₄ should be pressed to a density of 2000 kg/m³ (2.0 gm/cm³) and a thickness of at least 5 mm. Several specimens should be pressed in succession. Matched pairs should be selected to be representative of the contents of the bottle of powder. The pressed plaques should be kept in a covered desiccator when not in use. Some suppliers of BaSO₄ provide calibration values with each

⁷ One such material, Kodak White Reflectance Standard 6091, available from Eastman Kodak Company, Laboratory and Research Products Division, Rochester, NY 14650, has been found suitable for this purpose.

⁸ PTFE-M-12, available from Daikin Industries, Ltd., 1-1 Nishihitotsuya Yodogawa, Siesakusho, Setto-Shi, Osaka, Japan, has been found suitable.

⁹ TEFLON™ 7A, available from E. I. DuPont de Nemours & Co., Inc., Barley Mill Plaza, Wilmington, DE 19880, has been found suitable.

¹⁰ AlgoFlon F5, available from AUSIMONT USA, Inc., CN 1838-T, Morristown, NJ 07960, has been found suitable.

TABLE 1 6°/Diffuse Reflectance Factor of Eastman White Reflectance Standard^a Pressed BaSO₄ Powder^a

Wavelength, nm	Reflectance Factor
300	0.966
350	0.979
400	0.987
450	0.991
500	0.991
550	0.992
600	0.992
650	0.992
700	0.992
750	0.992
800	0.992
850	0.991
900	0.990
950	0.988
1000	0.986

^a Density = 2000 kg/m³ and thickness = 5 mm.

TABLE 2 6°/Diffuse Reflectance Factor of Pressed PTFE Powder^a

Wavelength, nm	Reflectance Factor ^a
300	0.984
350	0.990
400	0.998
450	0.993
500	0.994
550	0.994
600	0.994
650	0.994
700	0.994
750	0.994
800	0.994
850	0.994
900	0.994
950	0.994
1000	0.994

^a Density = 1000 kg/m³ and thickness ≥ 5 mm.

^b Accurate to ±0.002.

bottle of powder, other suppliers provide only 3 or 4 quality reference checks and a reference to published values of reference standards prepared from the powder. Table 1 gives the 6°/diffuse reflectance factor values for Eastman White Reflectance Standard.⁶

9.3 *Polytetrafluorethylene*—PTFE should be pressed to a density of 1000 kg/m³ (1.0 gm/cm³) and a thickness of at least 10 mm. The spectral reflectance reaches a broad maximum near this density. Several specimens should be prepared in succession. Matched pairs are selected to be representative of the contents of the bottle of powder. The pressed plaques should be kept in a desiccator when not in

TABLE 3 Average and Standard Deviation of 6°/Diffuse Reflectance Factors of 17 PTFE Plaques Prepared by 9 Laboratories

Wavelength, nm	Reflectance Factor	
	Average ^a	Standard Deviation ^b
300	0.9792	0.0063
350	0.9883	0.0021
400	0.9911	0.0018
500	0.9919	0.0020
600	0.9915	0.0023
700	0.9914	0.0023
800	0.9912	0.0024
1000	0.9910	0.0024

^a Average Density = 926.2 kg/m³.

^b Standard Deviation = 85.7 kg/m³.

use. PTFE can be very sensitive to particulate contamination and electro-statically attracts airborne particles. Such contamination can make the material slightly fluorescent and reduce its reflectance in the ultraviolet spectral region. Table 2 lists the 6°/diffuse reflectance factor values for PTFE as determined by the National Institute of Standards and Technology (NIST).

10. Precision and Bias

10.1 The National Institute of Standards and Technology and the Inter-Society Color Council Project Committee 22, Materials for Instrument Calibration, have carried out collaborative tests to determine the precision and bias of the preparation of PTFE reflectance factor standards (5). The standard deviation of three determinations of the reflectance factor of PTFE by the NIST ranged from 0.0002 to 0.0008 over the spectral range 300 to 1000 nm. The measured reflectances of PTFE from two manufacturers exhibited differences of from -0.002 to +0.004 over the same range with the largest differences near the ends of the range and a constant measurement uncertainty of ± 0.005. From the 9 laboratories participating in the round-robin experiment, 17 specimens were returned. The results are shown in Table 3 for the wavelength range 300 to 1000 nm.

10.2 CIE Publication Number 46 (6) cites literature references on the reproducibility of BaSO₄ pressings that range from 0.05 % to 1.0 % with the most common value in the range about 0.2 %. This puts the reproducibility of the plaque preparation near the level of the reproducibility of the international standardizing laboratories' ability to characterize the absolute reflectance of the material.

REFERENCES

- (1) *Publication CIE No. 46—A Review of Publications on Properties and Reflection Values of Material Reflection Standards*, available from U.S. National Committee CIE (International Commission on Illumination), % Director of Marketing, OEM Division, North American Philips Lighting Corp., Philips Square, CN6800, Somerset, NJ 08873.
- (2) *Publication CIE No. 44—Absolute Methods for Reflection Measurements*, available from U.S. National Committee CIE (International Commission on Illumination), % Director of Marketing, OEM Division, North American Philips Lighting Corp., Philips Square, CN6800, Somerset, NJ 08873.
- (3) Grum, F., and Wightman, T. E., "Absolute Reflectance of Eastman White Reflectance Standard", *Applied Optics*, Vol 16, 1977, pp. 2775-2776.
- (4) Weidner, V. R., and Hsia, J. J., "Reflection Properties of Pressed Polytetrafluoroethylene Powder," *J. Opt. Soc. Am.*, Vol 71, 1981, pp 856-861.
- (5) Weidner, V. R., Hsia, J. J., and Adams, B., "Laboratory Inter-comparison Study of Pressed Polytetrafluoroethylene Powder Reflectance Standards," *Applied Optics*, Vol 24, 1985, pp. 2225-2230.
- (6) *Publication CIE No. 38—Radiometric and Photometric Characteristics of Materials and Their Measurement*, available from U.S. National Committee CIE (International Commission on Illumination), % Director of Marketing, OEM Division, North American Philips Lighting Corp., Philips Square, CN6800, Somerset, NJ 08873.

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Standard Test Methods for Measurement of Gloss of High-Gloss Surfaces by Goniophotometry¹

This standard is issued under the fixed designation E 430; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 These test methods cover the measurement of the reflection characteristics responsible for the glossy appearance of high-gloss surfaces. Two test methods, A and B, are provided for evaluating such surface characteristics at specular angles of 30° and 20°, respectively. These test methods are not suitable for diffuse finish surfaces nor do they measure color, another appearance attribute.

1.2 As originally developed by Tingle and others (see Refs 1 and 2),² the test methods were applied only to bright metals. Recently they have been applied to high-gloss automotive finishes and other nonmetallic surfaces.

Referenced Documents

- 2.1 *ASTM Standards*:
 - D 523 Test Method for Specular Gloss³
 - D 2457 Test Method for Specular Gloss of Plastic Films and Solid Plastics⁴
 - E 171 Specification for Standard Atmospheres for Conditioning and Testing Materials⁵
 - E 179 Practice for Selection of Geometric Conditions for Measurement of Reflectance and Transmission Properties of Materials⁶
 - E 284 Terminology of Appearance⁶
 - E 308 Test Method for Computing the Colors of Objects by Using the CIE System⁶
 - E 1347 Test Method for Color and Color-Difference Measurement of Object-Color Specimens by Tristimulus (Filler) Colorimetry⁶

Terminology

3.1 Definitions:

3.1.1 Reflectance and Related Terms:

3.1.1.1 *reflectance, ρ , n* —ratio of the reflected radiant or luminous flux to the incident flux in the given conditions. (E 284)

(1) *Discussion*—The term *reflectance* is often used in a general sense or as an abbreviation for *reflectance factor*.

¹ These test methods are under the jurisdiction of ASTM Committee E-12 on Appearance of Materials and are the direct responsibility of Subcommittee E 12.03 on Geometry.

Current edition approved Aug. 15, 1991. Published November 1991. Originally published as E 430 - 71. Last previous edition E 430 - 78 (1983).

² The boldface numbers in parentheses refer to the list of references at the end of this method.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vol 08.02.

⁵ *Annual Book of ASTM Standards*, Vol 15.09.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

Such usage is not assumed in this method. The definition may require that the term be modified by adjectives denoting the spectral and geometric conditions of measurement.

3.1.1.2 *reflectance factor, R , n* —ratio of the flux reflected from the specimen to the flux reflected from the perfect reflecting diffuser under the same spectral and geometric conditions of measurement. (E 284)

3.1.1.3 *gloss reflectance factor, R_g , n* —ratio of the specularly reflected part of the (whole) flux reflected from the specimen to the flux reflected from a specified gloss standard under the same geometric and spectral conditions of measurement.

(1) *Discussion*—The gloss standard may be a black glass or a mirror, and may be assigned one of a variety of scale values G_s as specified. (E 284)

3.1.2 Gloss and Types of Gloss:

3.1.2.1 *gloss, n* —angular selectivity of reflected light, responsible for the degree to which reflected highlights or images of objects may be seen as superimposed on a surface. (E 284)

(1) *Discussion*—Gloss is responsible for the object's glossy appearance, not for an observer's perception of the appearance. At least six types or characteristics of gloss may be observed depending upon the character of the surface and the spatial distribution of the reflected light (3).

3.1.2.2 *specular gloss, n* —ratio of flux reflected in specular direction to incident flux for a specified angle of incidence and source and receptor angular apertures. (E 284)

3.1.2.3 *distinctness-of-image gloss, n* —aspect of gloss characterized by the sharpness of images of objects produced by reflection at a surface. (E 284)

3.1.2.4 *sheen, n* —the specular gloss at a large angle of incidence for an otherwise matte specimen. (The usual angle for measurement is 85°.) (E 284)

3.1.3 Terms Relating to Surface Characteristics:

3.1.3.1 *directionality, n —perceived*, the degree to which the appearance of a surface changes as the surface is rotated in its own plane, under fixed conditions of illumination and viewing. (E 284)

3.1.3.2 *texture, n* —the visible surface structure depending on the size and organization of small constituent parts of a material; typically, the surface structure of a woven fabric. (E 284)

3.1.4 Other Terms:

3.1.4.1 *aperture angle, 2θ , n* —angle subtended at a point on a specimen by the maximum dimension of the illuminator or receiver, within which the flux in a directional beam is contained.

(1) *Discussion*—In optics, the symbol θ is used for the

half angle; hence the recommended symbol here is 2_k (E 284)

3.1.4.2 *goniophotometer*, n —instrument that measures flux as a function of angles of illumination or observation. (E 284)

3.2 Terms Specific to This Test Method:

3.2.1 Terms Relating to Reflection Haze:

3.2.1.1 *reflection haze*, H , n —for a specified specular angle, ratio of flux reflected at a specified angle (or angles) from the specular direction to the flux similarly reflected at the specular angle by a specified gloss standard.

(1) *Discussion*—Modifiers may be used to specify the angles at which the haze is measured (for example, 2° or 5°); whether H or a logarithmic form is to be stated; or whether H is to be compensated for the luminance of the specimen by multiplication by Y_{specimen}/Y_n , where n denotes the reference white; or any combination of these.

3.2.2 Terms Relating to Metallic Reflection:

3.2.2.1 *metallic brightness*, n —freedom of a metal surface from diffuse haze or texture.

3.2.3 Terms Relating to Specimen Directionality:

3.2.3.1 *'with-machine' direction*, n —the axis of a specimen that is parallel to the direction of mill rolling or extrusion, or other surface-finish texture.

3.2.3.2 *'across-machine' direction*, n —the perpendicular to 'with-machine' direction.

3.3 Other appearance terms and definitions in Terminology E 284 are applicable to this test method.

4. Summary of Test Method

4.1 Several geometrically different measures of light reflected by a surface are proposed for use in describing its gloss appearance. In Test Method A, gloss reflectance factor is measured at 30° to the specimen normal using narrow illuminator and receiver aperture angles (0.5° wide maximum). Distinctness-of-image gloss is measured at 29.7° or 30.3° or both. Narrow-angle (2°) reflection haze is measured at 28 or 32° or both, and wide-angle (5°) reflection haze at 25 or 35° or both. The ratio of reflectance factors for 28 or 32° or both, perpendicular and parallel to the machine direction of the specimen, is computed as a measure of directionality. In Method B, specular gloss is measured at 20° according to Test Method D 523, and narrow-angle reflection haze is measured at 18.1 and 21.9° .

5. Significance and Use

5.1 The gloss of metallic finishes is important commercially on metals for automotive, architectural, and other uses where these metals undergo special finishing processes to produce the appearances desired. It is important for the end-products which use such finished metals that parts placed together have the same glossy appearance.

5.2 It is also important that automotive finishes and other high-gloss nonmetallic surfaces possess the desired finished appearance. The present method identifies by measurements important aspects of finishes. Those having identical sets of numbers normally have the same gloss characteristics. It usually requires more than one measurement to identify properly the glossy appearance of any finish (see Refs 3 and 4).

6. Apparatus

6.1 The apparatus shall be either an abridged goniophotometer (see Figs. 1A and 1B) or a full goniophotometer (see Fig. 2) that can be set to the specified specular, off-specular, and aperture angles given in Tables 1A and 1B. The abridged goniophotometer may have a fixed angle of incidence (for Test Method A, 30° ; for Test Method B, 20°) and specific fixed directions of view at which the flux from the specimen is measured (see Tables 1A and 1B).⁵

6.1.1 *Geometric Conditions for Test Method A*—The direction of incidence shall be 30° . The directions of view shall be opposite the direction of incidence at 30° deg for specular reflectance, 29.7 or 30.3° deg for distinctness of image comparisons, 28 or 32° deg for narrow-angle haze comparisons, and 25 or 35° deg for wide-angle haze comparisons. The angular dimensions of the mirror reflected image of the source slit in the plane of measurement and the angular dimensions of the receiver windows in this plane of measurement shall be as shown in Table 1A.

NOTE 1—The conventional notation indicating positive angles for incident conditions and negative angles for viewing conditions has been used, thus far, to reduce confusion. In the remaining sections, the negative notation is omitted, since only the viewing conditions are described.

6.1.2 *Geometric Conditions for Test Method B*—The direction of incidence shall be $20 \pm 0.1^\circ$. The directions of view shall be opposite the direction of incidence, at 20° for specular gloss measurement and at 18.1 and 21.9° for narrow-angle reflection haze measurement. The angular dimensions of the specularly reflected image of the source slit in the plane of measurement and the angular dimensions of the receiver windows in this plane of measurement shall be as shown in Table 1B.

6.1.3 *Spectral Conditions*—The measurement shall be made with visible light to give results in accordance with the CIE spectral luminous efficiency function $V(\lambda)$ (identical with $\bar{y}$) in the CIE 1931 standard observer) and CIE standard illuminant C (see Test Methods E 308 and E 1347). If another illuminant A, is used, this shall be specified in the report.

6.1.4 *Polarization*—The incident flux shall be unpolarized and the receiver shall be insensitive to the state of polarization of the reflected luminous flux.

6.1.5 *Clamp*—For Test Method A, a rotatable clamp of the type shown in Fig. 3 may be used for flattening and positioning the specimen during measurement.

7. Standards

7.1 Three calibrated standards of good planarity shall be available in either a set of metals or a set of nonmetals, depending upon which type of surface is measured.

7.2 High-Gloss Standards:

7.2.1 *High-Gloss Standard for Metals* shall be of aluminum, evaporated onto glass and covered with a protective coating of silicon monoxide, and calibrated for specular gloss and distinctness-of-image gloss.

7.2.2 *High-Gloss Standard for Nonmetals* shall be of highly polished black glass with a refractive index of approximately $n_D = 1.527$, calibrated for specular gloss and reflection haze, and assigned a scale value of $G_s = 89.4$ for a

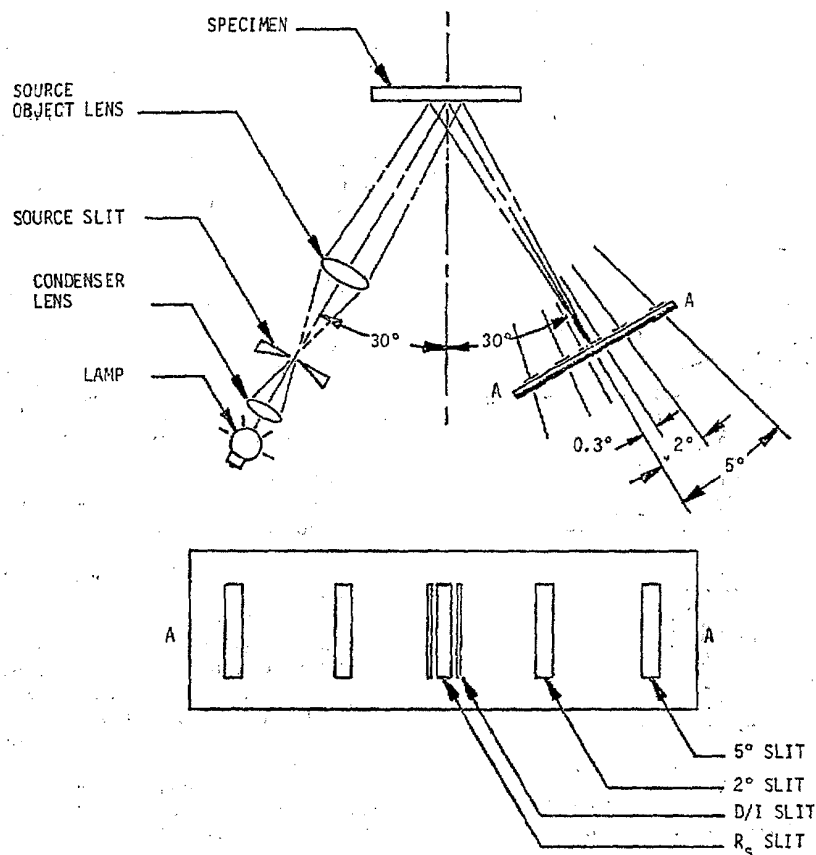


FIG. 1A Optical Diagram of the Abridged Goniophotometer

ular angle of 30° (Test Method A) or $G_s = 89.2$ for a specular angle of 20° (Test Method B).

2—To determine the scale value, calculate the first-surface reflectance (Test Method D 2457, Section 5.1) for $n_D = 1.567$ at the specular angle of interest; for 30° it is 5.0436 % and for 20° it is 4.3769 %. Assign this a scale value of 100. (Test Method D 523, Section 5.1) Repeat the calculation for $n_D = 1.527$ and the same specular angle; the result for 30° is 4.5069 % and for 20°, 4.3769 %. The new scale value for 30° is $100 \times (4.5069/5.0436) = 89.4$, and for 20°, $100 \times (4.3769/4.3769) = 89.2$. (The latter value is also given in Test Method D 523.)

8. Intermediate Standards:

8.1 *Intermediate Standard for Metals* shall be of either chromium evaporated onto glass and covered with a protective coating of silicon monoxide, or of bright sheet aluminum with protective coating and calibrated for specular and distinctness-of-image gloss.

8.2 *Intermediate Standard for Nonmetals* shall be of a ceramic material, such as porcelain enamel on steel, and calibrated for specular gloss and distinctness-of-image gloss.

8.3 *Diffuse Standards* shall have a reflectance factor substantially constant over the angular range of the instrument.

8.4 *Care of Standards*—It is essential that the standards

be kept clean and free of scratches as well as from contact with contaminating materials. The cleaning method specified by the instrument manufacturer shall be followed and the standards should be checked at regular intervals against reference standards held in reserve.

8. Specimens

8.1 Specimens shall be obtained from test samples by selecting areas that are plane and representative of each sample being tested. Every specimen must be at least 2 3/4 in. (70 mm) in the smallest dimension. Specimens may be larger so long as it is possible to insert them and flatten them properly for measurement.

9. Preparation and Calibration of Apparatus

9.1 The instrument must be used in a clean dry area free of drafts. Standard laboratory conditions are recommended (see Section 2 of Specification E 171). Voltage regulation to ± 0.01 % must be incorporated in the instrument, or supplied separately. Follow manufacturer's recommendations for instrument warmup.

9.2 *Calibration*—Adjust the instrument to read the same

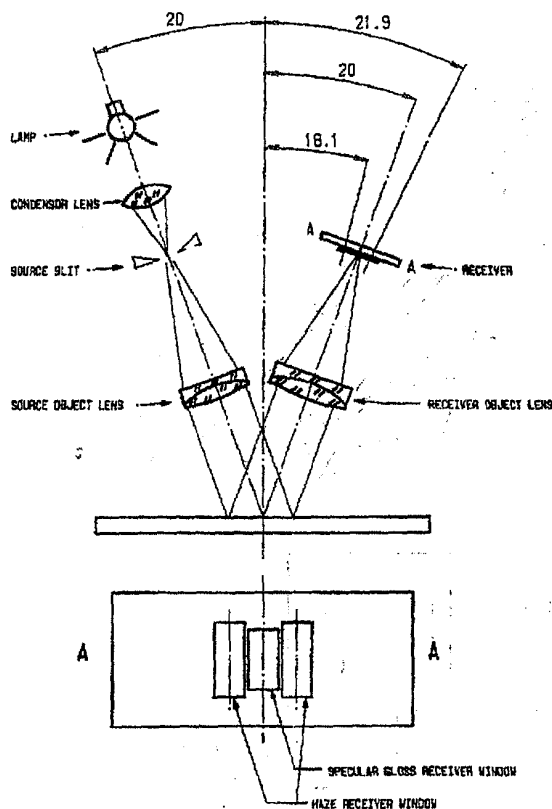


FIG. 1B Optical Diagram of the Apparatus for Method B

gloss reflectance factor for the intensity of light reflected from the diffuse standard through the specular, distinctness-of-image, and haze apertures. Adjust the instrument to read values of specular gloss and distinctness-of-image gloss assigned the aluminum mirror if metal surfaces are being measured; or the black gloss standard if nonmetal surfaces are being measured. If the instrument does not then read the appropriate intermediate standard within the limits set by the instrument manufacturer, refocus or recalibrate following the manufacturer's instructions.

10. Procedure

10.1 Bring the specimen to the instrument for measurement. Be sure the specimen is flat.

10.2 For Test Method A, be certain that the specular sensor is centered on the specimen-reflected specular light beam. One type of instrument, illustrated in Fig. 1A, provides for screw-controlled movement of the receptor assembly to achieve this centering. The full goniophotometer, shown in Fig. 2, identifies the specular direction by the peak of the goniophotometric curve.

10.3 Rotate the specimen in its own plane to find the orientation, giving the maximum specular signal. This specimen orientation is called the "machine direction" because it

generally coincides with the direction of travel of a sheet or film material through a processing machine.

10.4 Record the following quantities:

10.4.1 For Test Method A, readings of (a) gloss reflectance factor (specular gloss), $R_{s,30}$ at 30° ; (b) distinctness-of-image gloss; (c) 2° reflection haze, H_2 ; and (d) 5° reflection haze, H_5 . The quantities in (b), (c), and (d) may be either gloss reflectance factors or values of H , which are their ratios to the specular gloss reflectance factor recorded in (a).

10.4.2 For Test Method B, readings of (a) the 20° specular gloss, $R_{s,20}$; (b) the reflection haze, H ; and (c) the luminous reflectance, Y .

10.5 Measure at least three areas of each specimen.

10.6 From these same areas, read 2-deg haze for the "across-machine direction," being careful to flatten the test surface and orient the specular beam in each case.

10.7 Take readings on the standards at the end of the series of observations to ensure that the instrument has remained in calibration throughout the operation.

11. Calculation

11.1 For Test Method A, calculate the mean of three readings for each specimen for:

11.1.1 Specular gloss, $R_{s,30}$, at 30° .

11.1.2 Distinctness-of-image gloss, $100 \times (1 - H_{0.3})$, evaluated at 0.3° on either or both sides of the specular angle.

11.1.3 2° Reflection haze, $100 H_2$, evaluated at 2° on either or both sides of the specular angle.

11.1.4 5° Reflection haze, $100 H_5$, evaluated at 5° on either or both sides of the specular angle.

11.1.5 Directionality, $100 \times (H_{2, \text{across-machine}} / H_{2, \text{with-machine}})$, evaluated at 2° on either or both sides of the specular angle.

11.2 For Test Method B, calculate the mean of three readings of each specimen for:

11.2.1 Specular gloss, $R_{s,20}$ at 20° .

11.2.2 One or more of the following, as required:

11.2.2.1 Reflection haze, H_{20} .

11.2.2.2 Logarithmic reflection haze, $H_{20, \log} = 1285 \log[(H_{20}/20) + 1]$.

11.2.2.3 Compensated reflection haze, $H_{20, \text{comp}} = H_{20, \text{specimen}} - (H_{20, n} \times Y_{\text{specimen}} / Y_n)$.

NOTE 3— $H_{20, \log}$ may also be calculated as a compensated quantity by using $H_{20, \text{comp}}$ in place of H_{20} in 11.2.2.2. Compensated quantities shall be used when comparing specimens with different values of Y .

12. Report

12.1 The report for Test Method A shall contain the following:

12.1.1 The specular gloss $R_{s,30}$.

12.1.2 The distinctness-of-image gloss.

12.1.3 The 2° haze, H_2 .

12.1.4 The 5° reflection haze, H_5 .

12.1.5 The directionality.

12.2 The report for Test Method B shall contain the following:

12.2.1 The specular gloss $R_{s,20}$.

12.2.2 Either the logarithmic reflection haze or the compensated logarithmic reflection haze.

12.3 The report for both methods shall contain the following:

12.3.1 Identification of instrument used by model and serial number.

FIG. 2

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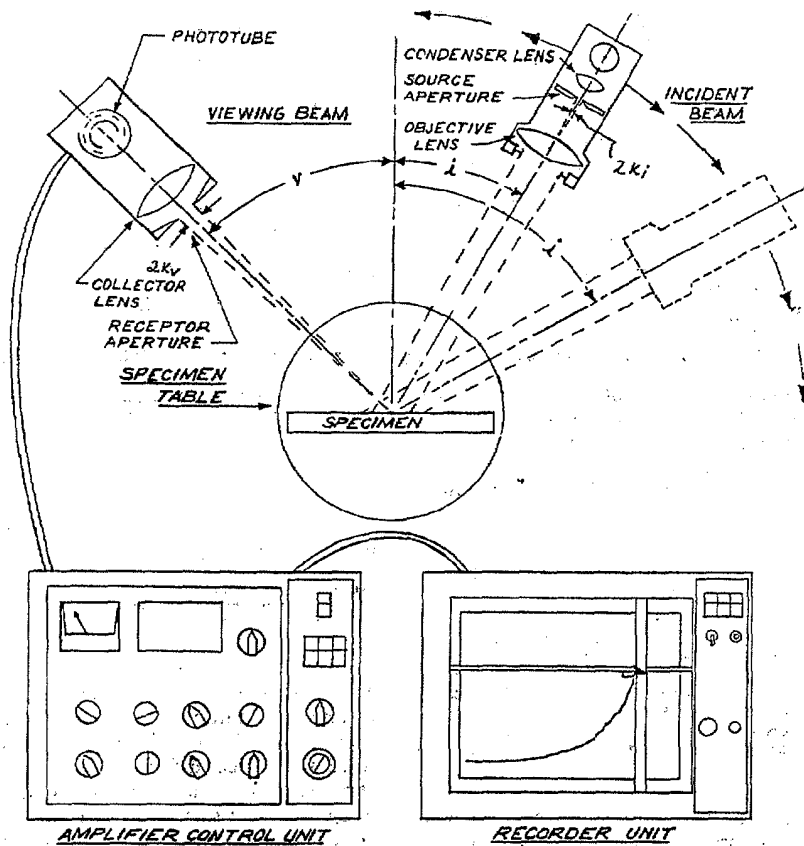


FIG. 2 Recording Goniophotometer Showing Viewing Angle, v , Incidence Angle, i , and Their Respective Field Angles, $2K_v$ and $2K_i$

TABLE 1A Dimensions of the Mirror Image of the Source-Slit, and of the Receptor Windows Measured in the Plane of the Receiving Windows (see Fig. 1A)

	Source-Slit Mirror Image, deg	Specular Receiver Window, deg	Distinctness-of-Image (± 0.29) Receiver Window, deg	Haze Receiver Windows, deg
Angle of center of window (measured from perpendicular to specimen surface)	30	30	30.3 and 29.7	28 and 32 or 25 and 35
Width (in the plane of the angle of reflection)	0.44 ± 0.01	0.4 ± 0.01	0.14 ± 0.01	0.4 ± 0.1 or 0.5 ± 0.1
Length (across the plane of the angle of reflection)	5.0 ± 1	3.0 ± 1	3.0 ± 1	3.0 ± 1

TABLE 1B Dimensions of the Specular Image of the Source-Slit and of the Receptor Windows Measured in the Plane of the Receiving Windows (see Fig. 1B)

	Source-Slit Specular Image, deg	Specular Gloss Receiver Window, deg	Haze Receiver Window, deg
Angle of center of window (measured from perpendicular to specimen surface)	20.0 ± 0.1	20.0 ± 0.1	18.1 ± 0.1 and 21.9 ± 0.1
Width (in the plane of the angle of reflection)	0.75 ± 0.1	1.8 ± 0.05	1.8 ± 0.1
Length (across the plane of the angle of reflection)	2.5 ± 0.25	3.6 ± 0.1	5.5 ± 0.25

12.3.2 Identification of gloss standards by number and scale value assigned, and

12.3.3 Identification of any specimens whose values on any scale differ by more than 3.0 in individual readings from the averages reported.

13. Precision and Bias

13.1 Test Method A:

13.1.1 An indication of the sensitivity of this test method is shown in Table 2 by the values of Spearman rank correlation coefficients. The data were obtained with a set of 20 aluminum and stainless steel specimens selected for a

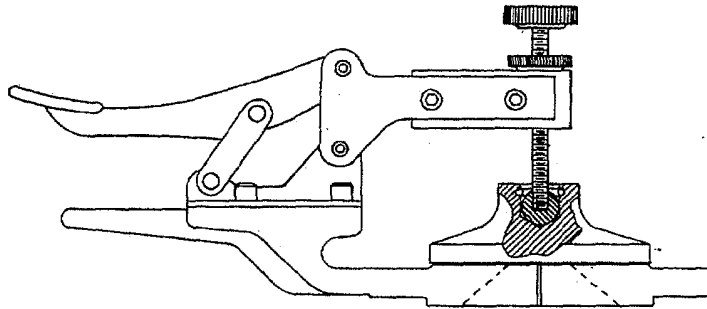


FIG. 3 A Rotatable Clamp Suggested for Flattening the Specimen and Positioning It During Measurement

TABLE 2 Rank Correlation Coefficients of Distinctness-of-Image and Haze Between the Dori-Gon Abridged Goniophotometer and Other Instruments, and with Visual Judgments

Dori-Gon Versus	Distinctness-of-Image ^a	Haze ^b	
		2 deg	5 deg
Visual	0.91	0.82	0.96
D10-5 Gonio	0.93	...	0.96
Alcoa DORI	0.87	...	...
Alcoa Abridged Gonio	...	...	0.98
D36B Distinctness-of-Image Glossmeter	0.94	...	...

^a Visual judgments of rank were made by sighting in the "with" machine direction only, whereas instrumental "with" and "across" directions were averaged.

^b Visual judgments of haze were made by ranking the specimens according to the amount of near specular reflectance (miliness) adjacent to the image of a concentrated light source. Correlation between the Dori-Gon and the D10-5 Gonio and between the Dori-Gon and the Alcoa Abridged Gonio at 2 deg were equivalent to those at 5 deg.

wide range of reflectance characteristics. Visual evaluations were compared with measurements using (1) the abridged goniophotometer, (2) a full goniophotometer, and (3) other instruments. A more complete report of these findings appears in Ref (5).

TABLE 3 Instrument Reproducibility Data

	Root-Mean-Square Differences from Goniophotometer Assigned Values	
	30-deg Specular Reflectance	Distinctness-of-Reflected Image
One instrument with receptor windows filled with optical fibers	1.4	1.5
Average, two instruments with segmented silicon photocell light receivers	2.2	1.1

13.1.2 *Instrument Reproducibility*—Five panels of anodized aluminum sheet, ranging from 12 to 77 in specular reflectance and from 24 to 97 in distinctness-of-image gloss, were calibrated with the full goniophotometer, then measured with three abridged goniophotometers. One of the abridged goniophotometers had optical fibers filling the receptor windows and two instruments had receptor windows of segmented silicon photocells. The results of the full goniophotometer calibration are given in Table 3.

13.2 Test Method B:

13.2.1 Precision and Bias will be determined.

REFERENCES

- (1) Tingle, W. H., and Potter, F. R., "New Instrument Grades for Polished Metal Surfaces," *Product Engineering*, Vol 27, March 1961.
- (2) Tingle, W. H., and George, D. J., "Measuring Appearance Characteristics of Anodized Aluminum Automotive Trim," Report No. 650513, Society of Automotive Engineers, May 1965.
- (3) Hunter, R. S., "Gloss Evaluation of Materials," *ASTM Bulletin* 186, ASTBA, December 1952.
- (4) Christie, J. S., "Instruments for Metallic Appearance," *Appearance of Metallic Materials*, ASTM STP 478, Am. Soc. Testing Mats., ASTTA, 1971.
- (5) Christie, J. S., "An Instrument for the Geometric Attributes of Metallic Appearance," *Applied Optics*, Vol. 8, No. 9, September 1969.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Abrasion Resistance of Pipeline Coatings¹

This standard is issued under the fixed designation G 6; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method of accelerated test is a procedure for determining the relative resistance of steel pipeline coatings to abrasion by a slurry of coarse abrasive and water. The method is intended to apply to the testing of all types of electrical insulating pipeline coatings and tapes, including thermoplastics, thermoset, and bituminous materials.

1.2 Pipeline coatings are not normally subjected to the type of abrasion herein specified.

1.3 Metallic protective coatings such as zinc may be compared visually, but do not meet the electrical requirements of this test method.

1.4 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 The values stated in SI units to three significant decimals are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

- G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²
- G 62 Test Methods for Holiday Detection in Pipeline Coatings³

3. Definition

3.1 *holidays*—small faults or pinholes which permit current drainage through protective coatings on steel pipe.

4. Summary of Test Method

4.1 Apparatus and materials are described whereby coatings on steel pipe are worn away by an abrasive slurry contained in a horizontally revolving drum. Specimens extend through both ends of the drum and are electrically insulated from contact with the test apparatus.

4.2 Periodic measurement of the electrical resistance between the specimen and the drum indicates the degree of

abrasion resistance of the coating.

5. Significance and Use

5.1 This procedure defines a test method for comparing the relative resistance of pipeline coatings to abrasion.

5.2 Abrasion resistance may be used to specify optimum coating thickness of candidate materials both in development and research work to study new coating systems or methods and in quality control.

6. Apparatus

6.1 *Drum*—A cylindrical steel container suitable for testing nine specimens simultaneously. Figures 1 to 3 show construction details which may be altered at the cap assembly if desired. A valve shall be provided to release any pressure built up during test.

6.2 *Apparatus for Revolving the Drum*—Any suitable apparatus used to revolve the drum at 30.48 m (100 linear ft)/min. A modified jar rolling mill³ has been found satisfactory for revolving the drum.

6.3 *Thickness Gage*, to be used in accordance with Method G 12.

6.4 *Holiday Detectors*, low-voltage wet-sponge type for thin film coatings (coatings thinner than 20 mils), and high-voltage type for thick film coatings (coatings thicker than 20 mils).

NOTE 1—Low voltage detectors are used to locate pinholes, voids, or thin spots in pipeline coatings. The potential used with wet-sponge detectors can be up to 100 V DC. Procedures for using these detectors are found in Method A of G 62. For use of high voltage detectors operating in the 900 to 20 000 Vdc range, see Method B of G 62.

6.5 *Volt-ohm-meter*—Any electrical test instrument for measuring electrical resistance of the circuit.

7. Reagents and Materials

7.1 The test slurry shall consist of the following:

- 7.1.1 *Aluminum Oxide Grit*,⁴ 13.6 kg (30 lb).
- 7.1.2 *Tap Water*, 5.68 L (1½ gal).

8. Test Specimen

8.1 A 19.1 mm test specimen 406 mm (16 in.) long shall be prepared with its surface preparation and coating procedures equivalent to that of production coated pipe. Only holiday-free specimens, in duplicate, shall be used in this test.

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

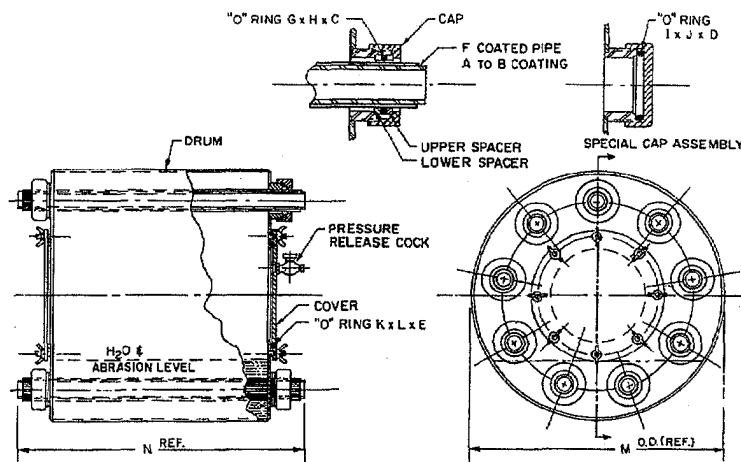
Current edition approved May 27, 1988. Published July 1988. Originally published as G 6 - 69 T. Last previous edition G 6 - 83.

² *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Figure 801-V Jar Mill (U.S. Stoneware, Inc., Akron, OH) provides a suitable means for revolving the drum.

⁴ Exolon aluminum oxide abrasive, No. 3 mesh, Type TA, untumbled, available from Exolon Co., Tonawanda, NY 14150, has been found satisfactory for this purpose.



Dimensions for Fig. 1:

Symbol	mm	in.
A	0.15	0.006
B	2.28	0.090
C	3.17	0.125
D	4.76	0.188
E	6.35	0.250
F	19.05	0.750
G	25.40	1.000
H	31.75	1.125
I	38.10	1.500
J	47.63	1.875
K	136.53	5.375
L	149.23	5.875
M	355.6	14.0
N	406.4	16.0

NOTE—Estimated weight includes water and abrasive 39.5 kg (87 lb)

FIG. 1 Abrasion-Tester Assembly

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8.2 Control specimens shall be included in each test, the selection of which is optional with the user.

9. Conditioning

9.1 The specimen shall be exposed to a room temperature of 21° to 25°C (70° to 77°F) for a period of 24 h before beginning the test.

10. Procedure

10.1 Perform the test at a room temperature of 21° to 25°C (70° to 77°F).

10.2 Measure and record the coating thickness of each specimen in accordance with Method G 12.

10.3 Check all specimens or holidays using an appropriate detector.

10.4 Position the specimens in the drum taking care not to damage the coating.

10.5 Load the apparatus with 13.6 kg (30 lb) of unused abrasive and 5.68 L (1½ gal) of water.

10.6 Test each specimen for electrical resistance after sealing the drum by using the volt-ohm-meter. Immerse the specimen in the slurry at the time the electrical resistance is measured. Reject and replace any specimen showing less than infinite resistance.

10.7 Revolve the drum at the rate of 30.5 m (100 linear ft)/min.

10.8 Determine the electrical resistance at 25-h intervals for a test period of 200 h.

10.9 Relieve the pressure built up during the test by opening the release valve each time the electrical resistance is measured.

11. Report

11.1 The report shall include the following:

11.1.1 Complete identification of test specimens, including name and code number of coating, size of the pipe, production date, production run number, and any other information that may be pertinent to identification,

11.1.2 Date of starting and of terminating the test,

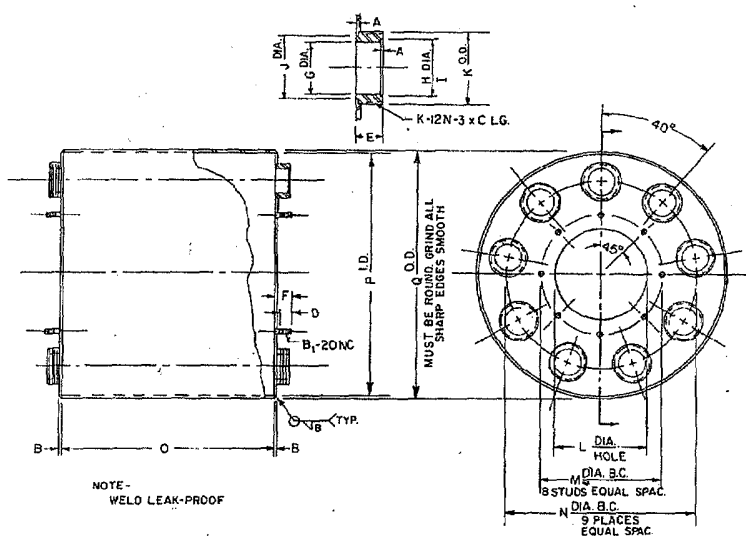
11.1.3 Coating thickness of the test specimen before testing and minimum coating thickness after testing,

11.1.4 Volt-ohm-meter readings at 25-h intervals, and

11.1.5 Description of coating appearance after testing.

12. Precision

12.1 Precision data are limited to two adjacent specimens taken from the same production-coated pipe, assuming that the production process was uniform with respect to pipe



Dimensions for Fig. 2:

Symbol	mm	in.
A	1.59	0.063
B	3.18	0.125
B ₁	6.35	0.250
C	12.70	0.500
D	19.05	0.750
E	22.23	0.875
F	25.40	1.000
G	38.10	1.500
H	41.25	1.624
I	41.28	1.625
J	44.45	1.750
K	50.8	2.0
L	127.0	5.0
M	165.1	6.5
N	266.7	10.5
O	304.8	12.0
P	349.25	13.75
Q	355.6	14.0

FIG. 2 Drum

surface condition and coating material. Specimens that were not adjacent in the as-produced condition, or were taken from different lengths of pipe may represent differing process conditions.

12.2 Repeatability—When more than one test of the same coating material is involved, the standard deviation of the results should not be greater than 1.0 times the hourly time interval between inspections for the specific coating material. The standard deviation⁵ of a set of numbers is the square root of the average of the squares of the deviations of the numbers from their average. The standard deviation of two or more specimen results may be found by the following

procedure:

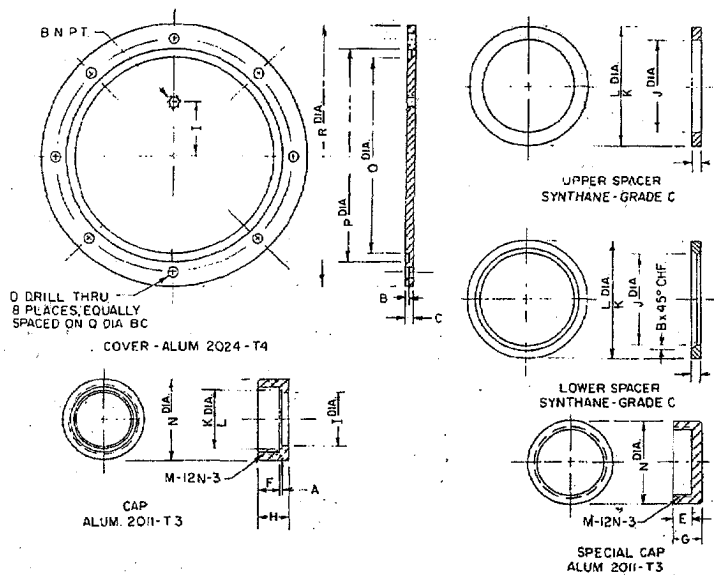
$$\text{Standard deviation, } \sigma = 1/n[(n\sum X^2 - (\sum X)^2)^{1/2}]$$

where:

- $\sum X$ = sum of all values of X , from X_1 to X_n inclusive,
- n = number of observed values (observations), and
- σ = standard deviation, the root-mean-square (rms) deviation of the observed values from their average.

12.3 Reproducibility—Agreement between laboratories should be such that the standard deviation of the results for a specific coating should not be greater than 1.5 times the inspection time interval.

⁵ For more information see the *ASTM Manual on Presentation of Data and Control Chart Analysis*, STP 15D, October 1976.



Dimensions for Fig. 3:

Symbol	mm	in.
A	1.59	0.063
B	3.18	0.125
C	6.35	0.250
D	7.94	0.313
E	12.70	0.500
F	15.88	0.625
G	19.05	0.750
H	22.23	0.875
I	38.10	1.500
J	25.68	1.281
K	41.25	1.624
L	41.28	1.625
M	50.80	2.0
N	51.05	2.250
O	136.53	5.375
P	149.23	5.875
Q	165.10	6.500
R	184.15	7.250

FIG. 3 Details

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Standard Test Methods for Cathodic Disbonding of Pipeline Coatings¹

This standard is issued under the fixed designation G 8; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover accelerated procedures for simultaneously determining comparative characteristics of insulating coating systems applied to steel pipe exterior for the purpose of preventing or mitigating corrosion that may occur in underground service where the pipe will be in contact with inland soils and may or may not receive cathodic protection. They are intended for use with samples of coated pipe taken from commercial production and are applicable to such samples when the coating is characterized by function as an electrical barrier.

1.2 This test method is intended for testing coatings submerged or immersed in the test solution at room temperature. When it is impractical to submerge or immerse the test specimen, Test Method G 95 may be considered where the test cell is cemented to the surface of the coated pipe specimen. If higher temperatures are required, see Test Method G 42. If a specific test method is required with no options, see Test Method G 80.

1.3 The values stated in SI units to 3 significant decimals are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

G 42 Method for Cathodic Disbonding of Pipeline Coatings Subjected to Elevated Temperatures²

G 80 Test Method for Specific Cathodic Disbonding of Pipeline Coatings³

G 95 Test Method for Cathodic Disbondment Test of Pipeline Coatings (Attached Cell Method)³

3. Summary of Test Methods

3.1 Both of the two test methods described subject the coating on the test specimen to electrical stress in a highly conductive, alkaline electrolyte. Electrical stress is obtained either by means of a sacrificial magnesium anode or from an

impressed current system. The coating is perforated before starting the test.

3.1.1 In Method A, a magnesium anode is used with no electrical monitoring during the test period. The results are determined by physical examination after the test period is concluded.

3.1.2 In Method B, either a magnesium anode or an impressed current system may be used. Electrical instrumentation is provided for measuring the current in the cell circuit. The electrical potential is also measured, and upon conclusion of the test period, the test specimen is physically examined.

3.1.3 In both test methods physical examination is conducted by comparing the extent of loosened or disbonded coating at the perforations in the immersed area with extent of loosened or disbonded coating at a new test hole in the coating made in an area that was not immersed.

4. Significance and Use

4.1 Breaks or holidays in pipe coatings may expose the pipe to possible corrosion, since after a pipe has been installed underground, the surrounding earth will be more or less moisture-bearing and it constitutes an effective electrolyte. Damage to pipe coating is almost unavoidable during transportation and construction. Normal soil potentials as well as applied cathodic protection potentials may cause loosening of the coating, beginning at holiday edges, in some cases increasing the apparent size of the holiday. Holidays may also be caused by such potentials. While apparently loosened coating and cathodic holidays may not result in corrosion, this test provides accelerated conditions for loosening to occur and therefore gives a measure of resistance of coatings to this type of action.

4.2 The effects of the test may be evaluated by either physical examination or monitoring the current drawn by the test specimen or both of these two. Usually there is no correlation between the two methods of evaluation but both methods are significant. Traditional physical examination consists of assessing the effective contact of the coating with the metal surface in terms of observed differences in the relative adhesive bond. It is usually found that the electrically stressed area propagates from the holiday to a boundary where the loosened coating leaves off for the more effective contact or bond attributed to an original condition throughout the specimen before electrical stressing was applied. Assumptions associated with test results include the following:

4.2.1 Attempting to loosen or disbond the coating at a new test hole made in the coating in an area that was not immersed represents maximum adhesion or bond as measured by the lifting technique used, and that the same lifting

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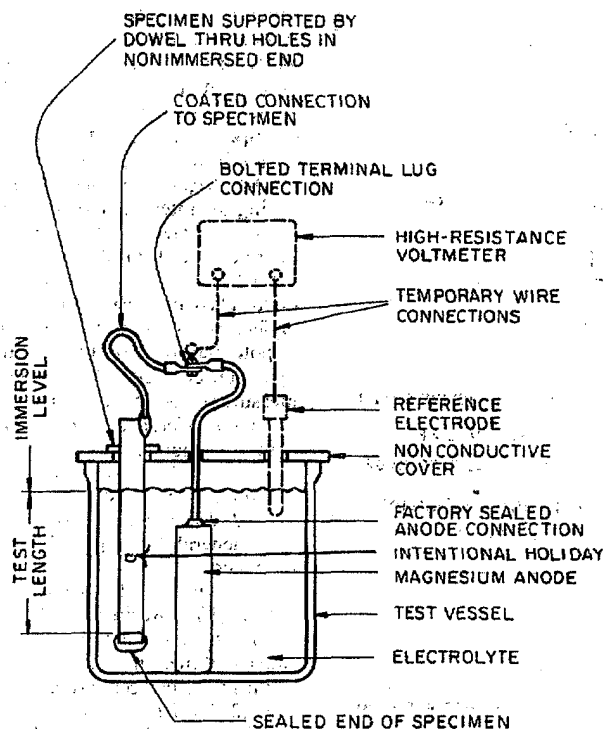
used for

¹ These test methods are under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and are the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 25, 1990. Published July 1990. Originally published as G 8 - 69 T. Last previous edition G 8 - 85.

² Annual Book of ASTM Standards, Vols 06.01 and 14.02.

³ Annual Book of ASTM Standards, Vol 14.02.



NOTE—Test hole made in non-immersed area after testing not shown (see Fig. 5).

FIG. 1 Test Assembly for Method A Using a Magnesium Anode

technique can be used at a test hole that was immersed thereby providing a means of comparing relative resistance to lifting.

4.2.2 Any relatively lesser bonded area at the immersed test holes in the coating was caused by electrical stressing and was not attributable to an anomaly in the application process. Ability to resist disbondment is a desired quality on a comparative basis, but disbondment per se in this test is not necessarily an adverse indication. The virtue of this test is that all dielectric type coatings now in common use will disbond to some degree thus providing a means of comparing one coating with another. Bond strength is more important for proper functioning of some coatings than others and the same measured disbondment for two different coating systems may not represent equivalent loss of corrosion protection.

4.2.3 The amount of current in the test cell is a relative indicator of the extent of areas requiring protection against corrosion; however, the current density appearing in this test is much greater than that usually required for cathodic protection in natural, inland soil environments.

5. Apparatus

5.1 Apparatus for Both Methods:

5.1.1 *Test Vessel*—A nonconducting material shall be used for the vessel or as a lining in a metallic vessel.

Dimensions of the vessel shall permit the following requirements:

5.1.1.1 Test specimens shall be suspended vertically in the vessel with at least 25.4-mm (1-in.) clearance from the bottom.

5.1.1.2 Each test specimen shall be separated from the other specimens, from the anodes and from the walls of the test vessel by at least 38.1 mm (1.500 in.).

5.1.1.3 Depth of electrolyte shall permit the test length of the specimen to be immersed as required in 7.4.

5.1.1.4 If electrical monitoring is to be performed as required in Method B, the reference electrode may be placed anywhere in the vessel, provided it is separated from the specimen and from the anode by not less than 38.1 mm (1.500 in.).

5.1.2 *Magnesium Anode*—The anode shall be made of a magnesium alloy having a solution potential of -1.45 to -1.55 V with respect to a CuCuSO_4 reference electrode in the electrolyte given in 6.1. It shall have a surface area not less than one third that of the total specimen area exposed to electrolyte (outside area exposed only). The anode shall be provided with a factory-sealed, 4107-cmil (14-gage Awg), minimum, insulated copper wire. Anodes without a factory seal may be used if the magnesium extends above the cover.

5.1.3 *Connectors*—Wiring from anode to test specimen shall be 4107-cmil (14-gage Awg), minimum, insulated copper. Attachment to the test specimen shall be by sol-

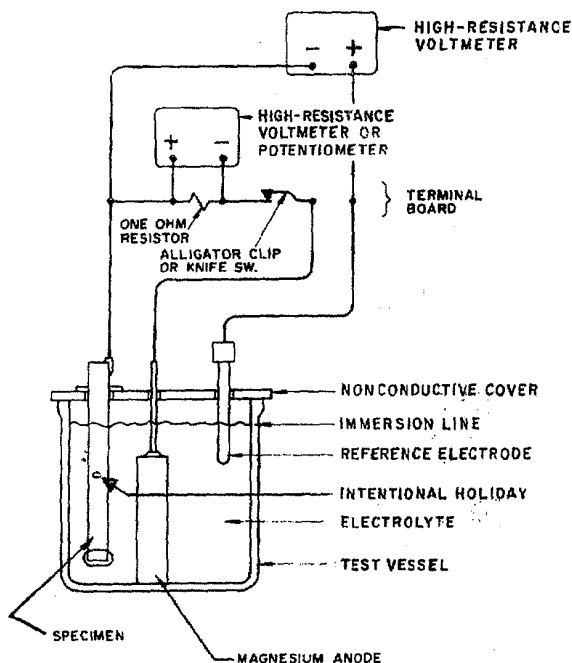


FIG. 2 Test Assembly for Method B Using a Magnesium Anode

dering, brazing, or bolting to the nonimmersed end, and the place of attachment shall be coated with an insulating material. A junction in the connecting wire is permitted, provided that it is made by means of a bolted pair of terminal lugs soldered or mechanically crimped to clean wire ends.

5.1.4 *Holiday Tools*—Holidays shall be made with con-

ventional drills of the required diameter. For use in preparing small-diameter pipe specimens such as 19.05 mm (0.750 in.) nominal diameter pipe, the use of a drill modified by substantially grinding away the sharp cone point has been found effective in preventing perforation of the metal wall of the pipe. A sharp-pointed knife with a safe handle is required for use in making physical examinations.

5.1.5 *High-Resistance Voltmeter*, for direct current, having an internal resistance of not less than 10 MΩ and having a range from 0.01 to 5 V for measuring potential to the reference electrode.

5.1.6 *Reference Electrode*, saturated CuCuSO_4 of conventional glass or plastic tube with porous plug construction, preferably not over 19.05 mm (0.750 in.) in diameter, having a potential of -0.316 V with respect to the standard hydrogen electrode. A calomel electrode may be used, but measurements made with it shall be converted to the CuCuSO_4 reference for reporting by adding -0.072 V to the observed reading.

5.1.7 *Thickness Gage*, for measuring coating thickness in accordance with Test Method G 12.

5.1.8 *Thermometer*, for measuring electrolyte temperature, general lab type, 1° subdivisions, 76.2 mm (3 in.) immersion.

5.2 *Additional Apparatus for Method B:*

5.2.1 *High-Resistance Voltmeter*, for direct current, having an internal resistance of not less than 10 MΩ and capable of measuring as low as $10 \mu\text{V}$ potential drop across a shunt in the test cell circuit.

5.2.2 *Precision Wire-Wound Resistor*, $1\text{-}\Omega \pm 1\%$, 1-W (minimum), to be used in the test cell circuit as a shunt for current.

5.2.3 *Volt-Ohm-Meter*, for initial testing of apparent coating resistance.

5.2.4 *Metallic Electrode*, used temporarily with the volt-ohm-meter to determine apparent initial holiday status of the test specimen.

5.2.5 *Additional Connecting Wires*, 4107-cmil (14-gage

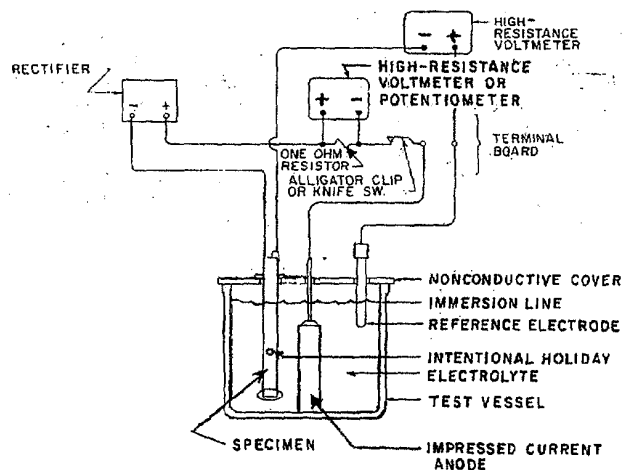


FIG. 3 Test Assembly for Method B Using an Impressed Current with One Specimen

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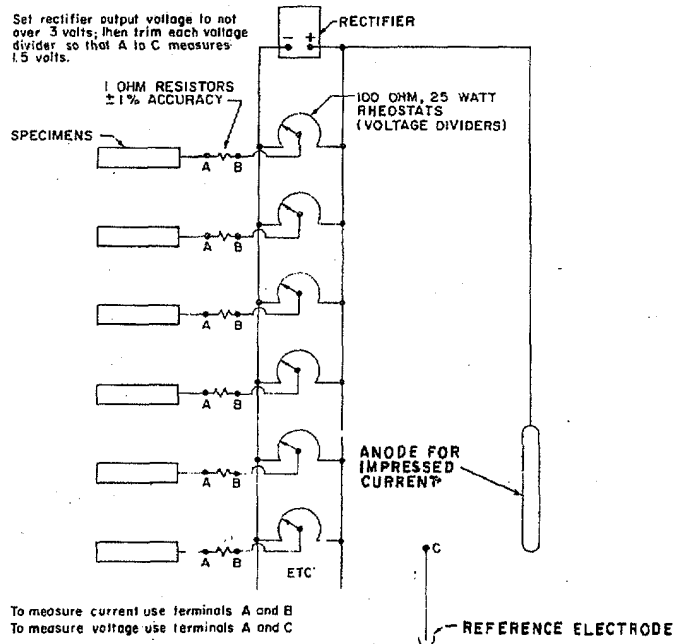


FIG. 4 Modification of Method B (Fig. 3) Using Impressed Current to Test More than One Specimen

(wg), minimum, insulated copper.

5.2.6 *Brass Studs*, used at a terminal board, together with alligator clips or knife switches, for making and breaking circuits. Alligator clips shall not be used to connect to electrodes or specimens at the top location of test cells.

5.2.7 *Zero-Resistance Ammeter*, capable of measuring direct current as low as 10 μ A may be used in the alternative method given in 9.1.3 and substituted for the apparatus described in 5.2.1 and 5.2.2.

5.2.8 *Direct-Current Rectifier*, capable of supplying constant voltage at a voltage of 1.50 ± 0.01 V, as measured between the specimen and reference electrode.

5.2.9 *Impressed Current Anode*, shall be of the nonconsumable type provided with a factory sealed, insulated copper wire.⁴

5.2.10 *Voltage Divider*, 100-Ω, 25-W rheostat, to be used if more than one specimen is to be tested as shown in Fig. 4.

6. Reagent and Materials

6.1 The electrolyte shall consist of potable tap water with the addition of 1 mass % of each of the following technical-grade salts, calculated on an anhydrous basis: sodium chloride, sodium sulfate, and sodium carbonate. Use freshly prepared solution for each test.

6.2 Materials for sealing the ends of coated pipe specimens may consist of bituminous products, wax, epoxy, or other materials, including molded elastomeric or plastic end caps.

⁴ Durion, a material found suitable for this purpose is available from Durion Co., Inc., Dayton OH.

6.3 Plywood or plastic material has been found suitable for the construction of test vessel covers and for the support through apertures of test specimens and electrodes. Wood dowels introduced through holes in the top ends of test specimens have been found suitable for suspending test specimens from the vessel cover.

7. Test Specimen

7.1 The test specimen shall be a representative piece of production-coated pipe. One end shall be plugged or capped, and sealed.

7.2 One or three holidays shall be made in each specimen. Three holidays are recommended. Recommended dimensions are given in Fig. 5. A specimen with one holiday shall have it drilled in the middle of the immersed length. If three holidays are used, they shall be drilled 120° apart with one in the center and the other two at locations one fourth the distance from top and bottom of the immersed test length. Each holiday shall be drilled so that the angular cone point of the drill will fully enter the steel where the cylindrical portion of the drill meets the steel surface. The drill diameter shall be not less than three times the coating thickness, but it shall never be smaller than 6.35 mm (0.250 in.) in diameter. The steel wall of the pipe shall not be perforated. With small-diameter pipes, where there is danger of perforating the pipe, the holiday shall be started with a standard 60° cone point and finished with a drill that has had a substantial portion of the cone point ground away.

NOTE 1—Before making the holiday, see 8.1.

7.3 The end of the pipe which will protrude above the

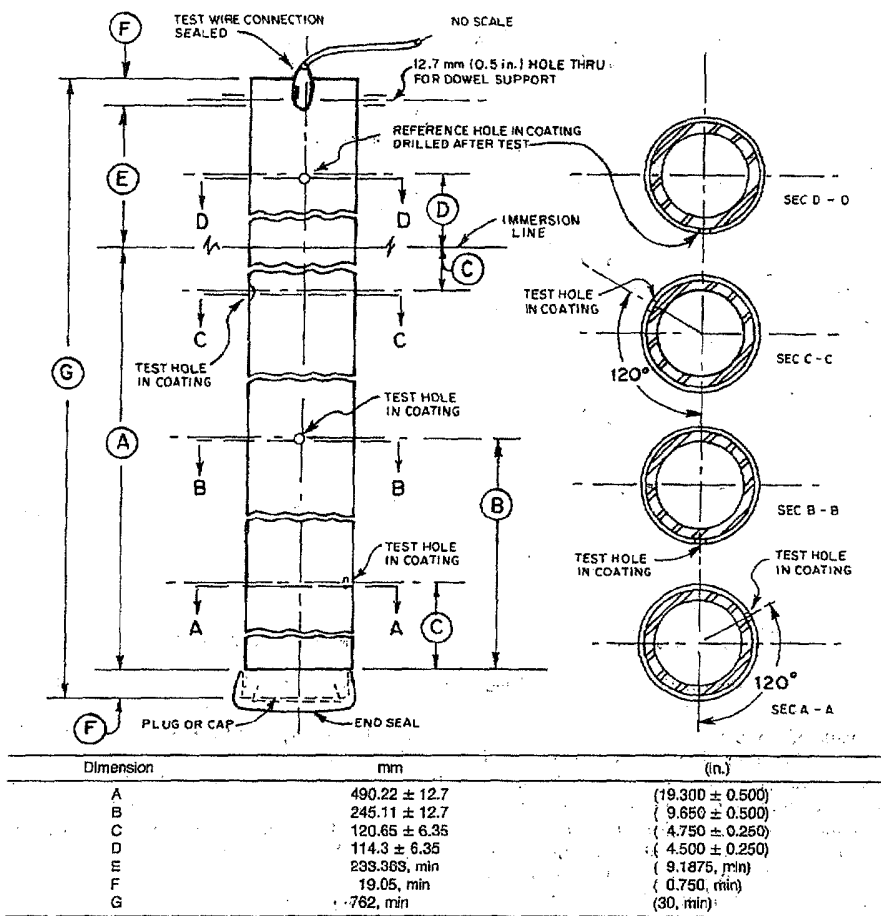


FIG. 5 Recommended Dimensions for Specimen

immersion line shall be provided with suitable supporting means and a separate wire connection for electrical purposes, soldered, brazed, or bolted to the pipe. The protruding end, including hanger and wire connections, shall be protected and sealed with an insulating coating material.

7.4 The specimen test area shall consist of the area between the edge of the bottom end seal and the immersion line. The bottom end seal area shall not be considered part of the area tested. Any suitable diameter and specimen length of pipe may be used, but the immersed area shall be not less than 23 227 mm² (36 in.²). An area of 92 900 mm² (1 ft²) has been found preferable when convenient.

8. Specimen Preparation

8.1 Before making artificial holidays, verify the continuity of the coating and the effectiveness of the end-cap seal as follows:

8.1.1 Immerse the test specimen and a metallic electrode in the electrolyte. Connect one terminal of the multimeter to the test specimen and the other terminal to the metallic

electrode. Measure the apparent resistance in ohms, making two determinations: one with the specimen connected to the positive terminal of the multimeter; and one with the specimen connected to the negative terminal.

8.1.2 Disconnect the specimen from the multimeter but leave it immersed for 15 min. Then, measure the resistance again as in 8.1.1.

8.1.3 A significant decrease in either resistance reading after 15-min will indicate a flaw in the coating or end-cap seal. Reject the specimen if the flaw is identified in the coating. If the flaw is in the end-cap seal, it may be repaired and the resistance remeasured as in 8.1.1 and 8.1.2.

8.1.4 The lowest resistance after 15 min of immersion shall be not less than 1000 M Ω but a stable reading below 1000 M Ω may not indicate a flaw and the specimen may be used for test. All resistance measurements shall be reported in the results.

8.2 Record initial holiday diameter(s).

8.3 Measure and record the minimum and maximum coating thickness in accordance with Method G 12, and the

Data Sheet and Report, Part I, for Method A and Method B Cathodic Disbonding of Pipeline Coatings

No. _____ Report No. _____ Initials _____ Date _____

_____ mm (in.) O.D. _____ mm (in.) Wall _____ mm (in.) Length

_____ API _____

_____ Method _____

_____ mm (in.)

Min. _____ Av. _____ At holidays: Top _____ Middle _____ Bottom _____

_____ Date finished _____

_____ mm² (in.²)

Initial holiday dia. mm (in.) _____

Final unsealed area mm² (in.²) _____

(→) Initial holiday area mm² (in.²) _____

= Net disbonded area mm² (in.²) _____

Disbonded Equivalent Circle Diameter mm² (in.²) _____

Top	Middle	Bottom	Average

5. Preliminary verification

Largest Disbonded Equivalent Circle Diameter (ECD) does not exceed			Verification of coating continuity before starting test per Sec. 7.6			
mm	in.	Spontaneous Holidays	Trial	Polarity	Megohms	
					Initial	After 15 min.
12.7	0.50	None	Initial	Plus		
25.4	1.00	None		Minus		
38.1	1.50	None		Plus		
50.8	2.00	None	Final	Minus		
More than 50.8	More than 2.00	Any				

Current: _____

Current was not continuous indicate interrupted time (min., hrs.): _____

FIG. 6 Suggested Form, Part I, for Use in Presenting Data for One Specimen Method A and Method B

where each holiday is made.

Procedure for Method A

Immerse the test specimen in the electrolyte and connect to the anode as shown in Fig. 1. Position the test specimen so that it faces away from the anode. Connect the anode with respect to test specimens as described in Fig. 1. Mark the correct immersion level of the test specimen with a grease pencil and maintain by daily addition of potable water as required. Perform the test at a temperature of 21 to 25°C (70 to 77°F). In order to ascertain that the test cell is functioning, measure the potential between test specimen and a reference electrode immediately after starting the test and immediately terminating it. Use temporary connections and instrumentation, as shown in Fig. 1. The potential measured shall

be -1.45 V to -1.55 V with respect to a CuCuSO₄ reference electrode. Use the instrument described in 5.1.5.

9.2 Duration of the test period shall be 30 days. Optionally, other test periods such as 60 or 90 days may be used.

9.3 An examination shall be performed immediately upon termination of the test period as follows:

9.3.1 At the end of the test period, disassemble the cell and rinse the test area with warm tap water. Immediately wipe the sample dry and visually examine the entire test area for any evidence of unintentional holidays and loosening of coating at the edge of all holidays, including the intentional holiday, and record coating condition, for example, color, blisters, cracking, crazing, adhering deposits, etc.

9.3.2 Drill a new reference holiday in the coating in an area that was not immersed. Follow the same drilling procedure as described in 7.2.

Initial Ohms +

[illegible]

9.3.3 Make radial 45° cuts through the coating intersecting at the center of both the intentional holiday and the reference holiday with a sharp, thin-bladed knife. Take care to ensure that coating is cut completely through to the steel substrate.

9.3.4 Attempt to lift the coating at both the reference holiday and the intentional holiday with the point of a sharp, thin-bladed knife. Use the bond at the reference holiday as a reference for judging the quality of the bond at the intentional holiday. Measure and record the total area of disbonded coating at the intentional holiday.

NOTE 2—The use of a transparent film having a grid laid out in small squares such as 2.54 mm (0.1 in.) on a side has been found useful. The film is placed against the unsealed area and the boundary of the unsealed area traced on the grid. The area is then obtained by counting the squares within the bonded area.

10.1 In addition to the procedure given in Section 9, monitor the progress of the test electrically in accordance with the schedule given in 10.2 as follows:

10.1.1 If a magnesium anode is to be used, install the test assembly shown in Fig. 2. If impressed current for a single specimen is to be used, install the test assembly shown in Fig. 3; if more than one specimen is to be tested, use the modification shown in Fig. 4.

10.1.2 Measure E_2 , the stress potential in volts between test specimen and reference electrode, with a multimeter without disconnecting the anode from the test specimen. Use the instrument described in 5.1.5. If a CuCuSO_4 electrode is used, immerse only temporarily.

10.1.3 Measure I_1 , the current demand in amperes, by

determining the potential drop across the 1- Ω resistor permanently installed in the test cell circuit with the multimeter described in 5.2.1. The voltage reading will be numerically equal to amperes.

NOTE 3—An alternative method of measuring current demand may be used by means of the instrument described in 5.2.4. In this method, the wire connection between test specimen and anode is temporarily broken and a zero-resistance ammeter temporarily interposed between the specimen and the anode. Reconnect the specimen to the anode with the connector wire as soon as this measurement is completed.

10.1.4 Measure E_1 , the polarized potential, in volts. Do this with the multimeter described in 5.1.5 connected between the test specimen and the reference electrode as follows:

10.1.4.1 Disconnect the anode from the test specimen while closely observing the multimeter. As the instrument pointer falls, it will dwell significantly at the polarized value before receding further. The dwell point is E_p . If a CuCuSO_4 electrode is used, immerse only temporarily.

10.2 Electrical Monitoring Schedule:

10.2.1 Electrical measurements at the start of the test are defined as the average of measurements taken on the second and third days after immersion. Measurements may be taken on the day of immersion in order to ascertain functioning of the test cell, but such measurements are not to be used in calculating the change in characteristics from start to target dates in the conduct of the test.

10.2.2 Make electrical measurements at the start of a test and on a target date after 30 days. The test may be continued for 60 or 90-day targets with intermediate and corresponding electrical measurements.

10.2.3 Take electrical measurements for intermediate

get dates and for the terminal date on 2 successive days for to and including the target date. The average of readings taken on the 2 days is defined as the target date measurement.

10.2.4 Rectifier current shall be continuous. Any interruptions must be reported.

Report (see Figs. 6 and 7):

11.1 The report for Method A shall include the following:

11.1.1 Complete identification of the test specimen, including:

11.1.1.1 Name and code number of the coating,

11.1.1.2 Size and wall thickness of pipe,

11.1.1.3 Source, production date, and production run number,

11.1.1.4 Minimum-maximum coating thickness, average thickness and the thickness at the holiday,

11.1.1.5 Immersed area,

11.1.1.6 Size and number of initial holidays, and

11.1.1.7 Resistance measurements verifying continuity of the coating and effectiveness of the end cap seal as required in 8.1.

11.1.2 Dates of starting and terminating test.

11.1.3 Tally of areas that have been found unsealed on the terminal date. Areas may be reported in square millimetres (square inches) or millimetres (inches) of equivalent circle diameter of the area, or both. If more than one holiday was used, the area per holiday may be reported as an average.

NOTE 4—Equivalent Circle Diameter (*ECD*) is obtained from the formula:

$$ECD = (A/0.785)^{1/2}$$

where:

A = area of holiday, mm^2 (in.^2)

11.1.4 Other information that may be pertinent.

11.2 The report for Method B shall include the following:

11.2.1 The data required in the report for Method A,

11.2.2 The relative resistances of the test specimen in ohms before the artificial holiday was made as described in 11.4, and

11.2.3 The results of starting, intermediate, and terminal electrical measurements. Report the following measurements:

11.2.3.1 Current demand in microamperes, or negative

characteristic of the logarithm of the current in amperes, or both,

11.2.3.2 The value of $\Delta E = E_2 - E_1$ in volts, and

11.2.3.3 Change from start to termination for values 11.2.3.1 and 11.2.3.2. If more than one holiday was used the average change per holiday may be reported for 11.2.3.1.

11.2.4 Any interrupted time of the rectifier current.

12. Precision and Bias

12.1 Precision data are limited to two adjacent specimens taken from the same production-coated pipe and assume that the production process was uniform with respect to pipe surface condition and coating material. Specimens that were not adjacent in the as-produced condition or were taken from different lengths of pipe may represent differing process conditions. The following data should be used for judging the acceptability of results: (These precision data are approximations based on limited data, but they provide a reasonable basis for judging the significance of results.)

12.2 Method A:

12.2.1 Repeatability—Duplicate results by the same worker should not be considered suspect unless they differ by more than 12.7 mm (0.5 in.) in value *ECD* in accordance with the following equation:

$$ECD = (A/0.785)^{1/2}$$

where:

A = unsealed area developed from 1 artificial holiday, mm^2 (in.^2).

12.2.2 Reproducibility—The results reported by one laboratory should not be considered suspect unless they differ from those of another laboratory by more than 25 mm (1 in.) for value *ECD* in the equation given in 12.2.1.

12.3 Method B:

12.3.1 Repeatability—Duplicate results by the same worker should not be considered suspect unless they differ by more than unity in the negative characteristic of the logarithm of the current demand in amperes.

12.3.2 Reproducibility—The results reported by one laboratory should not be considered suspect unless they differ from those of another laboratory by more than unity in the negative characteristic of the logarithm of the current demand in amperes.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Test Method for Water Penetration into Pipeline Coatings¹

This standard is issued under the fixed designation G 9; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers the determination of the apparent rate of depth of water penetration into insulating coatings applied to pipe.

1.2 The values stated in inch-pound units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

G12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

3. Summary of Method

3.1 The method consists of an immersion-type test where pipe specimens are suspended in an aqueous electrolyte for the duration of the test period. Electrical measurements of coating capacitance and dissipation factor are used to follow the water absorption rate of the test materials.

4. Significance and Use

4.1 The deterioration of an insulating coating film is intimately related to its moisture content. The water penetration test provides a means for monitoring the passage of moisture through a coating material by means of changes in its dielectric constant. When expressed in relation to time, the test data will reflect a rate of deterioration which is a characteristic of the coating material and will bear a relation to its expected useful life as an insulating coating. The test for water penetration will also provide information that is useful in establishing the optimum coating thickness for a given material.

5. Apparatus

5.1 *Immersion Cell*—Any suitable nonmetallic vessel to contain the test specimens. Dimensions of the vessel shall permit the following requirements:

5.1.1 Test specimens shall be suspended vertically with at least 25 mm (1.0 in.) clearance from the sides and bottom.

5.1.2 Test specimens shall be separated by not less than 25 to 40 mm (1 to 1.5 in.) and a vertically suspended anode shall be placed at an equal distance from each specimen not less than the separation of distance.

5.1.3 The test vessel shall be deep enough to allow for immersion of the samples in the electrolyte to the level specified in 8.1.

NOTE 1—Commercially available, glass battery jars in 2-dm³ (0.55-gal) and 10-dm³ (2.7-gal) sizes can be conveniently used with 19-mm (0.75-in.) and 51-mm (2.0-in. nominal) diameter specimens, respectively.

5.1.4 A suitable sample support plate fabricated from a material having a low dielectric constant shall be used to suspend the samples and anode above the immersion cell. The support plate shall contain an access hole for the reference electrode. A typical test cell is illustrated in Fig. 1.

5.2 *Electrolyte*, consisting of tap water with the addition of 1 weight % of each of the following technical-grade anhydrous salts: sodium chloride, sodium sulfate, and sodium carbonate.

NOTE 2—Add 30 g (1.05 oz) of sodium chloride for each dm³ (0.26 gal) of water.

5.2.1 The electrolyte in the immersion cell shall be maintained at the proper level by regular additions of tap water. The electrolyte shall not be reused after completion of the test.

5.3 *Voltage Source*—A direct current power supply, capable of supplying low ripple voltage shall be used to maintain a potential difference of 6.0 ± 0.1 V dc between each of the test specimens and a common electrode.

5.4 *Connectors*—Wiring connections from the anode to the specimen shall be of No. 18 AWG insulated copper. Attachment to the anode shall be sealed and kept above the level of the electrolyte. Attachment to the specimen shall be made by a method that will allow disconnection from the anode when the measuring bridge is in use. A convenient means for accomplishing this is through the use of insulated pin-type jacks.

5.5 *Capacitance Bridge*³—Measurements of equivalent specimen capacitance and coating dissipation factor shall be made with a low-voltage a-c, resistive-ratio-arm type measuring bridge having the following characteristics:

5.5.1 Oscillator frequency, 1 kHz $\pm$ 2 %,

5.5.2 Series capacitance range, 1 to 1100 pF $\pm$ 1 %,

5.5.3 Series capacitance sensitivity, 0.5 pF,

5.5.4 Dissipation factor range, 0.001 to 1.0 at 1 kHz, and

5.5.5 Dissipation factor sensitivity, 0.001 at 1 kHz.

¹ This method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 29, 1987. Published July 1987. Originally published as G 9 - 69 T. Last previous edition G 9 - 82.

² *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

³ A bridge found to meet the requirements specified is the General Model 1656 Impedance Bridge, GenRad, Concord, MA 01742.

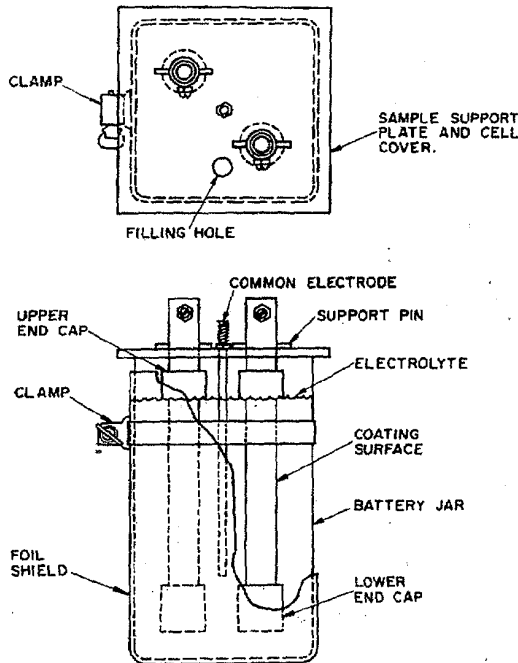


FIG. 1 Typical Test Cell

5.6 *Measuring Circuit*—Measurements of specimen capacitance and coating dissipation factor shall be made using a circuit that places the sample unknown in series with the

comparison circuit of the measuring bridge. Connection of the unknown to the measuring bridge shall be made in such a manner as to eliminate the introduction of stray capacitance into the measuring circuit. A diagram for connecting the test cell to the bridge is shown in Fig. 2. In this arrangement, both the test leads are shielded and the chassis of the bridge is grounded. The immersion cell shall also be shielded to avoid capacitance effects from surrounding objects.

NOTE 3—A shield for the test cell can conveniently be fabricated from most commercially-available tin or aluminum foils of approximately 0.0382-mm (0.0015-in.) thickness and formed around the container.

5.7 *Thickness Gage*—Measurements of coating thickness will be required for this test. Any instrument suitable for use with Test Method G 12 can be used.

5.8 *Anode*, fabricated from 4.76-mm (0.1875-in.) diameter AISI Type 303 stainless-steel rod, and shall be 178 mm (7.00 in.) long, with the upper 50 mm (2.00 in.) threaded to accept a locking nut.

6. Test Specimen

6.1 The test specimen shall be a representative piece of production-coated pipe and shall be free of obvious coating flaws or defects (see Fig. 3). Any suitable diameter and specimen length can be used. Physical limitations of the immersion cells suggested in 5.1.3, Note 1, make it necessary to restrict the over-all sample length to approximately 300 mm (12.0 in.) for both the 26.7-mm (0.75-in. nominal) and 60.3-mm (2.0-in. nominal) diameter coated pipe specimens.

6.2 The upper and lower ends of the test specimen shall be plugged and sealed with nonconductive caps of sufficient bulk to minimize effectively capacitive end effects in the measuring circuit. For this purpose, an end-cap thickness of

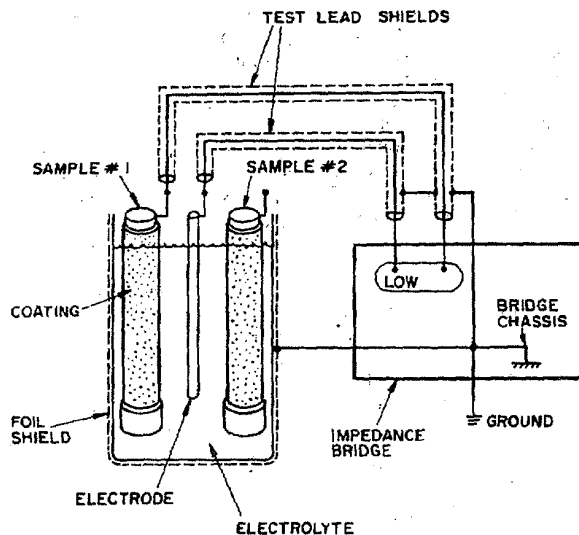


FIG. 2 Connecting the Test Cell to the Bridge

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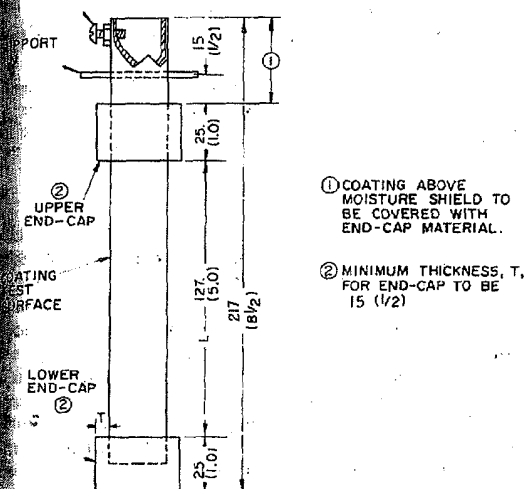


FIG. 3. Detail Drawing of Pipe Specimen

13 mm (0.5 in.) to 19 mm (0.75 in.) shall be maintained.

6.2.1 The end-cap material shall have a dielectric constant in the range from 2 to 6, bond well to the coating surface, and exhibit a low water-absorption rate. Several commercially available poly(vinyl chloride)-paraffin compounds,⁴ well suited for this purpose. They have a melting point in the 150 to 200°C (300 to 390°F) range, can be poured into molds around the pipe sample, and appear as resilient, malleable solids at room temperature.

NOTE 4—Using these materials, the end-caps can be applied to the required thickness by repeated dipping of the sample ends into a molten-wax bath, or through the use of light-weight, disposable molds of aluminum foil or paper formed around the pipe sample to allow the pressing of the caps directly to the surface of the coated pipe sample.

6.3 The end of the specimen which will protrude above the immersion line shall be provided with a suitable means of support and a separate wire connection for electrical purposes. The protruding end of the sample shall be waterproofed with a thin coating of end-cap material (see Fig. 1).

Preliminary Test Measurements

7.1 *Coating Thickness*—Measure and record the coating thickness by referring to Method G 12.

7.2 *Specimen Length*—Measure and record the length of exposed coating surface, between the end caps.

7.3 *End-Cap Capacitance*—Vertically suspend the test specimens and anode in the immersion cell, observing the clearances specified in 5.1.1 through 5.1.3. Fill the container with the electrolyte until it just covers the lower end cap.

* A material found suitable for this purpose is Boler Petroleum No. L-480-86 Blend, Boler Petroleum Co. 85 Old Eagle School Rd., Wayne, PA 19087.

Energize the impedance bridge and measure the series capacitance, C_c of the lower end cap.

7.4 *Initial Coating Capacitance*—Add additional electrolyte to the immersion cell until its level reaches the lower edge of the upper end cap. Immediately measure and record the initial series capacitance, C_0 , and dissipation factor, DF , of the specimen.

8. Procedure

8.1 Energize each specimen by connecting it to the negative side of the voltage source. Keep each sample energized and immersed to the lower edge of the upper end cap for the duration of the test period. Maintain the specified electrolyte level through regular additions of tap water.

8.2 Throughout the test make periodic measurements of the series capacitance and dissipation factor of the immersed specimens in the following manner:

8.2.1 Temporarily disconnect the test specimen from the voltage source. Verify that the electrolyte is at the proper level within the immersion cell. Connect the measuring bridge between the test specimen and stainless-steel anode. Energize the bridge and measure the series capacitance, C , and dissipation factor, DF , of the test specimen.

8.2.2 Using the observed value of series capacitance, C_s , calculate the apparent depth of water penetration by the method described in 9.2.

8.2.3 Repeat the measurements of series capacitance and specimen dissipation factor at periodic intervals throughout the duration of the test. The frequency of measurement will depend upon the rate of deterioration of the coating sample. Where the water penetration process is relatively rapid, daily readings of sample capacitance and dissipation factor will be required. Normally, readings made at weekly intervals will adequately define the penetration rate.

NOTE 5—Some coatings exhibit an initial rise in capacitance and dissipation factor, but reach a state of equilibrium in 6 to 9 months. An increase in capacitance and dissipation factor following this period of equilibrium, (or failure to reach equilibrium) indicates impending failure. A sample can be considered to have failed when the dissipation factor reaches a value of 1.0.

9. Calculations

9.1 *Dielectric Constant*—Calculate the dielectric constant, K_0 , for the coating film as follows:

$$K_0 = \frac{(C_0 - C_c) \ln [(2t_0 + d)/d]}{NL}$$

where:

K_0 = dielectric constant,

C_0 = initial coating capacitance, pF,

C_c = end-cap capacitance, pF,

d = outside pipe diameter, mm (in.),

t_0 = initial coating thickness, mm (in.),

L = exposed coating length, mm (in.), and

$N = 0.0556$ when d , t_0 , L , are in mm (1.413 when d , t_0 , L , are in in.).

9.2 *Apparent Depth of Penetration*—Calculate the depth of water penetration by applying the calculated value of K_0 from 9.1 and the measured value of equivalent series capacitance, C , to the following equations:

$$t_p = t_0 - t$$

$$t = (d/2)[(e^M) - 1]$$

where:

$M = NK_0 L / (C - C_0)$,

t = unpenetrated coating thickness, mm (in.),

C = series capacitance, pF, and

t_p = depth of penetration, mm (in.).

10. Report

10.1 The report shall include the following:

10.1.1 Complete identification of specimen, including:

10.1.1.1 Name and code number of the coating,

10.1.1.2 Size of pipe,

10.1.1.3 Source, production date, and production-run number,

10.1.1.4 Minimum, maximum, and average coating thickness,

10.1.1.5 Dates of starting and terminating test, and

10.1.1.6 Other information that may be pertinent,

10.1.2 Magnitude and polarity of d-c voltage applied to sample during the test period,

10.1.3 Length of the test period in days,

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10.1.4 Apparent depth of water penetration for the test period indicated,

10.1.5 Initial value of coating dissipation factor, and

10.1.6 Value of coating dissipation factor at the end of the test period.

NOTE 6—For the purpose of monitoring coating performance, plotted graphs of apparent depth of water penetration versus time in rectangular coordinates and coating dissipation factor versus time in semilogarithmic coordinates will render useful information over the duration of the test period.

11. Precision

11.1 Due to the range of coating formulations, thicknesses, densities, etc., found among commercially available coated pipe samples, the overall accuracy and reproducibility of the test results by these methods will tend to be poorer than those expected on insulating material of a more uniform nature.

11.2 The precision (reproducibility) of the dissipation factor and depth of penetration by these methods in general is considered to be such that when two tests are performed consecutively on the same specimen under identical conditions, the difference between the two results may normally be expected not to exceed $\pm 5\%$ of their mean.

Standard Test Method for Specific Bendability of Pipeline Coatings¹

This standard is issued under the fixed designation G 10; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

- 1.1 This method covers the specific determination of the effect of short-radius bends on coatings applied to 33.4-mm (1-in. nominal) diameter pipe.
- 1.2 *This standard may involve hazardous operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this practice to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*
- 1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

Referenced Documents

- 2.1 *ASTM Standards:*
- G 6 Test Method for Abrasion Resistance of Pipeline Coatings²
- G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

Summary of Method

3.1 The method consists of bending a 33.4-mm (1-in. nominal) diameter specimen of coated pipe around a mandrel to produce a range of short-radius bends. Coating failure in the form of cracking or loss of adhesion is detected through visual and electrical inspection of the bent specimen.

Significance and Use

4.1 This test will provide information on the ability of coatings applied to pipe to resist cracking, disbonding, or other mechanical damage as a result of bending. Because the test is applied to coated pipe from commercial production, the results can be directly used in the selection of similar materials for service. The test also has application as a quality control method when variations in coating application or material formulation will affect bending performance.

5. Apparatus

5.1 The bending apparatus shall be essentially as shown in Figs. 1 to 3 and shall include the following:

- 5.1.1 *Variable-Radius Mandrel*, constructed from four 19-mm (0.75-in.) thick pieces of plywood, bolted together and conforming to the geometric shape shown in Fig. 1. The

geometric construction is accomplished by laying out along the outer edge of the mandrel a series of seven consecutive arcs at decreasing radii of 610, 530, 460, 380, 300, and 230 mm (24, 21, 18, 15, 12, and 9 in.). The first five arcs shall be carried through a 45-deg angle to the next point of tangency. A 45-deg V-notch shall be cut into the edge of the mandrel for seating the pipe specimen. Holes shall be drilled at appropriate locations in the mandrel face for positioning the lever arm and fastening pipe clamps.

5.1.2 *Lever Arm-Roller Assembly*—A 1.83-m (72-in.) lever arm with nylon roller supplies the mechanical advantage necessary to bend the pipe specimen. The lever arm shall contain a series of holes which are used to maintain proper clearance between the roller and pipe sample during the bending operation.

5.2 *Thickness Gage*—Measurements of coating thickness will be required for this test. This shall be done in accordance with Method G 12.

5.3 *Holiday Detector*—A low-voltage d-c holiday detector of the wet-sponge type, specified in Method G 6, shall be used to locate breaks in the coating film.

6. Test Specimens

6.1 The test specimen should be 2.5 m (100 in.) in length. It should be representative of production-coated pipe and be free of obvious coating flaws or defects. Coating specimens shall be applied to 33.4-mm (1-in. nominal) diameter pipe.

7. Conditioning

7.1 The specimen shall be exposed to room temperature for a sufficient time to ensure thermal equilibrium in the pipe and coating. A temperature in the range from 20 to 30°C (68 to 85°F) shall be considered room temperature.

8. Procedure

8.1 Perform the test at a room temperature of 21 to 25°C (70 to 77°F).

8.2 Measure the applied coating thickness of each specimen in accordance with Test Method G 12.

8.3 Place the conditioned test specimen into the V-notch. Secure the pipe in place with clamp No. 1 and tighten the thumb screw. Remove the threaded handle from clamp No. 1 to allow for clearance of the lever arm. With the steel pivot pin at hole A of the lever arm (see Fig. 3), insert the pin into the socket on the bed of the bending jig. Place the nylon roller at position C on the lever arm. Apply the roller to the pipe specimen, and with a constant, even force with the lever handle, bend the pipe around the mandrel until the 460-mm (18-in.) radius bend has been made. Insert and secure pipe clamp No. 2. Continue bending the pipe until the 300-mm (12-in.) radius has been reached. Insert and secure pipe

¹ This method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1983. Published August 1983. Originally published as G 10 - 69T. Last previous edition G 10 - 77.

² *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

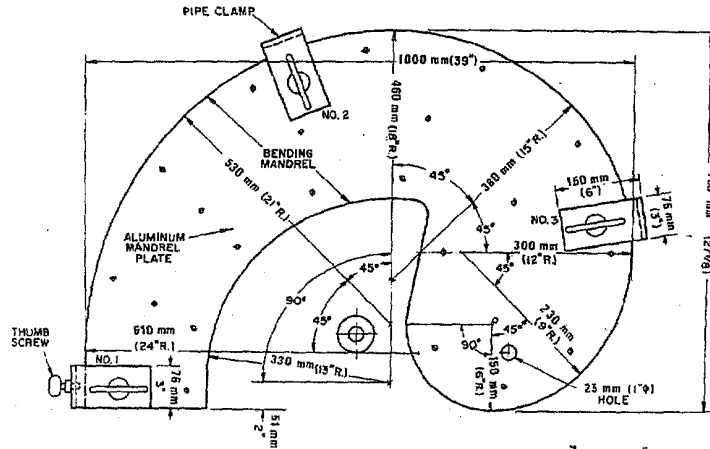


FIG. 1 Variable-Radius Mandrel

clamp No. 3. Reposition the lever arm by moving the pivot pin to the 25-mm (1-in.) diameter hole in the mandrel surface. Move the nylon roller to position B on the lever arm. Apply the roller to the pipe and complete the bending operation by forming the specimen through the 230-mm (9-in.) radius.

8.4 Examine the bent specimen with a wet-sponge holiday detector to determine at what point, if any, cracking of the coating film occurred. Note the number, size, location, and type of cracks present. Examine the pipe specimen for any loss of coating bond caused by the bending operation.

NOTE 1—The possibility of some coatings developing stress-induced cracks in the period following the bending operation should not be overlooked. If this is a consideration, the bent specimen should be retained for a 24-h post-bent inspection.

9. Report

9.1 The initial test report shall include the following:

- 9.1.1 Complete identification of the coated pipe tested, including: name and type of coating, average coating thickness, minimum coating thickness, maximum coating thickness, manufacturer's lot number, and date of manufacture.
- 9.1.2 Temperature of the pipe specimen as tested,
- 9.1.3 Radius at which cracking first-occurred,
- 9.1.4 Description of the type of cracking,
- 9.1.5 Location and extent of any loss in pipe-to-coating bond,
- 9.1.6 Number of specimens tested,
- 9.1.7 Any peculiar characteristics of the specimen noted during the test or after immediate removal from the test apparatus, and
- 9.1.8 Post-bend retention time.

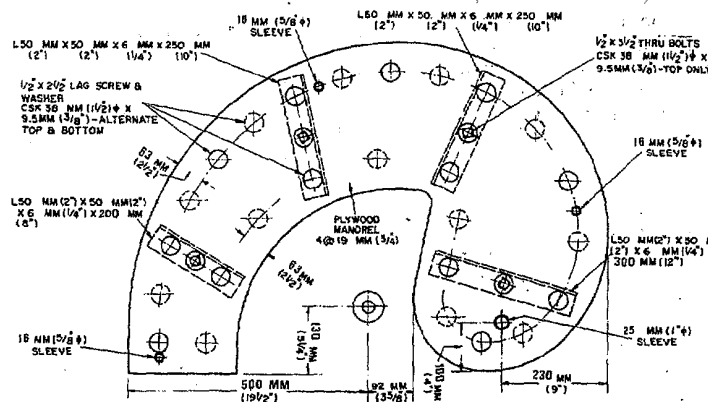


FIG. 2 Details of Mandrel Assembly and Support Brackets

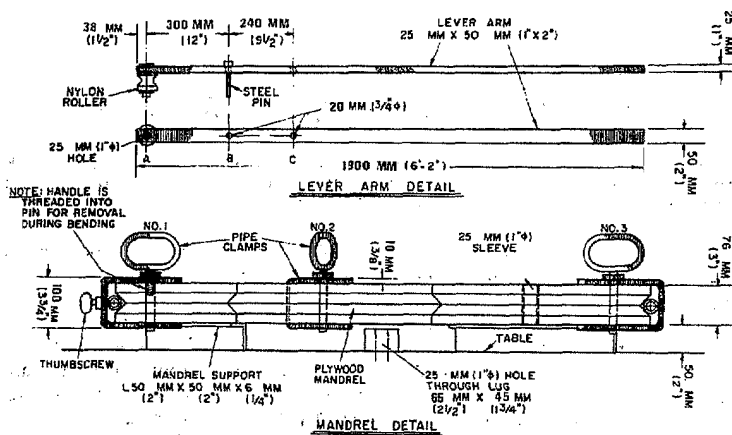


FIG. 3: Details of Lever Arm and Pipe Clamps

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Standard Test Method for Effects of Outdoor Weathering on Pipeline Coatings¹

This standard is issued under the fixed designation G 11; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is intended to define conditions for the exposure of coated pipe to weather.

1.2 This test method specifies qualifications for the samples, procedure to be followed in exposure to weather and procedure for evaluating effects of exposure including visual examination and other tests.

1.3 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 The values stated in SI units to three significant decimals are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

- G 8 Test Methods for Cathodic Disbonding of Pipeline Coatings²
- G 10 Test Method for Specific Bendability of Pipeline Coatings²
- G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²
- G 13 Test Method for Impact Resistance of Pipeline Coatings (Limestone Drop Test)²
- G 62 Test Methods for Holiday Detection in Pipeline Coatings³
- G 70 Test Method for Ring Bendability of Pipeline Coatings (Squeeze Test)³

3. Significance and Use

3.1 Since coated pipe may be stored outdoors for long periods before burial, weathering tests of the type described in this test method are needed to evaluate the stability of these coatings stored outdoors. The results obtained should be treated only as indicating the general effect of weathering. Exposure conditions vary greatly from year to year, from one part of a year to another, and from locality to locality. The results of short-term exposure tests in the north are more meaningful if exposure is started in the summer followed by a winter season. In southern areas where climatic conditions are more uniform throughout the year, the time of year when short-term exposure is started is less critical. In all localities,

the longer the exposure period, the more reliable are the results obtained.

4. Summary of Test Method

4.1 The effects of outdoor weathering on pipeline coatings after 6, 12, and 24 months' exposure are determined visually and by electrical means by comparing exposed samples of coated pipe with unexposed samples of coated pipe before and after impact and bending tests. At the beginning of the test the starting samples are qualified by a cathodic disbonding test.

5. Location of Test Sites

5.1 Weathering racks shall be located in cleared areas representative of local outdoor weather conditions.

6. Apparatus

6.1 *Racks*—The horizontal racks shall be any suitable pipe storage racks of sufficient height to prevent any undesirable effects of vegetation growth during the period of exposure. Racks may be constructed from a variety of materials, but pipe specimens must rest on a nonconductive surface. An example of a suitable rack is illustrated in Fig. 1.

7. Sampling

7.1 Each starting sample shall consist of coated 26.7-mm (1-in. nominal) diameter steel pipe from a production lot having a minimum length of 4.4 m (14.4 ft). The sample shall be from a lot produced under conditions capable of being duplicated on a production scale.

NOTE 2—Pipe having a nominal diameter of 33.4-mm (1-in. nominal) can be used and is the largest diameter that can be evaluated in accordance with Test Method G 10. Larger diameter pipes can be evaluated in accordance with Test Method G 70.

7.2 Each starting sample shall have a piece 600-mm (24-in.) long cut from one end by sawing for use in qualification testing (Section 8) and designated the "qualification sample." The remainder of each starting sample shall be designated the "sample."

7.3 The total number of samples for each coating in test shall be four, divided as follows:

- 7.3.1 One to be removed after 6 months exposure,
- 7.3.2 One to be removed after 12 months exposure,
- 7.3.3 One to be removed after 24 months exposure, and
- 7.3.4 One to be used for determining original control values, which should be obtained as soon as possible on receipt of the pipe.

8. Procedure for Qualification Testing

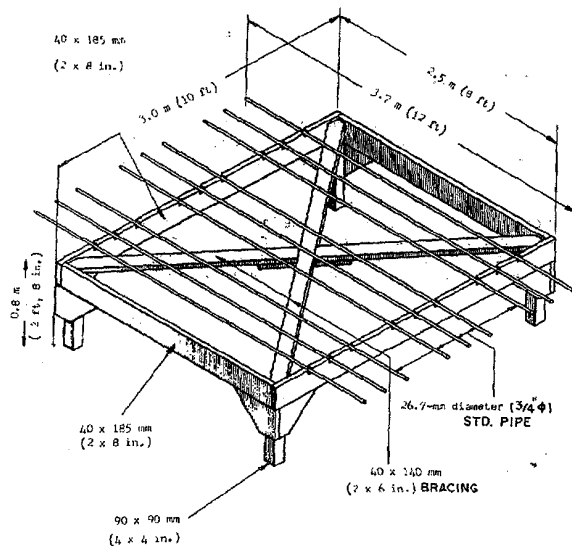
8.1 Test each qualification sample in accordance with Test Methods G 8, Method B.

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1988. Published July 1988. Originally published as G 11 - 69 T. Last previous edition G 11 - 83.

² Annual Book of ASTM Standards, Vols 06.01 and 14.02.

³ Annual Book of ASTM Standards, Vol 14.02.



All lumber is to be pretreated and painted.

FIG. 1 Construction Details of Outdoor Weathering Rack

Begin qualification testing at the same approximate sample testing.

If the results of the qualification testing are within the limits given in Test Methods G 8, consider the test to be reasonably equivalent and proceed with testing.

If the results of the qualification testing are not within the limits given in Test Methods G 8, consider the test to be unequivalent and discontinue exposure testing.

Procedure for Sample Testing

Identify the test samples with a letter, number, or symbol for ready identification after exposure. The marking should not interfere with either the exposure or the testing.

Note and record the appearance of the sample.

Measure and record coating thickness in accordance with Test Method G 12.

Check coating for holidays with the appropriate device as described in Test Methods G 62.

Make two 80-mm (3-in.) long scribes through the coating to bare metal. Begin one scribe at a point approximately 230-mm (9-in.) from one end of the pipe and continue it for 80-mm along the length of the pipe; begin the second scribe at a point approximately 230-mm from the other end of the pipe and continue it for 80-mm along the length of the pipe. One scribe should be on the top of the pipe and the other on the bottom.

Mount the samples on the rack as shown in Fig. 1 with the samples facing skyward and one facing toward the ground. Mount the three samples together so that like samples are mounted alongside one another.

After exposure, again note and record the appearance of the sample and check for holidays in the manner followed

prior to exposure. Next examine the sample visually for cracking, checking, blistering, corrosion, undercutting from the intentional scribe, and any other form of impairment of the coating.

9.8 Finally, cut a 300-mm (12-in.) specimen from one end of the sample, where the scribe is facing downward, and subject to an impact test in accordance with Test Method G 13. In the impact test, position the specimen so that the scribe faces downward.

9.9 Subject the remaining 2.5 m (8.2 ft) to a bend test in accordance with Test Method G 10. In the bend test, position the skyward face of the sample as the outside of the bend.

9.10 Follow the procedure in 9.7, 9.8, and 9.9 on samples removed after each of the three exposure periods and compare results with those obtained on the control sample in 7.3.

10. Report

10.1 The report shall include the following:

10.1.1 Results of qualification tests,

10.1.2 Sample number and description of coating system including: surface preparation, method of application, and coating thickness,

10.1.3 Appearance of sample and presence or absence of holidays prior to exposure,

10.1.4 Appearance of sample, presence, or absence of holidays after exposure, and duration of exposure,

10.1.5 Visual evidence of coating failure as noted by 9.7,

10.1.6 Results of impact and bend tests after exposure as compared with those for the control sample, and

10.1.7 Type of environment and exposure.

11. Precision

11.1 The precision requirements for qualification testing

in Section 8.3 of this test method and the precision requirements given in Test Method G 12 shall apply. Variations in samples and qualification samples shall be within respective precision limits for this test method to be valid.

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Scope

1.1 This test method is applicable to the measurement of the thickness of nonmagnetic coatings on ferrous and nonferrous metal surfaces. It does not apply to the measurement of the thickness of magnetic coatings on ferrous metal surfaces.

1.2 This test method requires the use of magnetic induction equipment and equipment for the measurement of the thickness of the coating.

1.3 This test method is applicable to the measurement of the thickness of coatings in decimal units.

Summary

2.1 This test method is a nondestructive method for the measurement of the thickness of nonmagnetic coatings on ferrous and nonferrous metal surfaces.

Significance and Use

3.1 Magnetic induction methods are used for the measurement of the thickness of nonmagnetic coatings on ferrous and nonferrous metal surfaces. Adequate function of the magnetic induction method is required for the measurement of the thickness of coatings in decimal units.

3.2 The magnetic induction method is used for the measurement of the thickness of coatings in decimal units.

Apparatus

4.1 The magnetic induction gage is used for the measurement of the thickness of nonmagnetic coatings on ferrous and nonferrous metal surfaces. The magnetic induction gage is used for the measurement of the thickness of coatings in decimal units.

4.2 The magnetic induction gage is used for the measurement of the thickness of coatings in decimal units.

¹ This test method is a nondestructive method for the measurement of the thickness of nonmagnetic coatings on ferrous and nonferrous metal surfaces. It does not apply to the measurement of the thickness of magnetic coatings on ferrous metal surfaces.



Standard Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel¹

This standard is issued under the fixed designation G 12; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes the nondestructive measurement of the thickness of a dry, nonmagnetic coating applied to the external surface of steel pipe. The method is recommended for coating thicknesses up to 6 mm (0.240 in.) and for any diameter pipe, but not smaller than 10 mm (0.5 in.). It does not apply to excessively soft films.

1.2 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

2. Summary of Test Method

2.1 The coating thickness is determined by an instrument that measures thickness by nondestructive techniques, based on variations in magnetic flux or magnetic attraction between its detection unit and the magnetic base material.

3. Significance and Use

3.1 Measurements of film thickness are an essential part of most ASTM test methods related to coatings on steel pipe. Adequate thickness is important for a coating to fulfill its function of preventing or mitigating corrosion of steel pipelines.

3.2 The accuracy of the thickness measurements may be influenced by the deformability of the coating. This test method is not applicable to coatings that would be readily deformable under the force exerted by the probe of the measuring instrument.

4. Apparatus

4.1 The apparatus shall be a nondestructive-type thickness gage capable of being standardized over its range of intended use. It shall be designed so that variations in magnetic flux or magnetic attraction between its detection unit and the steel base can be calibrated to indicate the thickness of the coating material.

4.2 It shall be suitable for measuring thicknesses of dry, nonmagnetic coatings on either a flat or a circular base.

5. Standardization of Instruments

5.1 The instrument shall be standardized in accordance with the manufacturer's instructions before use by employing suitable thickness standards. The standardization shall be checked at frequent intervals during use.

5.2 Nonmagnetic standards of uniform thickness are available in either of two types, foil or coated substrate, as supplied or recommended by the manufacturer of the instrument.

5.2.1 *Foils (Shims)*—The thickness of the foil should be as close as possible to the expected thickness of the coating to be measured. The foil should be placed on a smooth, clean, low-carbon, steel plate, making certain that there is intimate contact between the foil and the substrate. It is recommended that single foils of proper thickness be used and that foils be replaced frequently, since they are subject to indentation. Foils are advantageous for standardizing on curved surfaces and are often more readily available than coated standards.

NOTE 1—For some instruments there is an effective depth of penetration of the field created by the instrument probe. This is the critical depth of thickness at which the instrument will no longer be affected by increases of substrate thickness. Since it depends on the instrument and substrate, it should be determined experimentally.

5.2.2 *Coated Substrate Standards*²—These standards consist of nonmagnetic coatings of known thickness permanently bonded to a low-carbon steel (1010 grade) substrate, 8 mm (0.30 in.) thick. The thickness standard should be as close as possible to the expected thickness of the coating to be measured.

NOTE 2—If the curvature of the coating to be measured is sufficient to preclude standardization on a flat surface, the standardization should be done with a standard foil on a substrate with the same curvature as the coating to be measured.

6. Procedure

6.1 Operate each instrument in accordance with the manufacturer's instructions.

6.2 Take a minimum of twelve readings per piece of pipe as follows: Take three equally spaced readings along the top surface; rotate the pipe 90° three times, taking three equally

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G3.06 on Durability of Pipeline Coatings and Linings.

Current edition approved June 24, 1983. Published August 1983. Originally published as G 12 - 69 T. Last previous edition G 12 - 77.

² Coated substrate standards may be obtained from the U. S. Department of Commerce, National Bureau of Standards, Standard Materials Unit, Washington, DC 20234.

spaced readings after each rotation. Record the average, minimum, and maximum of the twelve readings as the coating thickness.

NOTE 3—Instruments with two contact poles shall be positioned on the coating surface with the poles in the same plane.

NOTE 4—The anchor pattern of the steel pipe substrate must be considered for its effect on the accuracy of the measurements if the anchor pattern is greater than 25 % of the coating thickness.

NOTE 5—Foreign materials such as dirt and grease shall be removed by suitable cleaning without removing any coating materials.

NOTE 6—Measuring instruments and methods may depend on the technique of the operator. For example, the pressure applied to a probe and the rate of applying a balancing force to a magnet will vary from one individual to another. Often such differences can be reduced or eliminated either by having the gage calibrated by the same operator who will make the measurement or by using constant-pressure probes. Probes must be positioned perpendicular to the surface of the specimen at the point of measurement. Some magnetic instruments of the magnetic-attraction type require the pole piece to be in a vertical position. If a magnetic instrument is to be used in a horizontal or upside-down position, its calibration for that position should be verified.

NOTE 7—Instruments using magnetic attraction are sensitive to the speed with which the magnet is removed from the specimen. The magnet must be removed so that thickness measurements are not affected by speed of removal.

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7. Report

7.1 The report shall include the following:

7.1.1 Complete identification of the specimen including name and code number of the coating; pipe diameter; source; production data, including surface preparation of the substrate and depth of anchor pattern, if known; and production run number.

7.1.2 Name and type of instrument used, and method of standardization, and

7.1.3 Number of thickness measurements taken, and the average, minimum, and maximum value of the measurements.

8. Precision

8.1 The repeatability and reproducibility of thickness measurements will depend on the instrument design, the operator, and the particular measuring application.

8.1.1 *Repeatability*—When the same instrument is used by the same operator, duplicate measurements on the same sample should agree with each other within $\pm 5\%$.

8.1.2 *Reproducibility*—Separate operators using different instruments on a known thickness standard should obtain average results agreeing with each other within 10 % of the thickness standard.

Standard Test Method for Impact Resistance of Pipeline Coatings (Limestone Drop Test)¹

This standard is issued under the fixed designation G 13; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method covers the determination of the relative resistance of pipeline coatings to impact by observing the effects of falling stones on coated pipe specimens.

1.2 *This standard may involve hazardous operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units are to be regarded as the standard.

Referenced Documents

2.1 ASTM Standards:

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

G 62 Test Methods for Holiday Detection in Pipeline Coatings³

2.2 AASHTO Standard:

M80-51 (No. 67) Specification for Coarse Aggregate for Portland Cement Concrete

3. Summary of Test Method

3.1 The impact resistance of pipeline coatings is determined by dropping weighed amounts of a specified type of limestone through a chute onto a coated pipe specimen. Results are reported as the number of drops required to pierce through the coating to bare metal, as determined visually or electrically.

4. Significance and Use

4.1 This test is intended to simulate the effects of backfilling after pipe has been placed in the trench. The backfill is often rocky soil and, if it is unscreened and the coated pipe is unshielded by sand or other protective padding, the falling rocks may seriously damage the coating.

5. Apparatus

5.1 The impact apparatus shall be essentially as shown in Fig. 1 and shall include the following:

5.1.1 *Box with Chute*, providing a means of dropping

stones from a height of 1830 mm (6.0 ft) measured to the top of a piece of coated pipe under test. Construction details are shown in Fig. 2.

NOTE 1—The box and chute described in Fig. 2 are designed for testing coatings on 100-mm (4-in.) and 150-mm (6-in.) pipe specimens. Smaller diameter pipe may be used by inserting wood V-blocks in the trough in the box below the chute.

5.1.2 *Stones*, hard, coarse, limestone aggregate, conforming to AASHTO Designation: M80-51 (No. 67) which is taken from American Association of State Highway and Transportation Officials "Standard Specifications for Coarse Aggregate for Portland Cement Concrete."

NOTE 2—These specifications cover the quality and size of coarse aggregate. No. 67 designates a 19-mm (¾-in.) to No. 4 size stone with the following sieve analysis:

Square Opening, mm (in.)	Weight % Passing
25 (1)	100
19 (¾)	95 to 100
10 (⅝)	20 to 55
No. 4	0 to 10

5.1.3 *Bucket*—Any suitable bucket that will hold 16 kg (35 lb) of stones.

5.1.4 *Holiday Detectors*—Two types are needed as described in Test Method G 62.

NOTE 3—A holiday is defined as small faults or pinholes that permit current drainage through protective coatings on steel pipe.

5.1.5 *Thickness Gages*—Any instruments suitable for use with Test Method G 12.

6. Test Specimens

6.1 Test specimens shall be 600 mm (24 in.) long and shall be cut from a representative piece of coated pipe. Only holiday-free specimens shall be used in the test.

7. Procedure

7.1 Perform the test at a room temperature of 21 to 25°C (70 to 77°F).

7.2 The procedure consists of dropping up to 10 buckets of stones on coated specimens. The number of buckets of stones required represents the resistance of the coating to this type of impact.

7.3 Place the test specimen in the trough below the chute. Insert the retaining board in the top part of the chute and hold it in place across the chute opening with a suitable metal or wood clip. Weigh 16 ± 0.2 kg (35 ± 0.5 lb) of stones into the bucket. Lift the bucket to the top of the chute. Empty the stones into the trough formed by the hopper walls and the retaining board so that the stones are distributed evenly in the trough. Detach the holding clip from the

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved April 28, 1989. Published June 1989. Originally published as G 13 - 69 T. Last previous edition G 13 - 85.

² Annual Book of ASTM Standards, Vols 06.01 and 14.02.

³ Annual Book of ASTM Standards, Vol 14.02.

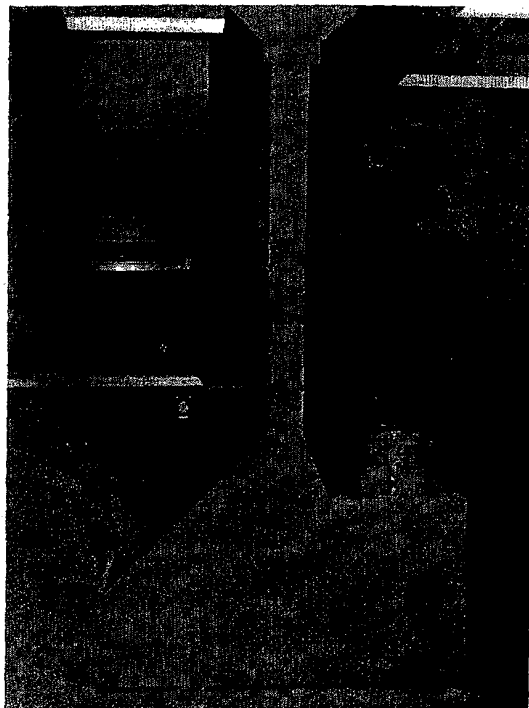


FIG. 1 Box, Chute, and Bucket

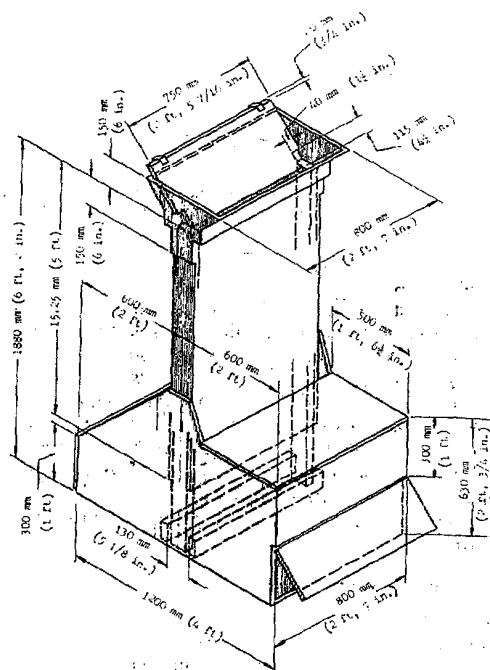
retaining board and tilt the board forward to release the stones. Experience with this procedure has indicated that the stones are so constricted within the narrow confines of the chute that they fall in a uniform manner.

7.4 After each drop, remove the specimen. Examine the specimen visually for breaks in the coating. If there are no visual breaks, check with the holiday detector appropriate for the coating thickness for holidays in the coating. If there is no visual or electrical evidence of holidays, repeat the test until the first holiday is detected and record the number of drops and the number of holidays detected. In repeating the test, the specimen should be replaced in the trough so that the same portion of the coated surface is always subjected to the falling stones. After the same stones have been used five times, they should be replaced.

7.5 If no failure has occurred after 10 buckets, terminate the test.

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NOTE—Framing lumber 40 by 90 mm (2 by 4 in.); all other material 20 mm (3/4 in.).

FIG. 2 Construction Details of Box and Chute

NOTE 4—As indicated in 7.4, the same charge of stones may be used five times. Experiments in which the same charges of stones were dropped more than five times have shown relatively little attrition of the stones.

8. Report

8.1 The report shall include the following:

8.1.1 Complete identification of the specimen including: name and code number of the coating, pipe diameter, coating thickness, source, production data, and production-run number,

8.1.2 Approximate pipe temperature at the beginning and at the end of the test, and

8.1.3 Number of buckets of stone to produce the first holiday and the number of holidays produced during this drop.

NOTE 5—If there is a large number of holidays after a given number of drops, the report should state this by the term "too numerous to count."



Standard Test Method for Impact Resistance of Pipeline Coatings (Falling Weight Test)¹

This standard is issued under the fixed designation G 14; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the energy required to rupture coatings applied to pipe under specified conditions of impact from a falling weight.

1.2 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

G 62 Test Methods for Holiday Detection in Pipeline Coatings³

2.2 SAE Standard:

Grade 52100 Steel⁴

3. Summary of Test Method

3.1 This test method uses a falling fixed weight having a specified diameter impact surface, *tup*, which is restrained vertically and dropped from varying heights to produce impact energies over the required range. Electrical inspection is used to detect resultant breaks in the coating. Impact resistance is determined as the amount of energy required to cause penetration of the coating film.

4. Significance and Use

4.1 The ability of a pipe coating to resist mechanical damage during shipping, handling, and installation will depend upon its impact resistance. This test method provides a systematic means for screening coating materials with regard to this property.

5. Apparatus

5.1 This test method can be successfully used with impact apparatus conforming to the following specifications:

5.1.1 *Tup*—The tup shall be made up from a tup body and a tup nose having a combined, fixed weight of 1.361 kg (3.00 lb) and shall be used over a drop range of 0.61 to 1.22 m (2 to 4 ft). With most coatings, a 1.361-kg (3.00-lb) tup dropped through a distance of 914 mm (3 ft) yields suitable results. The tup nose shall have a 15.875-mm ($\frac{5}{8}$ -in.) hemispherical head.

NOTE 1—Frequent replacement of the tup nose can be avoided if it is cut from steel capable of being hardened to a hardness of Rockwell C/45 while retaining an impact toughness of at least 15 ft-lb (20.34 J). Ball bearings conforming to SAE Grade 52100 have also been found suitable for this purpose.

5.1.2 *Drop Tube*—A tube 1.52 m (5 ft) long shall be used to contain the tup and guide it during free fall. The drop tube shall be constructed of steel, aluminum, or any other suitably rigid material and internally sized to provide a minimum of friction to the falling tup. A scale shall be attached for measuring the height of drop to the nearest 2.54 mm (0.10 in.).

5.1.3 *Specimen Holder*—The base plate of the apparatus shall include a device for positioning and holding the pipe specimen on line with the axis of the vertical drop tube.

NOTE 2—An arrangement using a V-notch vise made of metal with spring clamp is recommended for this purpose. Glancing blows, caused by an out-of-plumb condition between drop tube and pipe sample, will cause erratic test results.

5.1.4 *Apparatus Support*—Both the apparatus and sample shall be firmly supported and secured to a rigid base to optimize energy transfer from the tup to the specimen.

5.2 A design for the test apparatus appears in Figs. X1.1 through X1.3 of Appendix X1.

5.3 *Thickness Gage*—Measurements of coating thickness will be required for this test, and shall be done in accordance with Method G 12.

5.4 *Holiday Detector*—A suitable detector as specified in Test Methods G 62 shall be used to locate breaks in the coating film.

6. Test Specimen

6.1 The test specimen shall be a 406.4 mm (16 in.) long piece of Schedule 40, 60.325 mm (2.375 in.) outside diameter coated pipe prepared with its surface preparation and coating procedures equivalent to that of production coated pipe.

6.2 Seven specimens shall be required for the test.

7. Conditioning

7.1 The specimen shall be exposed to a room temperature of 21 to 25°C (70 to 77°F) for a period of 24 h before beginning the test.

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1988. Published July 1988. Originally published as G 14 - 69 T. Last previous edition G 14 - 83.

² *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

8. Preliminary Measurements

8.1 Measure the applied coating thickness of each specimen in accordance with Test Method G 12.

8.2 Place test specimen in sample holder and lightly place tup on surface of the coating. Adjust either the drop tube or the attached scale so that the wing bolt (lifting pin) is at the zero mark of the scale.

8.3 Make a preliminary set of impact readings to determine the approximate starting point for the test. This shall be done by striking the first specimen from a height sufficient to cause failure of the coating film. Consider any penetration a failure if it is detectable with a suitable Holiday Detector as specified in Test Methods G 62.

8.3.1 Reduce the height by 50 % and make a second exploratory drop at a fresh area on the pipe surface. Continue testing in this manner, with the corresponding reduction in height between drops, until the coating fails to break.

NOTE 3—Choose test locations at the specimen surface in a random manner and keep at least a 76.2-mm (3-in.) distance between adjacent points of impact and within 38 mm (1½ in.) from the ends. Choosing test points in any regular pattern will bias the experiment and introduce error into the test results.

8.3.2 Repeat the test at the height immediately preceding the occurrence of the nonfailure to determine if an approximate level for the mean impact strength has been bracketed. Two successive reversals of coating performance between failure and nonfailure will give sufficient indication that the point has been reached.

9. Procedure

9.1 Perform the test at a room temperature of 21 to 25°C (70 to 77°F).

9.2 Begin testing from the approximate height determined in 8.2 and corresponding to the point at which the first nonfailure was registered. Maintain a fixed increment between adjacent testing heights.

9.3 Use a suitable detector, as specified in Test Method G 6, to determine penetration or lack thereof of the coating after each individual impact.

9.4 If the coating film is penetrated on the initial drop, make the next test at the next lower height increment. If the first specimen does not fail, make the second test at the next higher increment.

9.5 In a similar manner, determine the height of fall by the performance of the coating on each preceding drop. Maintain a constant height increment between readings. Continue to apply this "up-and-down" method⁴ until 20 successive impact readings have been made.

⁴ This system of testing, nomenclature, and calculation is described by Dixon, W. J. and Mood, A. M., "A Method for Obtaining and Analyzing Sensitivity Data," *Journal Am. Statistical Assn.*, JSTNA, Vol 43, March, 1948, p. 109.

10. Calculation

10.1 Calculate the mean value of impact strength, m , in g/cm (or in./lb) as follows:

$$m = \left[h_0 + d \left(\frac{A}{N} \pm \frac{1}{2} \right) \right] \times W$$

where:

h_0 = minimum height at which the less frequent event occurs, cm (or in.),

d = increment in height of drop, cm (or in.),

A = sum of the frequency of occurrence at each height increment times the number of increments above the h_0 value for each observation in the N total,

N = total number of the less frequent event (coating failures or nonfailures), and

W = tup weight, g (or lb).

NOTE 4—The minus sign is used when the calculation of the mean is based on the total number of coating failures and the plus sign when it is based on the nonfailures.

10.2 Calculate the sample standard deviation, s , in gram-centimetres (or inch-pounds) as follows:

$$S = 1.620 dW \left(\frac{(NB - A^2)}{N^2} + C \right)$$

where:

C = 0.737 when d is in cm, or 0.029 when d is in in.,

d , N , A , and W are as defined in 10.1, and

B = sum of the frequency of occurrence at each height increment times the square of the number of increments above the h_0 value for each observation in the N total.

NOTE 5—Adequate definition of coating impact strength will result only if the height increment, d , has been properly chosen. When testing polymeric films in the range from 0.254 to 1.016 mm (0.010 to 0.040 in.), height increments of from 5.1 to 12.7 mm (0.2 to 0.5 in.) have been found suitable with the 1.361-kg (3.0-lb) tup. Larger increments may be necessary for thicker materials. If after making the calculations of 10.1 and 10.2, the ratio of the height increment to the standard deviation (d/s) is less than 0.20, the test should be repeated using a larger value for the increment d . This procedure will result in an improved estimate of the coating impact strength.

10.3 An illustration of the use of these equations appears in Appendix X2.

11. Report

11.1 The report shall include the following:

11.1.1 Complete identification of the specimen including:

11.1.1.1 Name and code number of the coating,

11.1.1.2 Size of pipe,

11.1.1.3 Source, production date, and production-run number,

11.1.1.4 Minimum, maximum, and average coating thickness,

11.1.1.5 Date of test, and

11.1.1.6 Other information that may be pertinent,

11.1.2 Average impact strength in g/cm (or in./lb),

11.1.3 Sample standard deviation in g/cm (or in./lb).

APPENDIXES

(Nonmandatory Information)

X1. SUGGESTED DESIGN FOR TEST APPARATUS

X1.1 A design for the impact test apparatus is shown in Figs. X1.1 to X1.3.

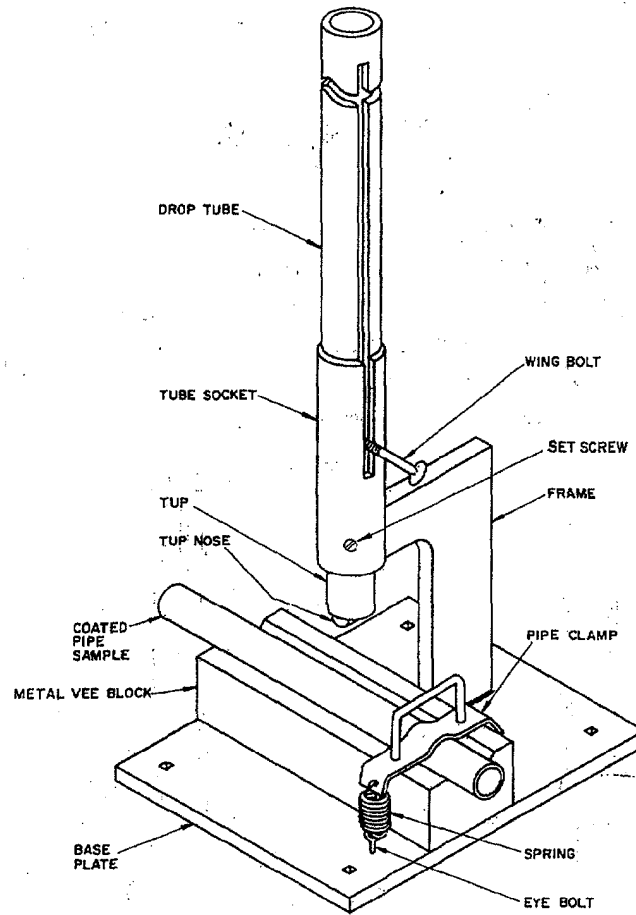


FIG. X1.1 Test Apparatus Assembly

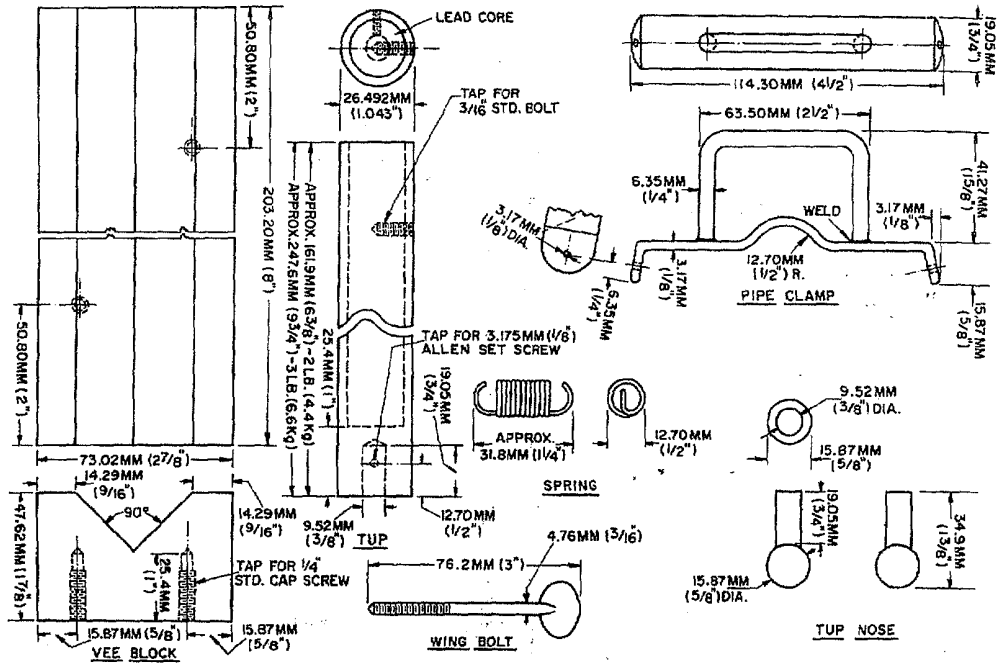


FIG. X1.2 Detailed Design of Test Apparatus

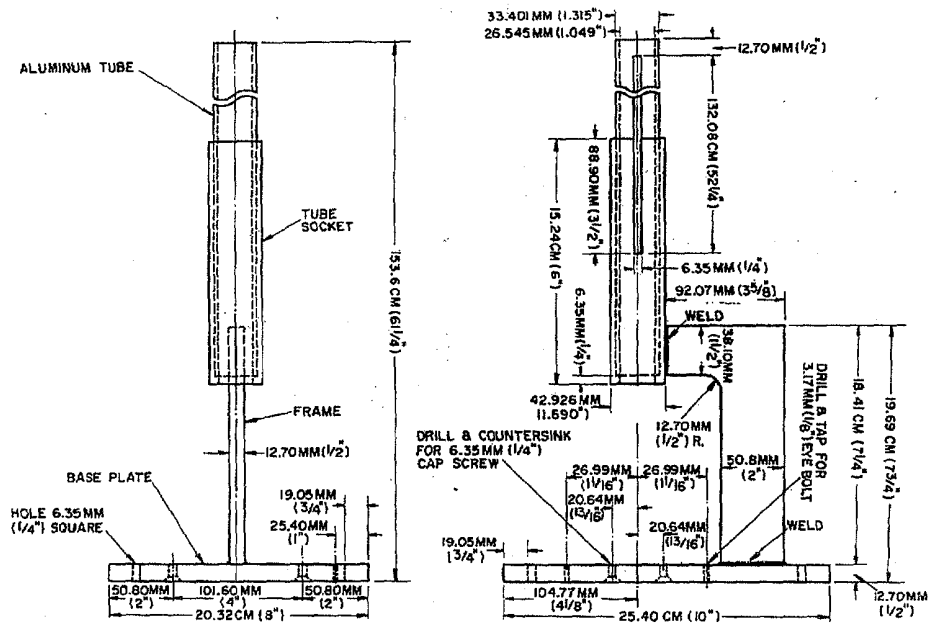


FIG. X1.3 Detailed Design of Test Apparatus

X2. SAMPLE CALCULATIONS

X2.1 Test results for 20 drops with a 1.361-kg (3-lb) tup are given in Table X2.1.

TABLE X2.1 Test Results

Test No.	Height of Drop, in.	Failed	Test No.	Height of Drop, in.	Failed
1	14.1	yes	11	13.5	no
2	13.8	no	12	13.8	no
3	14.1	no	13	14.1	yes
4	14.4	yes	14	13.8	yes
5	14.1	yes	15	13.5	no
6	13.8	no	16	13.8	yes
7	14.1	no	17	13.5	yes
8	14.4	yes	18	13.2	no
9	14.1	yes	19	13.5	no
10	13.8	yes	20	13.8	yes

Height increment = 0.3 in.

Failures = 11

Nonfailures = 9

Nonfailures at 13.2 in. (h_0) = 1; at 13.5 in. = 3; at 13.8 in. = 3; at 14.1 in. = 2 $A = (0 \times 1) + (1 \times 3) + (2 \times 3) + (3 \times 2) = 15$ $B = ((0)^2 \times 1) + ((1)^2 \times 3) + ((2)^2 \times 3) + ((3)^2 \times 2) = 33$

$$m = \left[13.2 + 0.3 \left(\frac{15}{9} + \frac{1}{2} \right) \right] 3.0 = 41.55 \text{ in.} \cdot \text{lb}$$

mean impact resistance = 41.55 in.·lb

$$S = 1.620 \times 0.29 \times 3.0 \left(\frac{9 \times 33 - (15)^2}{(9)^2} \right) = 1.338 \text{ in.} \cdot \text{lbs.}$$

Sample standard deviation = 1.338 in.·lb

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Penetration Resistance of Pipeline Coatings (Blunt Rod)¹

This standard is issued under the fixed designation G 17; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This accelerated test method is used to determine the relative resistance of steel pipeline coatings to penetration or deformation by a blunt rod under a specified load. The test method is intended to apply to the testing of all types of nonmetallic pipeline coatings subjected to various temperatures.

1.2 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

2. Summary of Test Method

2.1 The depth or rate of penetration or deformation that is caused by a weighted blunt rod to a coating system applied to steel pipe is measured over a period of time with a micrometer depth gage. Three consecutive identical readings taken at specified intervals conclude the test.

3. Significance and Use

3.1 Since pipeline coatings are subjected to concentrated pressures exerted by the weight of the pipe and the backfill, this test is intended primarily for determining the resistance of the coating to penetration or deformation under controlled conditions.

3.1.1 Deformation of a compressible coating does not signify a potential weakness in underground burial service, if the coating is not penetrated. Deformation cannot be compared to penetration into and through a coating.

4. Apparatus

4.1 Test apparatus shall consist of a dead weight tester that can press the flat tip of a rod against the coated pipe. The flat-tipped end of the rod contacting the coating shall have a diameter of $6.350 \text{ mm} \pm 0.0254 \text{ mm}$ ($0.250 \text{ in.} \pm 0.001 \text{ in.}$) and together with supplementary weight and any other weight-contributing parts shall have a total weight of 4.453 kg (9.817 lb), resulting in a unit pressure of 14.060

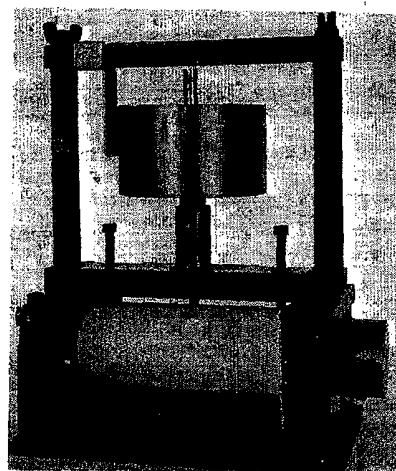


FIG. 1 Blunt Rod Penetration Tester

kg/cm² (200 psi) against the coating. Detailed design of the apparatus is shown in Fig. 1 through 6.

4.2 *Depth Gage*, a device capable of measuring the penetration movement of the rod accurately to 0.0254 mm (0.001 in.).

4.3 *Temperature Chamber*, a thermostatically controlled enclosure to provide heating and cooling of the specimen and test apparatus (excluding depth gage) to within $\pm 2^\circ\text{C}$ (3.6°F) of the desired test temperature. This equipment is optional when the test can be run within $\pm 2^\circ\text{C}$ (3.6°F) of the desired test temperature without its use.

4.4 *Temperature Controlled Box*, to fully enclose the test apparatus except the indicating means of the depth gage, as optional equipment in cases where a test is to be run at a temperature higher than room temperature. The enclosure shall be thermostatically controlled, capable of maintaining the temperature to within $\pm 2^\circ\text{C}$ (3.6°F) of the test temperature, which normally will be in the range of 23 to 76.6°C (73.4 to 170°F).

4.5 *Thermometer*, a temperature measurement device accurate to $\pm 0.5^\circ\text{C}$ ($\pm 1^\circ\text{F}$).

5. Test Specimens

5.1 The test specimens shall be 150 mm (6 in.) long and prepared with its surface preparation and coating procedures

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on the Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1988. Published July 1988. Originally published as G 17 - 71 T. Last previous edition G 17 - 83 ϵ .

TABLE 1 Dimensions for Fig. 3

Symbol	mm	in.
A	1.59	0.063
B	3.18	0.125
C	7.94	0.313
D	8.73	0.343
E	9.53	0.375
F	10.32	0.406
G	12.70	0.500
H	15.86	0.625
I	19.05	0.750
J	20.64	0.813
K	25.40	1.000
L	38.10	1.500
M	55.56	2.188
N	63.50	2.500
O	76.20	3.000
P	111.13	4.375
Q	127.00	5.000
R	184.15	7.250
S	215.90	8.500
T	50.80	2.000
U	88.90	3.500

TABLE 2 Dimensions for Fig. 4

Symbol	mm	in.
A	0.79	0.031
B	1.59	0.063
C	3.99	0.157
D	11.11	0.438
E	12.70	0.500
F	12.70	0.500
G	12.71	0.500
H	12.72	0.501
I	13.49	0.531
J	14.29	0.563
K	15.88	0.625
L	17.46	0.688
M	19.05	0.750
N	19.06	0.751
O	25.40	1.000
P	38.10	1.500
Q	60.33	2.375
R	92.08	3.625
S	101.60	4.000
T	120.65	4.750
U	141.29	5.563
V	184.15	7.250
W	203.20	8.000

equivalent to that of production coated pipe. Duplicate tests shall be run.

6. Conditioning

6.1 The specimen shall be exposed to the test temperature for a period of 24 h before beginning the test. If the test temperature is the same as room temperature, it shall be 21° to 25°C (70° to 77°F).

7. Procedure

7.1 Perform the test at room temperature or at the selected temperature.

7.2 Accurately measure the thickness coating in the area to be tested. Assemble the sample and end plates. Position the sample in the test unit so that the known film thickness

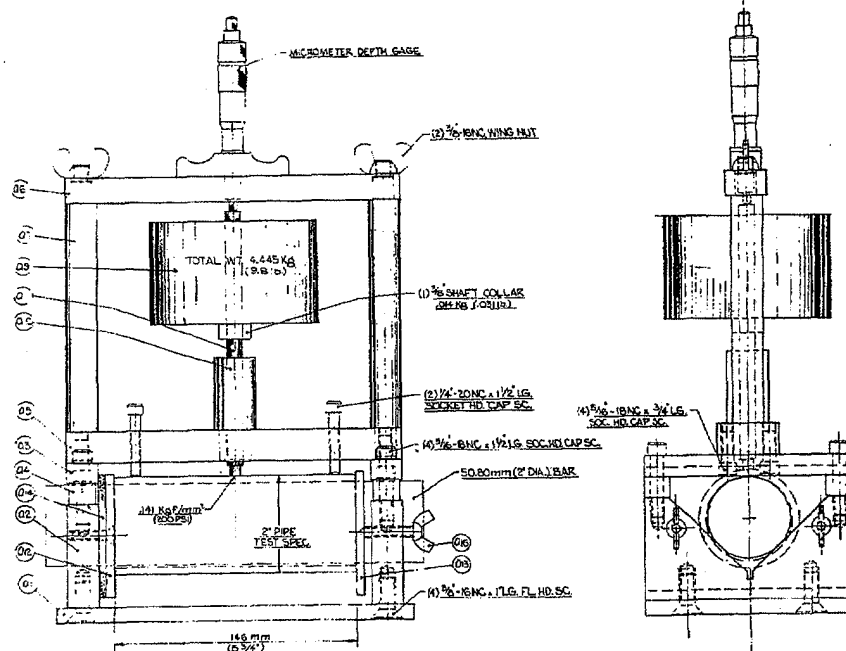


FIG. 2 Blunt Rod Penetration Tester, Assembly

TABLE 3 Dimensions for Fig. 5

Symbol	mm	in.
A	1.59	0.063
B	6.35	0.250
C	6.35	0.250
D	9.50	0.374
E	9.53	0.375
F	9.55	0.376
G	10.32	0.406
H	19.05	0.750
I	19.06	0.750
J	19.07	0.751
K	25.40	1.000
L	64.29	2.531
M	63.50	2.500
N	101.60	4.000
O	165.10	6.500

area is directly under the weighted rod and hold the sample in position by use of the cap screws.

7.3 Loosen the shaft collar and allow the weight to rest on the shaft guide. Using the depth gage take a measurement without the load applied. Raise the weight and tighten the set screw in the collar so that the weight rests on the shaft.

7.4 Take measurements every 24 h.

7.5 Continue the test until the maximum penetration has been reached as evidenced by three consecutive unchanged measurements.

8. Calculation

8.1 Calculate the net change and the percent change as follows:

TABLE 4 Dimensions for Fig. 6

Symbol	mm	in.
A	4.76	0.188
B	6.35	0.250
C	7.14	0.281
D	13.49	0.531
E	38.10	1.500
F	44.45	1.750
G	52.38	2.062
H	63.50	2.500
I	76.20	3.000
J	86.90	3.500
K	127.00	5.000
L		

$$C_N = M_F - M_I$$

$$C_P = (C_N/T) \times 100$$

where:

C_N = net change,
 M_F = final measurement,
 M_I = initial measurement,
 C_P = percent change, and
 T = film thickness.

9. Report

9.1 The report shall include the following:

9.1.1 Complete identification of specimens, including name and code number of coatings, size of the pipe, source, production date, production run number, and any other information that may be pertinent to identification,

9.1.2 Film thickness of the specimen at the test location in millimetres (inches),

9.1.3 Temperature at which the test was conducted,

9.1.4
inches
9.1.5
or defo
9.1.6
inches
9.1.7

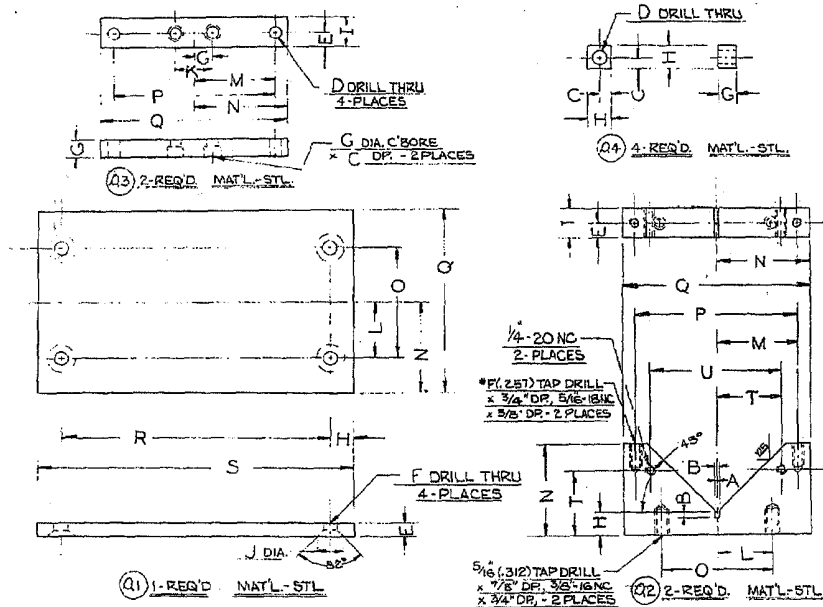


FIG. 3

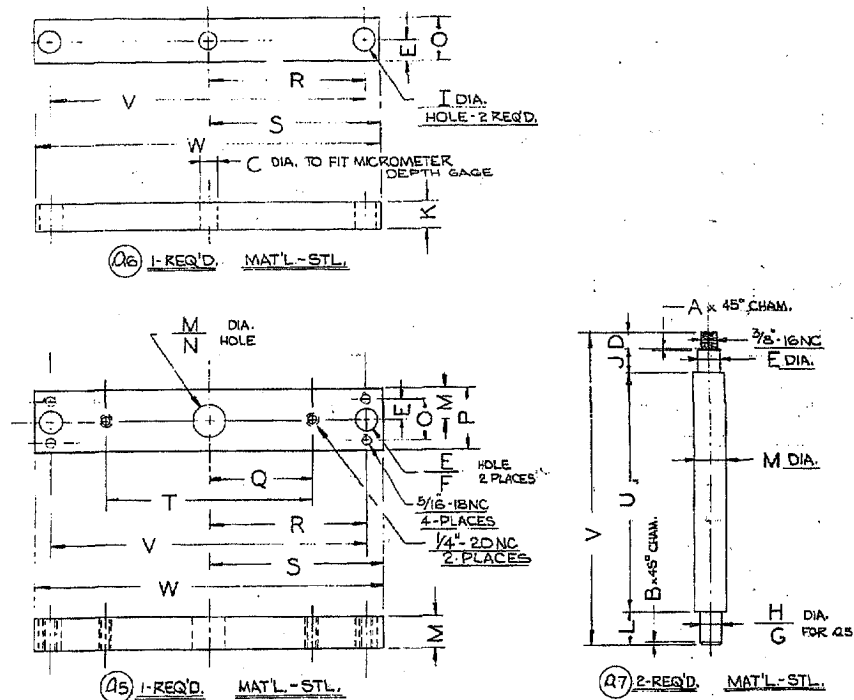


FIG. 4

- 9.1.4 Final and initial measurements in millimetres (inches),
- 9.1.5 Whether the weighted rod has caused penetration or deformation,
- 9.1.6 Net penetration or deformation in millimetres (inches) and the percent penetration or deformation, and
- 9.1.7 Number of hours or days to net penetration or

deformation.

10. Precision

10.1 *Precision Data* are limited to two adjacent specimens taken from the same source coated pipe and assume that the coating process was uniform with respect to pipe surface condition and coating material. Specimens that were not

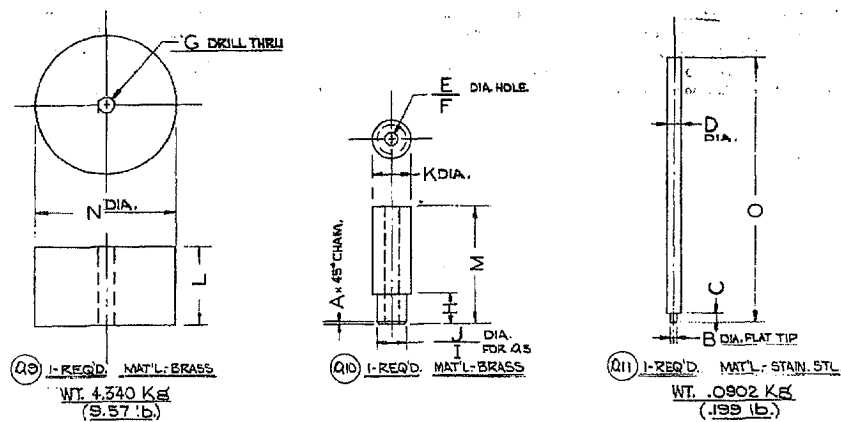


FIG. 5

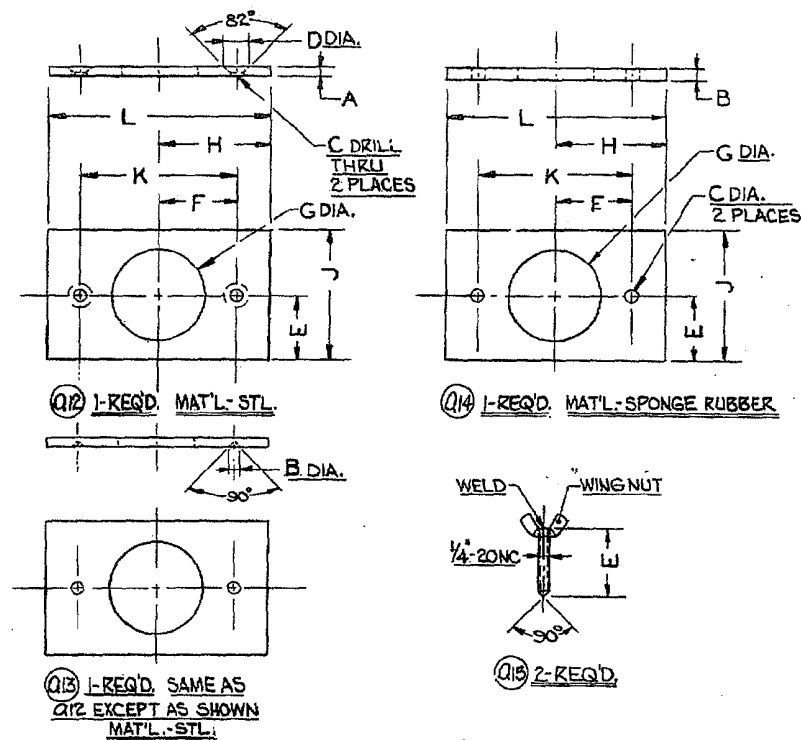


FIG. 6

adjacent in the as-produced condition or were taken from different lengths of pipe may represent differing process conditions.

10.2 Repeatability—Initial and final measurements

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should be ± 0.0127 mm (0.0005 in.) or 5 %.

10.3 Reproducibility—The results reported by one laboratory should not differ from those of another laboratory by more than ± 5 % of the net penetration.



Standard Test Method for Joints, Fittings, and Patches in Coated Pipelines¹

This standard is issued under the fixed designation G 18; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes determination of the comparative corrosion preventative characteristics of materials used for applications to joints, couplings, irregular fittings, and patched areas in coated pipelines. The test method is applicable to materials whose principal function is to act as barriers between the pipe surface and surrounding soil environment.

1.2 The test method described employs measurements of leakage current, capacitance, and dissipation factor to indicate changes in the insulating effectiveness of joint and patching materials.

1.3 *This standard may involve hazardous operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 The values stated in SI units to three significant decimals are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

3. Summary of Test Method

3.1 The test method consists of an immersion test where coated pipe specimens, each containing a simulated joint, tee, or patched area, are suspended in an electrolyte and placed under cathodic protection by connecting the specimens to the negative (-) terminal of a 6-V d-c power supply (see Fig. 1). An anode, also immersed in the electrolyte and connected to the positive (+) terminal of the power supply, completes the test circuit. Joint or patch performance is followed through periodic determinations of leakage current measured as voltage drops across a calibrated resistor in the anode-to-cathode circuit.

3.2 Capacitance and dissipation factor measurements are used to supplement the periodic leakage current determinations.

4. Significance and Use

4.1 The exposed metal surfaces at joints, fittings, and

damaged areas in an otherwise coated pipeline will be subjected to corrosion if allowed to come in contact with the soil environment. The performance of joint and patching materials designed to function as protective coverings will depend upon such factors as the ability of the material to bond to both the pipe coating and exposed metal surfaces, the integrity of the moisture seal at lapped joints, and the water absorption characteristics of the joint material.

4.2 The existence of substantial leakage current through the coating joint, patch, or fitting is reliable evidence that the material has suffered a significant decrease in its performance as a protective barrier. In a similar manner, measured changes in joint capacitance and dissipation factor are useful because they are related to the water absorption rate of the joint material. Water permeating an insulating barrier increases its capacitance and its progress can be measured through the use of a suitable impedance bridge.

5. Apparatus

5.1 *Test Vessel*, nonconducting, shall be used to contain the test specimens. Dimensions of the vessel shall permit the following requirements:

5.1.1 The test vessel shall be large enough to allow for suspension of the specimens in a vertical position and equidistant from a centrally located anode. The specimens shall not touch either each other, the walls, or bottom of the test vessel.

5.1.2 The test vessel shall be deep enough to allow for immersion of the specimens in the electrolyte to the lower edge of the upper moisture shield (see Fig. 2).

NOTE 1—A commercially available, 42 L (11-gal) waste container of high-density polyethylene can be conveniently used as a test vessel and will accommodate up to six test specimens of a size indicated in 7.2.

5.2 *Support Plate*, fabricated from a nonconductive material, to suspend the specimens in the test vessel. The support plate shall contain an access hole for the reference electrode. A typical test cell is illustrated in Fig. 3.

5.3 *Anode*, fabricated from 9.525 mm (0.375-in.) diameter 300 series stainless steel rod, 609.6 mm (24.00 in.) long. Other inert anodes such as carbon or platinum may be used.

5.4 *D-C Voltmeter*, to serve the dual purpose of (1) measuring leakage current as a potential drop across a 1,000- Ω shunt in the measuring circuit and (2) measuring the potential of the test specimen with reference to a Cu-CuSO₄ half cell. The instrument characteristics for these functions shall be:

5.4.1 *Voltage Range*—50 μ V full scale to 10 V full scale in overlapping 1 $\times$ and 3 $\times$ ranges.

5.4.2 *Accuracy*— ± 3 percent of full scale on all ranges.

5.4.3 *Input Resistance*—Greater than 10 M Ω on all ranges.

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved Sept. 30, 1988. Published November 1988. Originally published as G 18 - 71 T. Last previous edition G 18 - 83.

² *Annual Book of ASTM Standards*, Vols 06.01 and 14.02.

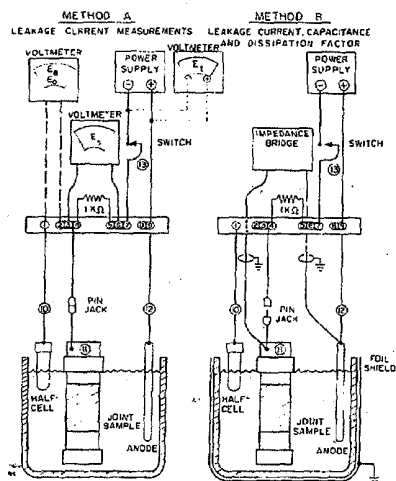


FIG. 1 Test Circuits

5.5 *Thickness Gage*—Measurements of coating thickness will be required for this test. Any instrument suitable for use with Test Method G 12² can be used. However, the choice of measuring gage shall be compatible with the joint coating thickness that will be encountered in the test.

5.6 *Ohmmeter*—Measurements for end-cap integrity shall be made with a suitable ohmmeter capable of reading resistance to an upper limit of $1000 \text{ M}\Omega \pm 5\%$.

5.7 *Reference Electrode*—A Cu-CuSO₄ half cell of conventional glass or plastic tube with porous plug construction, but preferably not over 19.05 mm (¾ in.) in diameter, having a potential of -0.316 V with respect to the standard hydrogen electrode.

NOTE 2—A saturated calomel half cell may be used, but measurements made with it shall be converted to the Cu-CuSO₄ reference for reporting by adding -0.072 V to the observed reading.

5.8 *Voltage Source*—A battery or rectifier-type power supply shall be used to maintain a potential difference of $6.0 \pm 0.1 \text{ V}$ dc between each of the test specimens and the Cu-CuSO₄ half cell. Where multiple specimens are tested, a suitable voltage-dividing circuit will be required for individual control of the voltage applied to each specimen.

5.9 *Circuit Wiring* from the anode to specimen shall be of No. 18 Awg insulated copper. A switch for disconnecting each specimen from its voltage source shall be included in the circuit. A $1000 \Omega \pm 1\%$ percent, 1-W (minimum) precision resistor shall be placed in the anode-to-cathode circuit as a shunt for current. A diagram illustrating the test cell wiring appears as Fig. 1.

5.10 *Capacitance Bridge*—Measurements of specimen capacitance and dissipation factor shall be made with a low-voltage a-c, resistive ratio arm-type bridge³ having the following characteristics:

5.10.1 *Oscillator Frequency*, 1 kHz $\pm 2\%$ tolerance.

5.10.2 *Series Capacitance Range*, 100 pF to 1100 pF accuracy $\pm 1\% \pm 1 \text{ pF}$, whichever is larger.

5.10.3 *Dissipation Factor Range*, 0.002 to 1.0 at 1 kHz accuracy $\pm 5\%$ or ± 0.001 dissipation, whichever is larger.

5.11 *Connectors*—Miniature, pin-type, insulated jacks shall be used at the point of connection to each test specimen. The jacks serve two important functions: (1) they permit the disconnection of the specimen from the voltage source when the impedance bridge is in use, and (2) disconnection of the specimen from the test circuit also removes the effect of stray capacitance due to excessive lead length that may introduce error into impedance bridge measurements.

5.12 *Foil Shield*—As an additional safeguard against stray capacitance effects, wrap the entire test vessel in heavy-gauge aluminum foil as shown in Fig. 3 and ground the shield.

6. Materials

6.1 *Electrolyte*—The electrolyte shall consist of potable tap water with the addition of 3 weight percent of technical-grade sodium chloride.

6.2 Materials for sealing the ends of the specimens may consist of waxes, epoxies, or other suitable materials. However, they should have a dielectric constant in the range from 2 to 6, and exhibit a low water-absorption rate. It is also important that the end-cap material maintains good adhesion to any coated pipe surfaces throughout the test period.

7. Test Specimen

7.1 The joint specimen shall be prepared from a representative piece of 60.325 mm (2.375 in.) outside diameter, Schedule 40, production-coated pipe.

7.2 Each piece of coated pipe shall be 381 mm (15.0 in.) long and serve as a carrier for the particular joint material to be tested.

7.3 The simulated coating joint shall be made by cleaning away a 101.6 mm (4.0-in.) band of pipe coating starting from a point 203.2 mm (8.0 in.) below the upper end of the pipe. The coating may be removed by power brushing or any other suitable device that will remove all of the coating in the area indicated and leave a clean metal surface behind.

7.4 The lower end of the test specimen shall be plugged flush with a stopper and sealed or capped with a material meeting the requirements of 6.2.

7.5 When used, the primer shall be applied to the test specimen in sufficient quantity to coat the test specimen from a point 88.9 mm (3.50 in.) below its upper end and ending at a point 317.5 mm (12.5 in.) from the upper end. This will allow for a 12.7-mm (0.5-in.) band of excess primer beyond each tape margin.

7.6 The joint material to be tested shall be applied to the prepared pipe specimen (using a spiral wrap for tapes) and starting from a point 101.6 mm (4.0 in.) below the upper end of the pipe section and ending at a point 304.8 mm (12.0 in.) from the upper end. The 203.2 mm (8.0 in.) of joint material thus applied should overlap the bared section of pipe by 50.8 mm (2.0 in.) at each end. This distance represents the typical cut-back distance encountered in the field joining, through welding, of coated pipe in 60.325 mm (2.375 in.) outside diameter size. A diagram of the joint specimen appears as Fig. 2.

³ Instruments found to meet these requirements are the General Radio Type 1650-B Impedance Bridge and the Heath Co. Model 1B-28 Impedance Bridge (Kit).

7.7 The manner of applying the joint material shall be done in accordance with the manufacturer's specifications. The supplier of the joint material should specify the desired time interval between the application of the material and the start of the test.

NOTE 3—Materials that are intended for use as a field-applied patch over damaged areas on coated pipelines can be tested using the same procedures, with the patching compound applied, instead, to the bared area of the simulated joint.

7.8 The upper 76.2 mm (3.0 in.) of the completed joint test specimen shall be coated with the material used for the lower end cap. This moisture shield can be conveniently made, in the case of some waxes and epoxies, with several successive brush or dip-applied applications. The thickness of the moisture shield should be approximately 3.175 mm (0.125 in.).

8. Testing Temperature

8.1 Perform all tests at a room temperature of 21 to 25°C (70 to 77°F).

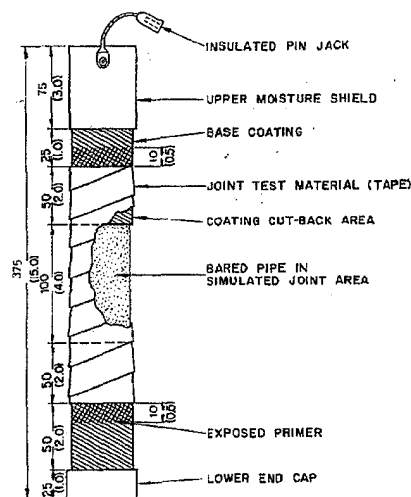
9. Preliminary Test Measurements

9.1 *Coating and Joint Thickness*—Measure and record the thickness of the base coating which lies exposed at each end of the test joint. Measurements shall be made in accordance with Method G 12. In a like manner, measure and record the average thickness of the joint covering.

10. Procedure for Leakage Current Measurements

10.1 Suspend the joint test specimens in the test vessel, observing the clearances specified in 5.1.1 and 5.1.2. Fill the vessel with electrolyte, bringing the fluid level up to a point midway between the end cap and the lower edge of the joint material.

10.2 Connect one terminal of the ohmmeter to the test specimen and the other terminal to the central anode. The



NOTE—All dimensions are in millimetres with inches in parentheses.

FIG. 2 Joint Test Specimen

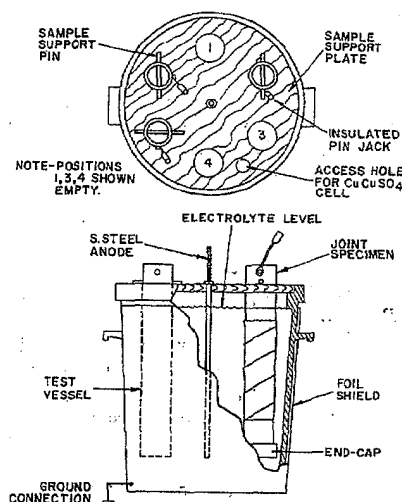


FIG. 3 Joint Test Cell

anode should be in contact with the electrolyte. Measure the apparent sample-to-anode resistance in ohms. The reading shall remain above 1000 MΩ for 15 min. Readings below this value probably indicates a faulty end-cap seal which should be repaired before the joint specimen is totally immersed.

10.3 Totally immerse the joint test specimens up to the lower edge of the moisture shield. This level shall be maintained throughout the test period by regular additions of tap water. The test shall be performed at room temperature.

10.4 Connect each joint test specimen in series with a 1000-Ω ± 1% (1-W) resistor, a suitable switch, and the negative (-) terminal of the voltage source.

10.5 Connect the central stainless steel anode to the positive (+) terminal of the voltage source.

10.6 Energize the voltage source. Adjust the voltage to each specimen by connecting a voltmeter between each sample and the Cu-CuSO₄ half cell and varying the voltage output until 6.0 V are measured between sample and half cell (see Fig. 1).

10.7 As soon as the circuit is energized and voltage adjusted to each joint test specimen, measure and record the voltage across each 1000-Ω shunt resistor.

10.8 Measure and record the closed circuit potential, E_B , and open circuit potential, E_o , of each joint specimen with reference to the saturated Cu-CuSO₄ half cell.

NOTE 4—Any drop in closed circuit potential (E_B) of a joint specimen greater than 20% of the terminal voltage (E_t) probably indicates a developing current leak in the joint area. In a like manner, any open circuit potential (E_o) greater than 0.05 V may indicate the presence of a developing conductive path across the test joint. (Both voltages are referenced to the Cu-CuSO₄ half cell.)

10.9 Using the d-c voltmeter in 5.4 measure and record the voltage appearing across the terminals of the voltage supply.

10.10 Continue, on a periodic basis, the measurements specified in 10.7 through 10.9. Calculate the apparent joint

resistance by the method described in 12.1.

10.11 Prior to removal, reexamine a suspected joint failure to confirm that any observed drop in joint resistance is not due to a faulty end-cap seal. This can best be accomplished by removing the specimen from the test cell and allowing it to dry. When dry, reimmerse it to just above the level of the lower end cap and check the integrity of the cap by the method outlined in Section 8.

11. Procedure for Measurement of Capacitance and Dissipation Factor

11.1 The series capacitance and dissipation factor between each joint sample and the central stainless-steel anode shall also be measured. These measurements shall be made at the time of initial immersion and continued on an identical schedule with the voltage readings. They shall be made in the following manner:

11.2 Temporarily disconnect the test specimen from the voltage source. Connect the impedance bridge between the test specimen and stainless-steel anode. Energize the bridge and measure the equivalent series capacitance, C , and dissipation factor, DF , or the test specimen using a "null" technique to balance the measuring bridge.

11.3 A continued rise in joint capacitance and dissipation factor from the values recorded at the time of initial immersion, will indicate progressive absorption of water by the joint material.

11.4 The joint test shall continue for 180 days or until the joint resistance falls to a value of 4600Ω for 1 m^2 ($50\,000 \Omega$ for 1 ft^2) of immersed joint surface.

12. Calculations

12.1 *Joint Resistance*—Calculate the resistance, R_j , for the joint sample as follows:

$$R_j = 1000 A[(E_B - E_o)/E_s]$$

where

R_j = resistance, Ω/cm^2 (Ω/ft^2),

A = immersed sample area (excluding end cap), cm^2 (ft^2),

E_B = closed circuit potential of sample (reference Cu-CuSO_4), V (switch closed),

E_o = open circuit potential of sample (reference Cu-CuSO_4), V (switch open), and

E_s = voltage across $1000\text{-}\Omega$ shunt resistor, V, (switch closed).

13. Report

13.1 All test reports shall include the following:

13.1.1 Complete identification of the joint specimen including:

13.1.1.1 Name and code number of the base coating,

13.1.1.2 Thickness of the base coating,

13.1.1.3 Name and code number of the joint material, including type (that is tape, two-part mix, etc.),

13.1.1.4 Method of application of joint material, including amount of overlap.

13.1.1.5 Where applicable, the width and thickness of the joint material (tapes),

13.1.1.6 Where applicable, the name, code number, and type of primer,

13.1.1.7 Where applicable, the method of application of primer,

13.1.1.8 Average thickness of the joint material, as applied, and

13.1.1.9 Dates of starting and terminating the test and other information that may be pertinent.

13.2 The following readings shall be recorded on a periodic basis:

13.2.1 Voltage across the $1000\text{-}\Omega$ shunt resistor, E_s ,

13.2.2 Closed-circuit potential of the joint specimen referenced to the Cu-CuSO_4 half cell, E_B ,

13.2.3 Open-circuit potential of the joint specimen referenced to the Cu-CuSO_4 half cell, E_o ,

13.2.4 Terminal voltage at the power supply, and

13.2.5 Calculated value of joint resistance, R_j ,

13.2.6 Initial series capacitance of the joint sample, and

13.2.7 Initial dissipation factor of the joint sample.

NOTE 5—The frequency of experimental readings will depend upon the performance of each joint specimen. Specimens that show a high rate of change may require more frequent watching if the point of failure is to be recorded. Normally, weekly readings have been found to be adequate. Joint performance can be conveniently followed through plots of Joint Resistance versus Time and Dissipation Factor versus Time in semilogarithmic coordinates, and Capacitance Change versus Time in rectangular coordinates.

14. Precision

14.1 Due to the range of joint coating formulations, thicknesses, densities, etc. found among commercially available materials, the over-all accuracy and reproducibility of test results by these methods will tend to be poorer than those expected on straight runs of coated pipe. The fact that most joint materials are hand-applied introduces an inherent factor of variability into the test results. The basic purpose of these methods is to provide a means for the comparative screening of combinations of joint materials and base coatings for insulating effectiveness.

14.2 The precision (reproducibility) of the joint resistance determination by these methods is considered to be such that when two tests are performed consecutively on the same specimen and under identical conditions of application, the difference between the two results may normally be expected not to exceed $\pm 10\%$ of their mean.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Disbonding Characteristics of Pipeline Coatings by Direct Soil Burial¹

This standard is issued under the fixed designation G 19; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method describes the determination of the relative disbonding characteristics of damaged coatings on steel pipe by cathodic protection potentials in direct soil burial. This test method is intended to apply to the testing of all types of nonmetallic pipeline coatings and tapes including thermoplastics, thermoset, and bituminous materials.

1.2 Results may vary widely when test sites are in different geographical areas of the country, and even in different localities.

1.3 This test method is limited to nonconducting, or nonmetallic pipe coatings and is not applicable to conducting materials such as zinc coatings on steel pipe.

1.4 This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.5 The values stated in SI units to three significant decimals are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

G 62 Test Methods for Holiday Detection in Pipeline Coatings³

3. Summary of Test Method

3.1 Apparatus and materials are described whereby protective coatings on steel pipe are subjected to disbonding by an electrical stress. Specimens with intentionally damaged areas are buried in soil at an outdoor site and electrically connected to a magnesium anode. After test, the disbonded coating is removed, the exposed area measured, and comparisons are made to other specimens similarly exposed.

4. Significance and Use

4.1 Coated pipe is seldom, if ever, buried without some damage to the coating. Hence, an actual soil-burial test can contribute significant data, provided the method of testing is

controlled and the test specimen monitored and the relationship between the area disbonded, the current demand, and the mode of failure is fully understood.

4.2 Means are provided for measuring and following the electrical potential and current flow and relating these data to the final measurement of disbonded area.

5. Apparatus.

5.1 *Anode*—A standard packaged magnesium anode, minimum 4.082 kg (9 lb), with a factory-sealed, 4107-cmil (14-gage Awg) minimum, insulated copper wire shall be used. A solution potential of not less than -1.45 V with respect to a copper-copper sulfate reference electrode is required. Use sufficient anodes to maintain required potential.

5.2 *Connectors*—The wiring circuit from anode to test specimen and from specimen to reference electrode should be 4107-cmil (14-gage Awg) minimum insulated copper wire. Attach the wires to the test specimen as shown in Fig. 1 by soldering or brazing at the air-exposed end, and coat the place of attachment with insulating material. A junction box is optional for connecting the resistor in series between the anode and the test specimen.

5.3 The instruments used shall include the following:

5.3.1 *Voltmeter*, a suitable instrument such as a high impedance ($>10\text{M}\Omega$) analog multimeter having a sensitivity of $50\ 000\ \Omega/\text{V}$ minimum and a multiple range from 0.01 to 2 V for direct current is used for measuring the potential between specimen and the reference electrode,⁴ current between specimen and anode, and the resistance of the circuit. The same instrument shall be used for measuring current between specimen and anode. Alligator clips on the leads are permissible.

5.3.2 *Volt-Ohm-Meter* for measuring resistance of the circuit.

5.3.3 *Reference Electrode*, consisting of a copper-copper sulfate half cell in a conventional glass or plastic tube with porous plug construction, but preferably not over 19.05 mm ($3/4$ in.) in diameter, having a potential of -0.316 V with respect to a standard hydrogen electrode. A calomel electrode may be used, but measurements made with it should be converted to the copper-copper sulfate reference electrode for reporting, by adding -0.092 V to the observed reading.

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1988. Published July 1988. Originally published as G 19-71 T. Last previous edition G 19-83.

² Annual Book of ASTM Standards, Vols 06.01 and 14.02.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ A pipe-to-soil Voltmeter-Ammeter, Agra Engineering Co., Tulsa, Okla., has been found suitable for this test.

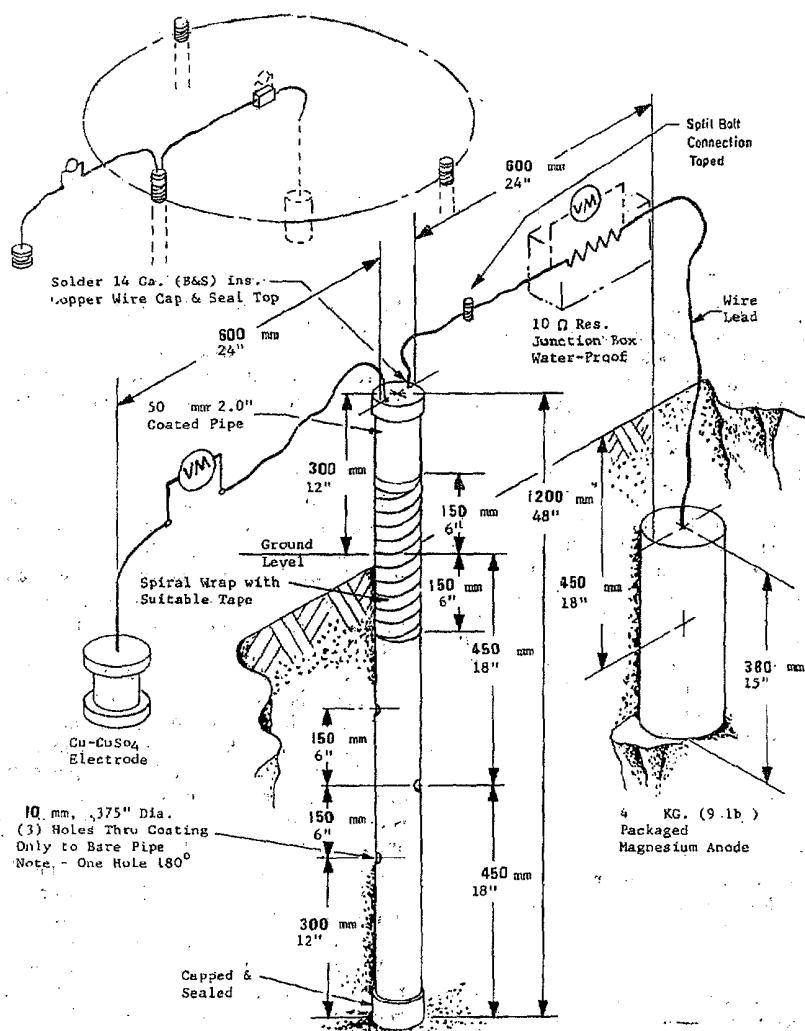


FIG. 1. Three Specimens in a Circle with a Common Anode

5.3.4 A suitable instrument is used to measure the soil resistivity using the four-pin method.⁵ Pins should be spaced 762 mm (30 in.) apart.

5.3.5 *Thickness Gages*, to be used in accordance with Test Method G 12.

5.3.6 *Holiday Tools*—Holidays in the specimen are made with conventional drills of the required diameter. A 9.525-mm (0.375-in.) drill modified by substantially reducing the cone angle has been found effective in preventing perforation of thin-wall pipe or tubing. A sharp-pointed knife with a safe

handle is required for removing disbonded coatings to make physical examinations. A micrometer-type depth gage is used for measuring coating thicknesses at the edge of holidays.

5.3.7 *Holiday Detectors*—Selected in accordance with Test Method G 62.

6. Test Specimen

6.1 A 1219.2 mm (48-in.) long specimen shall be prepared with its surface preparation and coating procedures equivalent to that of production coated pipe. Only holiday-free specimens may be used in this test and four samples should be prepared.

⁵ A Vibroground instrument, Associated Research Inc., 3758 Belmont Ave., Chicago, Ill., has been found suitable for measuring soil resistivity.

6.2 Measure the coating thickness of all specimens at four points 90 deg apart and approximately 355.6 mm (14 in.) from each end.

6.3 Cap and seal one end of each specimen. Check the seal for current leakage before and after test by immersion in 1 percent sodium chloride solution for ½ h. Test with an ohmmeter sensitive to at least 1 MΩ.

6.4 Provide the unsealed end of the pipe with two lengths of insulated 4107-cmil (14-gage Awg) copper wire as in 5.2.

6.5 Make 3 holidays 304.8 mm (12 in.), 457.2 mm (18 in.), and 609.6 mm (24 in.), respectively, from the sealed end of the pipe by drilling holes through the coating so that the drill will fully enter the steel. The center holiday is to face the anode with the remaining two holidays facing 180 deg away from the anode. The drill diameter may not be less than two times the coating thickness, and never smaller than 9.525 mm (0.375 in.) in diameter. Do not perforate the steel wall of the pipe. With thin-wall steel pipe where there is danger of perforating the pipe, start the holiday with a standard 60-deg cone-point drill and finish with a substantially reduced cone-angle drill.

6.6 To prevent mechanical damage, apply a spiral wrap of suitable tape from approximately 152.4 mm (6 in.) from the top of the pipe specimen to 457.2 mm (18 in.) from the top. When the test specimens are buried, the taped area will extend 152.4 mm above and 152.4 mm below ground.

6.7 The specimen test area will consist of the area between the edge of the bottom end seal and the ground level. The bottom end-seal area is not considered part of the area tested. Any suitable diameter and length of pipe may be used, but the buried area should not be less than 23 227.2 mm² (36 in.²). An area of 93 000 mm² (1 ft²) has been found preferable.

7. Test Site

7.1 Any level location may be used, provided the site will not be disturbed for the duration of the test.

8. Procedure

8.1 Lay out the test site with stakes so that the anode location will be the center of a circle as shown in Fig. 1. Locate the pipe specimens 20° apart around the circumference so that the surface of the anode when placed in the center will be 609.6 mm (24 in.) from the nearest holiday of each specimen equally. Three specimens can thus be protected by one anode.

8.2 Installation of Test Specimens:

8.2.1 Dig suitable holes with an auger or posthole digger to accommodate the anode and test specimens at the test site.

8.2.2 Insert the anode at the center of the circle. The center of the anode should be 457.2 mm (18 in.) below ground. Pipe specimens should be inserted so that 304.8 mm (12 in.) are above the ground level, and oriented so that the single intentional holiday faces the anode and the remaining two holidays are 180 deg away from the anode. Maintain a distance of 609.6 mm (24 in.) between the surface of the anode and the nearest specimen and holiday.

8.2.3 Refill all holes with soil or a soil and water slurry. Firmly tamp the soil so that it is in intimate contact with the specimen and anode. No wood or other foreign material

should contact the pipe coating or the anode.

8.3 Electrical Measurements:

8.3.1 Determine in several areas the soil resistance in ohm-centimetres by the four-pin method.

8.3.2 Measure the initial pipe to soil potential with reference to a copper-copper sulfate half cell with the electrode 609.6 mm (24 in.) from the pipe as shown in Fig. 1. Record the closed-circuit potentials.

8.3.3 Connect the anode lead to one test specimen lead at the junction box with a 10 Ω ± 1 % wire-wound resistor connected in series between the anode and test specimen.

8.3.4 Measure the voltage, E , across the 10-Ω resistor, R , and convert to current, I as follows:

$$I = E/R = E/10$$

8.3.5 Measure the polarized potential, in volts with the analog multimeter described in 5.3.8 connected between the test specimen and the reference electrode as follows:

8.3.5.1 Disconnect the anode from the test specimen while closely observing the analog multimeter. As the instrument pointer falls, it will dwell significantly at the polarized value before receding further. The dwell point is the polarized potential.

8.3.6 Determine electrical measurements at 30-day intervals for a test period of 18 months or longer depending on soil conditions.

9. Report

9.1 The report shall include the following:

9.1.1 Complete identification of the test specimens, including name and code number of coating, size of the pipe, source, production date, production run number, and any other information that may be pertinent to identification.

9.1.2 Date of starting and of terminating the test.

9.1.3 Coating thickness of the test specimen before testing.

9.1.4 After subtracting the initial holiday areas, report the total disbonded area, average disbonded area, and disbonded area at each holiday in square millimetres (square inches) after testing.

9.1.5 Condition of the pipe surface under the disbonded coating.

9.1.6 Pipe-to-soil potentials, initial readings and at 30-day intervals.

9.1.7 Current readings between anode and pipe specimens, initial readings and 30-day intervals.

9.1.8 Soil resistivity readings in ohm-centimetres.

9.1.9 Polarized potentials, initial readings and 30-day intervals.

10. Precision

10.1 Precision data are limited to two adjacent specimens taken from the same production-coated pipe, assuming that the production process was uniform with respect to pipe surface condition and coating material. Specimens that were not adjacent in the as-produced condition, or were taken from different lengths of pipe may represent differing process conditions.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

Scope

1.1 This standard covers the use of the test method for the determination of the relationship between the reported and actual values of the test method. It is applicable to the use of the test method for the determination of the relationship between the reported and actual values of the test method.

References

2.1 ASTM D 543
D 883
G 8 Test Method for Coefficient of Friction
G 12 Test Method for Film
G 17 Test Method for Coefficient of Friction

Summary

3.1 This standard covers the use of the test method for the determination of the relationship between the reported and actual values of the test method. It is applicable to the use of the test method for the determination of the relationship between the reported and actual values of the test method.

Significance and Use

4.1 This standard covers the use of the test method for the determination of the relationship between the reported and actual values of the test method. It is applicable to the use of the test method for the determination of the relationship between the reported and actual values of the test method.

¹ This test method is under development by the Subcommittee G03 on Coefficient of Friction, published as an annual book.
² Annual book.
³ Annual book.



Standard Test Method for Chemical Resistance of Pipeline Coatings¹

This standard is issued under the fixed designation G 20; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method is intended for evaluating the resistance of pipe coating materials when exposed to various concentrations of reagents or suspected soil contaminants. The test serves as a guide to investigators wishing to compare the relative merits of pipe-coating materials in specific environments. The choice of reagents, concentrations, duration of immersion, temperature of test, and properties to be reported are necessarily arbitrary and should be chosen to reflect conditions known to exist along the pipeline right-of-way.

1.2 *This test method may involve hazardous operations and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 543 Test Method for Resistance of Plastics to Chemical Reagents²
- D 883 Definitions of Terms Relating to Plastics²
- G 8 Test Methods for Cathodic Disbonding of Pipeline Coatings³
- G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel³
- G 17 Test Method for Penetration Resistance of Pipeline Coatings (Blunt Rod)³

3. Summary of Test Method

3.1 This test method consists of an immersion-type test in a closed container where coated pipe specimens are in long-term contact with both the liquid and vapor phase of the test reagent. Specimens exposed in this manner are inspected for visible signs of chemical attack. Subsequent tests for cathodic disbonding in accordance with Test Method G 8, or penetration under load in accordance with Test Method G 17, may be applied to determine if the specimens have undergone any loss of mechanical or bonding properties.

4. Significance and Use

4.1 The data obtained for short-term tests are of interest

only in eliminating the most unsuitable materials or for indicating a probable order of resistance in any particular media.

4.2 Test conditions should take into account the manner and duration of immersion, the reagent, the temperature of the system, the area exposed above and below the liquid level, and other performance factors selected for the particular test.

5. Apparatus

5.1 *Thickness Gage*, capable of measuring the coating thickness in the manner prescribed by Test Method G 12.

5.2 *Test Container*—A transparent closed container, sized to completely encase the pipe specimen and large enough to provide adequate exposure to both the liquid and vapor states of reagent.

NOTE 1—For example, a 2-L (2.0-qt) capacity, Mason-type jar with a 70-mm (2.75-in.) diameter neck has been found suitable for use with 2 in. pipe and is illustrated in Fig. 1.

5.2.1 To avoid pressure build-up within the test containers, the threaded cup shall be replaced with a solid-rubber stopper. A positive venting device, such as a water seal, shall be used when testing with volatile solvents at elevated temperatures.

5.2.2 A separate container shall be used for each test specimen.

5.3 *Oven or Constant-Temperature Room or Bath*—To ensure uniformity of test results, the test cells and specimen shall be maintained at the test temperature $\pm 5^\circ\text{C}$ (9°F) over the duration of the test period.

5.4 *Auxiliary Testing Devices*—Supplemental equipment used to determine specific mechanical properties of specimens before and after immersion shall conform to the requirements prescribed in the applicable ASTM test method.

6. Test Specimen

6.1 The test specimen shall be prepared with its surface preparation and coating procedures equivalent to that of coated pipe. A control specimen shall be retained for comparison.

6.2 In order to utilize the test container specified in 4.2, the pipe specimen, with end caps, shall be restricted to a maximum overall length of 180 mm (7.0 in.) and a diameter of 65 mm (2.5 in.).

6.3 Both ends of the pipe specimens shall be plugged with inert stoppers and coated with an epoxy-base coating material.

6.4 Specimens with Holidays:

6.4.1 A holiday shall be made in the surface of the coated pipe specimen at a point 25 % of the distance between the

¹ This test method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 27, 1988. Published July 1988. Originally published as G 20 - 71 T. Last previous edition G 20 - 83.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

end caps. It shall be made by drilling a radial hole through the coating so that the angular cone point of the drill will fully enter the steel where the cylindrical portion of the drill meets the steel surface. The drill diameter shall not be less than three times the coating thickness, but it shall never be smaller than 6 mm (0.25 in.) in diameter. The steel wall of the pipe shall not be perforated.

6.4.2 A second identical holiday shall be drilled into the coating surface at a point 25 % of the distance between the end caps of the specimen (see Fig. 1). Both holidays should lie in the same pipe axis.

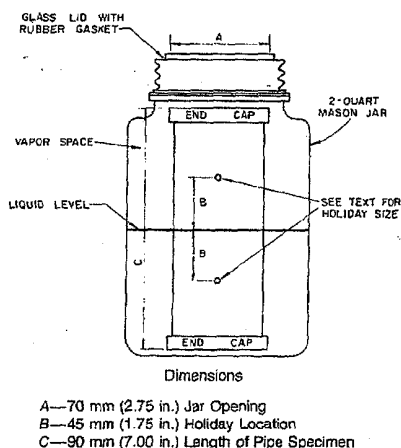
6.5 Specimens without intentional holidays shall also be prepared for testing.

NOTE 2—Intentional holidays shall be made in only those specimens for which a comparison of disbonding properties is desired. Other samples used for supplemental mechanical testing shall be run without holidays.

7. Reagents

7.1 The reagents selected for coating-resistance tests should be those anticipated to occur in the environment or in the product being carried in the pipeline at the temperatures and in the concentrations expected. The numbers in parentheses refer to the list of standard reagents given in Section 4 of Test Method D 543.

- 7.1.1 Acetic Acid (5 %) (4.4.2).
- 7.1.2 Acetone (4.4.3).
- 7.1.3 Carbon Disulfide.
- 7.1.4 Gasoline.
- 7.1.5 Hydrochloric Acid (10 %) (4.4.23).
- 7.1.6 Kerosine (4.4.28).
- 7.1.7 Lime Water, Saturated.
- 7.1.8 Methyl Alcohol (4.4.29).
- 7.1.9 Methyl Ethyl Ketone.
- 7.1.10 Nitric Acid (10 %) (4.4.33).
- 7.1.11 Sodium Carbonate Solution (20 %) (4.4.38).
- 7.1.12 Sodium Chloride Solution (10 %) (4.4.40).
- 7.1.13 Sodium Hydroxide Solution (10 %) (4.4.42).



NOTE—Dimensions shown for 2 in. IPS pipe.

FIG. 1 Chemical Immersion Test Schematic of Test Cell

- 7.1.14 Sulfuric Acid (30 %) (4.4.46).
- 7.1.15 Toluene (4.4.48).
- 7.1.16 Transformer Oil (4.4.49).
- 7.1.17 Trichlorethylene.
- 7.1.18 Other selected environments.

8. Precautions

8.1 Safety precautions should be taken to avoid personal contact, to eliminate toxic vapors, and to guard against explosion hazards in accordance with the hazardous nature of the particular reagents being used.

9. Procedure

9.1 Place a single specimen in a vertical position in each test container.

9.2 Fill the container with the selected reagent so that the liquid level covers one half of the coated pipe specimen up to a point midway between the two intentional holidays. Stopper the 704 Container to prevent evaporation of the reagent and against contamination.

9.3 Maintain the reagent level at the original level.

9.4 The basic immersion test for a particular coating material shall consist of twelve specimens, six with holidays and six without, each in an individual test cell. Remove duplicate samples of each from the test at 30, 60, and 90 day intervals. Additional investigations of pre- and post immersion mechanical properties will require additional specimens. It is recommended that in all cases provisions be made for duplicate test specimens.

9.5 Remove the specimen after 30, 60, and 90 days of immersion. Wash with running water all specimens removed from acid, alkali, or other aqueous solutions and wipe them dry with a soft, clean, cotton cloth or paper tissue. Specimens removed from volatile solvents such as acetone, alcohol, etc., need no rinsing before wiping dry.

9.6 Observe and report before, immediately after, and 2 h later the appearance of specimen after exposure to reagent on the basis of visual examination for evidence of loss of gloss, developed texture, decomposition, discoloration, softening, swelling, injury, bubbling, blistering, cracking, solubility, etc. as defined in D 883.

9.7 Mechanically probe each of the intentional holidays in accordance with Section 8.3.3 of Test Method G 8 to see if there has been any loss of coating bond during the test period.

9.8 Perform other planned mechanical tests to determine if any degradation of coating properties has occurred through reagent exposure.

10. Report

- 10.1 The report shall include the following:
 - 10.1.1 Complete identification of the material tested, including type, source, manufacturer's code, and previous history,
 - 10.1.2 Method of preparing test specimen,
 - 10.1.3 Temperature of test,
 - 10.1.4 Description of media, including ASTM designation,
 - 10.1.5 Duration of immersion,
 - 10.1.6 Outside diameter of test specimen,
 - 10.1.7 Initial thickness of coating,

- 0.1.8 General appearance of specimen after immersion,
- 0.1.9 Immersion area in square millimetres (square inches),
- 0.1.10 Vapor phase area in square millimetres (square inches), and
- 0.1.11 Disbonded area after immersion in square millimetres (square inches).

11. Precision and Bias

11.1 As there are no direct measurements involved with this test method, no precision and bias statement is necessary. Measurements made by procedures for other test methods should refer to the precision and bias statements therein.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Practice for Operating Light-Exposure Apparatus (Carbon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials¹

This standard is issued under the fixed designation G 23; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice covers the basic principles and operating procedures for light-exposure apparatus with and without water spray employing a carbon-arc light source.

1.2 This practice does not specify the exposure conditions best suited for the material to be tested. It is limited to the method of obtaining, measuring, and controlling the conditions and procedures of the exposure. Sample preparation, test conditions, and evaluation of results are covered in ASTM test methods or specifications for specific materials.

NOTE 1—Attention is called to the following test methods and practices for more information on use of this practice for specific materials:

Practices D 529, D 822, D 904, D 1499, D 3361, D 3815, E 765 and Test Methods C 732, C 734, C 741, D 750, D 3424 and D 3583.

1.3 This practice includes four procedures:

1.3.1 *Method 1*—Continuous exposure to light and intermittent exposure to water spray.

1.3.2 *Method 2*—Alternate exposure to light and darkness and intermittent exposure to water spray.

1.3.3 *Method 3*—Continuous exposure to light without water spray. Specific exposure conditions for testing fabric are found in AATCC Test Method 16A.

1.3.4 *Method 4*—Alternate exposure to light and darkness without water spray.

1.4 The values stated in SI units are to be regarded as the standard. The inch-pound unit equivalents of the SI units may be approximate.

1.5 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

¹ This practice is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.03 on Simulated and Controlled Environmental Tests.

Current edition approved Oct. 26, 1990. Published December 1990. Originally published as E 42 - 42 T. Last previous edition G 23 - 89. Replaces E 42 - 69 and combines G 23 - 69 (1975) and G 25 - 81.

Practice G 25, Standard Recommended Practice for Operating Enclosed Carbon Arc Type Apparatus for Light Exposure of Nonmetallic Materials, is to be discontinued since it now is covered in this edition of G 23 and its Methods 3 and 4.

C 732 Test Method for Aging Effects of Artificial Weathering on Latex Sealants²

C 734 Test Method for Low-Temperature Flexibility of Latex Sealants After Artificial Weathering²

C 741 Test Method for Accelerated Aging of Wood Sash Face Glazing Compound²

D 529 Practice for Accelerated Weathering Test of Bituminous Materials³ (Carbon-Arc Method)

D 750 Test Method for Rubber Deterioration in Carbon-Arc or Weathering Apparatus⁴

D 822 Practice for Conducting Tests on Paint and Related Coatings and Materials Using Filtered Open-Flame Carbon-Arc Light- and Water-Exposure Apparatus⁵

D 904 Practice for Exposure of Adhesive Specimens to Artificial (Carbon-Arc Type) and Natural Light⁶

D 1499 Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Exposure of Plastics⁷

D 3361 Practice for Operating Light- and Water-Exposure Apparatus (Unfiltered Open-Flame Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products Using the Dew Cycle⁵

D 3424 Method for Evaluating the Lightfastness of Printed Matter⁵

D 3583 Methods of Testing Joint Sealant, Hot-Applied, Elastomeric Type for Portland Cement Concrete Pavements, or Joint Sealant, Hot-Applied Elastomeric, Jet-Fuel-Resistant Type, for Portland Cement Concrete Pavements⁸

D 3815 Practice for Accelerated Aging of Pressure Sensitive Tapes by Carbon-Arc Exposure Apparatus⁹

E 765 Practice for Evaluation of Cover Materials for Flat Plate Solar Collectors¹⁰

2.2 AATCC Standards:¹¹

Blue Wool Lightfastness

Test method 16A Colorfastness to Light, Carbon-Arc Lamp, Continuous Light

² Annual Book of ASTM Standards, Vol 04.07.

³ Annual Book of ASTM Standards, Vol 04.04.

⁴ Annual Book of ASTM Standards, Vol 09.01.

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ Annual Book of ASTM Standards, Vol 15.06.

⁷ Annual Book of ASTM Standards, Vol 08.01.

⁸ Annual Book of ASTM Standards, Vol 04.03.

⁹ Annual Book of ASTM Standards, Vol 15.09.

¹⁰ Annual Book of ASTM Standards, Vol 12.02.

¹¹ Available from the Secretary, American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

Laboratory _____	
Material _____	
G 23 Test Method No. _____	
Reference Standard Used: _____	
Other ASTM Test No. _____	Method No. _____
Exposure Apparatus: ASTM Type _____	
Mr. Model _____	Serial No. _____
Light Source: Enclosed _____	
Open Flame _____	
Age _____ h.	
Filters: Type _____	
Elapsed Exposure Time: _____ h	
Exposure Conditions: Program _____ h	
Light _____ min	Dark _____ min
Black-Panel Temperature _____ °C (°F)	
Dry Bulb Temperature _____ °C (°F)	
Relative Humidity _____ %	
Specimen Water Spray _____ min	_____ min
Rack Spray (when used) _____ min	_____ min
Specimen Spray Water Type: _____	
Specimen Spray Nozzle Type: Mfg. Designation _____	
Specimen Relocation Procedure During Exposure: _____	
Identify Properties to be Determined on Test Specimens and Identify Test Procedures or Methods Used for Property Measurement.	
Operator/Date: _____	
Supervisor/Date: _____	

FIG. 1 Report Form

2.3 ISO Standard.¹² ISO Gray Scale

3. Significance and Use

3.1 Several types of apparatus with different exposure conditions are available for use. No single operating procedure for light exposure apparatus with or without water can be specified as a direct simulation of natural exposure. This practice does not imply expressly or otherwise an accelerated weathering test.

3.2 Since natural environments vary with respect to geography, topography, and different exposure periods, it may be expected that the effects of natural exposure will vary accordingly. Furthermore, all materials are not affected equally by the same environment. Therefore, results obtained by use of this practice should not be represented as equivalent to those of any natural weathering test until the degree of quantitative correlation has been empirically established for the material in question.

3.3 Variations in results may be expected among instruments of different types and when operating conditions among similar type instruments vary within the accepted limits of this practice. Therefore, no reference should be made to results from use of this practice unless accompanied by the report form as specified in Fig. 1.

4. Apparatus¹³

4.1 The apparatus employed shall use one or two carbon-arc lamps as the source of radiation, and shall be one of the following types, or equivalent. The term "cycle" is defined as the total time for all exposure conditions (light, light plus water spray, dark periods) that are repeated.

NOTE 2—Several models of carbon-arc type exposure devices are no longer commercially available and should be considered obsolete. Exposures in these devices are not recommended and should only be made when mutually agreed upon by all interested parties.

4.1.1 *Type D*—Twin enclosed carbon-arc lamp apparatus, with a 762 mm (30 in.) diameter specimen drum rotating at 1 rpm, automatic control of temperature and cycle, and manually adjusted humidifier.

4.1.2 *Type DH*—Same as Type D, except with automatic control of the humidity.

4.1.3 *Type E*—Single open-flame sunshine carbon-arc lamp apparatus, with a 959 mm (37.75 in.) diameter specimen rack rotating at 1 rpm, automatic control of temperature and cycle, and manually adjusted humidifier.

4.1.4 *Type EH*—Same as Type E, except with automatic control of the humidity.

4.1.5 *Type H*—Single enclosed carbon-arc lamp apparatus, with a 508 mm (20 in.) diameter specimen rack, rotating at 1 rpm, automatic control of temperature and cycle, and manual regulation of the humidity.

4.1.6 *Type HH*—Same as Type H, except with automatic control of the humidity.

4.2 The apparatus should consist of a suitable frame within which is located a test chamber, and necessary compartments for housing control and regulating equipment.

4.3 Provision should be made for mounting or supporting the test specimens in a circular rack or drum that is rotated around the arc or arcs. This provides uniform distribution of the radiation on all specimens around the circumference of the rack. It does not, however, improve the distribution of the radiation along the vertical axis.

4.4 Adequate ventilation should be provided in the test chamber to prevent contamination of the specimens from combustion products of the arc.

4.5 The apparatus should include equipment necessary for measuring and controlling the following:

4.5.1 Arc current,

4.5.2 Arc voltage,

4.5.3 Black-panel temperature (Note 3),

¹² Available from American National Standards Institute, 1430 Broadway, New York, NY 10017.

¹³ Available from the Atlas Electric Devices Co., 4114 N. Ravenswood Ave., Chicago, IL 606132 and from Suga Test Instruments Co., Ltd. 4-14, Shinjuku 5-chome, Shinjuku-ku, Tokyo, 160, Japan.

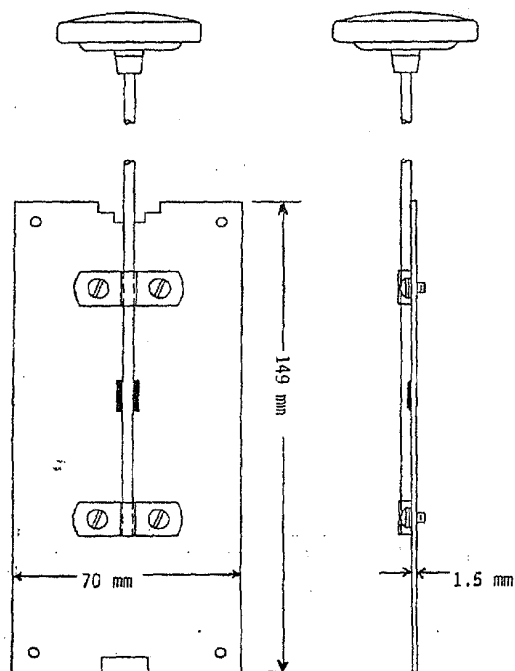


FIG. 2 Black Panel Thermometer Unit Using Dial Type Thermometer

4.5.4 Water-spray pressure,
4.5.5 Operating schedule or cycle,
4.5.6 Exposure time, and
4.5.7 Relative humidity (Types DH, EH, and HH only).
4.6 Types DH, EH, and HH apparatus are equipped with thermostatically actuated vaporizing units for adding moisture to the air as it passes through the conditioning chamber prior to its entry into the test chamber. Type H and some Types D, and E have manually regulated, electrically operated vaporizing units. Relative humidity of the air in the test chamber is calculated from the readings of the wet- and dry-bulb thermometers, either indicating or reporting, whose sensing portion is located in the air stream at its point of exit from the test chamber.

4.7 The black-panel thermometer unit should consist of a stainless steel panel 1 by 70 by 150 mm (.060 by 2.75 by 5.875 in.) to which is mechanically fastened a temperature sensing device. This device shall be capable of measuring temperature to a repeatability of $4 \pm 1^\circ\text{C}$ (see Figs. 2 and 3). The face of the panel with the temperature sensing device attached should be finished with two coats of baked-on black enamel selected for its resistance to light and water.

4.8 Detailed requirements and operating conditions of the apparatus are given in Table 1 and Figs. 4 to 6.

5. General Procedure

5.1 Prepare specimens of a suitable size and shape for mounting in the drum or rack of the apparatus in accordance

with the detailed requirements specified for the material to be tested.

5.2 Mount the test specimens, except those whose shape or other physical characteristics make it impractical, vertically both above and below the horizontal center line of the source of radiation. To assure that specimens receive the greatest uniformity of radiant exposure, reposition them vertically in a sequence which will provide each specimen equivalent exposure periods in each location. When the exposure interval does not exceed 24 h, each specimen should be located equidistant from the horizontal axis of the arc. For exposure intervals not exceeding 100 h, daily rotation of the specimens is recommended. Other methods of achieving uniform total irradiation may be employed if mutually agreed upon by concerned parties.

5.3 For enclosed arcs only, where physical characteristics do not permit suspension of specimens in a vertical position, expose them horizontally on a rack 165 mm (6.5 in.) below the horizontal center of the source or sources of radiation. Mount the specimens on a circular horizontal rack equipped with turntables, so that each specimen is rotated on its own axis as all of the specimens are rotated around the source or sources of radiation.

5.4 Temperature measurement and control should be based on the black-panel thermometer unit. Support the panel with the thermometer attached in the specimen drum or rack in the same manner as the test specimens so that it will be subjected to the same influences.

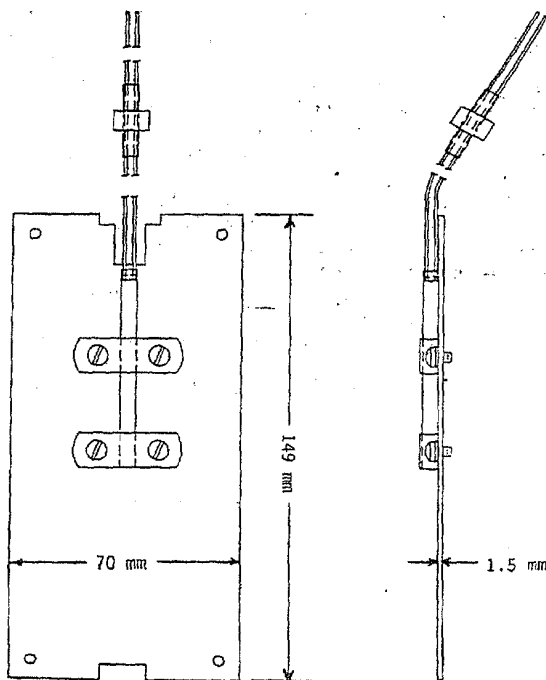


FIG. 3 Black Panel Thermometer Unit Using RTD Thermocouple Sensor

TABLE 1 Detail Requirements and Operating Conditions of Light- and Water-Exposure Apparatus

	Type ^A		
	D, DH	E, EH	H, HH
Line voltage, V: 208 to 250	X	X	X
Arc voltage, V: 120 to 145	X		X
48 to 52		X	
Arc current, A: 15 to 17, ac	X		X
58 to 62, ac		X	
12 to 14, dc	X		X
58 to 62, dc		X	
Carbon electrodes, upper: neutral cored or solid ^B	X		X
copper-coated sunshine		X	
Carbon electrodes, lower: neutral cored or solid ^B	X		X
copper-coated sunshine ^C			
Flat panels of optical, heat-resistant glass with light transmission properties similar to Fig. 7. #7740 Pyrex brand Filter panes ^E (3.0 mm) typically will transmit 1 % at 278 nm, 5 % at 285 nm and 50 % at 306 nm. #7058 Corex brand Filterpanes ^F (2.5 mm) typically transmits 1 % at 262 nm, 5 % at 262 nm and 50 % at 287 nm.		X	
Globe of optical, heat-resistant glass with nominal 1 % cut-off at 275 nm, with an increase in transmission to 91 % at 370 nm. ^D	X		X
Diameter of specimen rack or drum, mm (in.): 508 (20) 762 (30) 960 (37.7)	X		X
Speed of rotation of specimen drum or rack: 1 rpm	X	X	X
Automatic arc feed: Solenoid-operated	X		X
Motor-operated		X	
Spray (see figure indicated for arrangement, location, and capacity)	Fig. 4	Fig. 5	Fig. 6

^A "X" in column indicates application to that type of apparatus.

^B No. 70 Solid Carbon Electrodes and No. 20 Cored Carbon Electrodes or equivalent available from Atlas Electric Devices Co. or from Suga Test Instruments Co., Ltd.

^C No. 22 Copper-Coated Sunshine Carbon Electrodes and No. 15.5 Copper-Coated Sunshine Carbon Electrodes or equivalent available from Atlas Electric Devices Co. or from Suga Test Instruments Co., Ltd.

^D No. 9200-PX Globe or equivalent.

^E 7740 Pyrex is a tradename for a soda borosilicate glass.

^F 7058 Corex is a tradename for a potash lithia borosilicate glass.

5.4.1 Program the instrument to operate in a continuous light-on mode. Fill specimen rack with blanks and the black-panel thermometer. Operate in this mode while establishing the black-panel temperature according to the manufacturer's instructions. Allow the machine to come to operating temperature and equilibrate (allow 4 h of continuous running time and between 25 to 35 min after a spray cycle). Unless other temperatures and tolerances are specified in the applicable ASTM test method or detailed material specifications, the black-panel temperature shall be $63 \pm 2.5^\circ\text{C}$ ($145 \pm 5^\circ\text{F}$) (Note 3).

5.4.2 Compare the black-panel temperature with the specification. If the black-panel temperature is within 2.5°C or 5°F of specification, no adjustment is necessary. If the observed temperature difference is greater than 2.5°C or 5°F , adjust the temperature controller and recheck the black-panel temperature during the next cycle. It is recommended that this procedure be performed after every 200 h of machine operation.

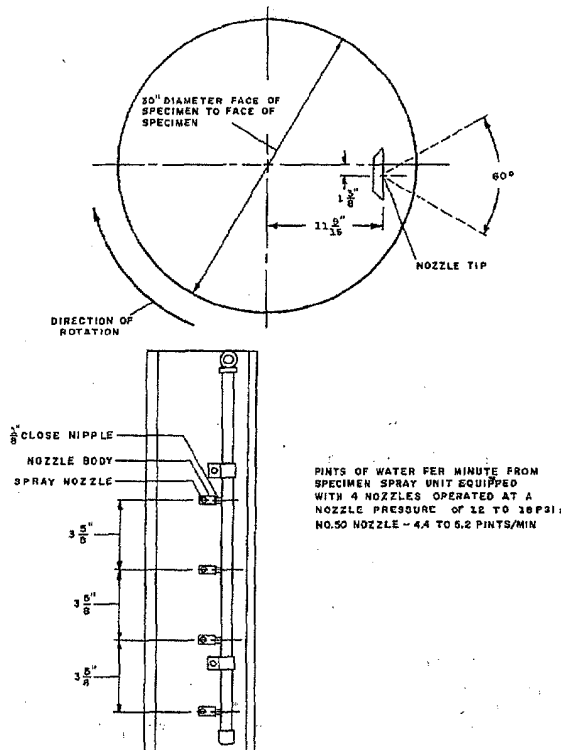
NOTE 3—Where desired, other black-panel temperatures may be employed provided they are specifically noted in the report of test results. Instructions for adjusting the intensity of the arc and the fading rate are supplied with the apparatus or are available upon request from the manufacturer. Consideration should always be given to installing the instrument in a room where the temperature and humidity conditions are controlled. The magnitude of the effects due to variation in the air

supply can only be partially determined when the intake air is not controlled.

5.5 The water from the specimen spray should strike the test specimens in the form of a fine spray equally distributed over the test specimens. Unless otherwise specified in the applicable ASTM test method or detailed material specification, the water pressure, number, and type of nozzles should be in accordance with the detailed requirements for the various types of apparatus as indicated in Figs. 4 to 6. The pH of the water shall be 6.0 to 8.0, contain less than 1 ppm solids and leave no objectional deposits or stain on the specimens. The presence of very low levels of silicates in spray water has been shown to leave deposits on some specimens. It is recommended that the temperature of the water should be $16 \pm 5^\circ\text{C}$ ($60 \pm 9^\circ\text{F}$). Recirculation is not recommended and must not be done unless the recirculated water is treated to meet the above requirements.

5.5.1 Set the cycle control unit cam until the machine is in a spray cycle. Observe the spray manifold. All spray heads should spray a stream of mist or water which reaches the panel surface. If any of the spray heads are not satisfactory, remove the spray manifold and clean each spray head. Install the manifold and recheck the spray operation.

5.6 The flat glass filter panes used in Types E or EH devices cannot be used for more than 2000 h. Monitor age and position of the filter panes so that the oldest can be



PINTS OF WATER PER MINUTE FROM SPECIMEN SPRAY UNIT EQUIPPED WITH 4 NOZZLES OPERATED AT A NOZZLE PRESSURE OF 12 TO 18 PSI: NO.50 NOZZLE - 4.4 TO 5.2 PINTS/MIN

Metric Equivalents

mm	3.3	34.9	92.1	101.6	293.2	762	83 to 124 kPa	2.5 to 2.9 dm ³
in.	1/8	1 3/8	3 5/8	4	11 5/16	30	12 to 18 psi	4.4 to 5.2 pt

FIG. 4 Specimen Spray Arrangement for Types D and DH Apparatus

removed every 250 h. Filter globes used in type D or DH devices cannot be used for more than 2000 h. Replace the oldest of the filter globes every 1000 h. Replace the filter globe in Type H or HH devices after 2000 h of use or when pronounced discoloration or milkiness develops. Clean filters each day by wiping with a clean, dry non-abrasive cloth or towel. Filters can also be cleaned by washing with water, or a solution of detergent and water.

5.7 Unless otherwise specified in the applicable ASTM test method or detailed material specifications, operate Types E and EH apparatus with the filters in place and with the carbon electrodes specified in Table 1. If operated without filters or with other types of carbon electrodes, state this in the report of test results.

METHOD 1—CONTINUOUS EXPOSURE TO LIGHT AND INTERMITTENT EXPOSURE TO WATER SPRAY

6. Apparatus

6.1 The apparatus may be any of the recommended type equipped with water spray nozzles.

6.2 Program the instrument for continuous light and intermittent water spray according to the manufacturer's instructions. Since there are numerous light and water spray

cycles which can be used, the cycle program selected must be by mutual agreement among the interested parties. Historical convention has established a cycle of 102 min of light followed by 18 min of light and water spray. This cycle permits the attainment of the maximum black-panel temperature during the light only portion of the exposure.

6.2.1 When testing the effects of photodegradation only, the water spray may be omitted (refer to Method 3).

6.3 In Types DH, EH and HH apparatus, dry and wet bulb temperature controls, humidifier and immersion heater controls should be adjusted as specified.

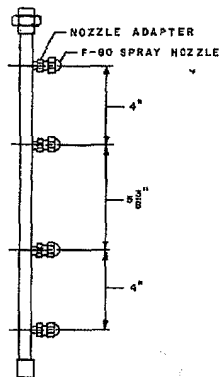
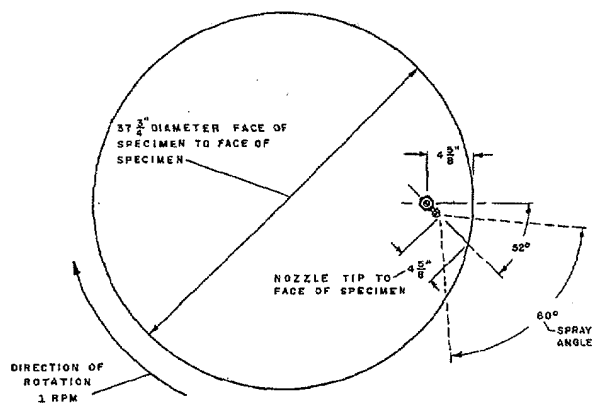
6.4 In Type H and in Types D and E with humidifier, humidity may be adjusted but not controlled.

METHOD 2—ALTERNATIVE EXPOSURE TO LIGHT AND DARKNESS AND INTERMITTENT EXPOSURE TO WATER SPRAY

7. Apparatus

7.1 The apparatus shall be Type DH, EH and HH apparatus with automatic humidity control and water spray nozzles.

7.2 Operation during the light on period should be as described in Section 5.



4 NOZZLES EMPLOYED
1 TYPE EMPLOYED-F-80

PINTS OF WATER PER MINUTE FOR
SPECIMEN SPRAY UNIT EQUIPPED WITH
4 NOZZLES OPERATED AT A NOZZLE
PRESSURE OF 18 TO 25 PSI: 46 TO 64
PINTS PER MINUTE.

Metric Equivalents

mm	101.6	117.5	136.5	266.7	959	124 to 172 KPa	0.26 to 0.36 dm ³
in.	4	4½	5½	10½	37¾	18 to 25 psi	0.46 to 0.64 pt

FIG. 5 Specimen Spray Arrangement for Types E and EH Single Open-Flame Sunshine Carbon-Arc Lamp Apparatus

7.2.1 Separate controls for temperatures and humidification may be adjusted during the dark period for automatic control as the cycle alternates from light to dark. In Type EH apparatus, a spray rack to cool the specimens by wetting the unexposed back surface can result in development of condensation on the exposed specimen surface during the dark interval.

7.3 All Types DH, EH, H, HH and some Type D, and E may be programmed to operate with alternating light and dark intervals without control of relative humidity.

METHOD 3—CONTINUOUS EXPOSURE TO LIGHT WITHOUT WATER SPRAY

8. Apparatus

8.1 The apparatus shall be Type D or DH, E or EH, H or HH light-exposure devices programmed for continuous light only, according to the manufacturer's instructions.

8.2 Adjust the controls on the apparatus so that the black panel temperature is $63 \pm 2.5^{\circ}\text{C}$ ($145 \pm 5^{\circ}\text{F}$) (Note 3), and for a relative humidity of $30 \pm 5\%$ in devices with automatic humidity control.

8.3 Expose the materials to be tested as determined by mutual agreement among the concerned parties or, when not otherwise specified, in accordance with one of the following:

8.3.1 Versus One AATCC Blue Wool Lightfastness Standard—Expose the test specimen and any mutually agreed upon AATCC Blue Wool Lightfastness Standard partially masked until the difference between its masked and unmasked portion exhibits a color change equal to Step 4 of the ISO Gray Scale. Report the results by any mutually agreed-upon method of measuring change in the test specimen.

8.3.2 Versus Set of AATCC Blue Wool Lightfastness Standards—Expose the test specimen and a set of AATCC Blue Wool Lightfastness Standards partially masked until a maximum permissible amount of change, as determined by specified or mutually agreed-upon method of measurement, occurs in the test specimen. Assign it a classification number equal to that of the numbered standard that most nearly exhibits a change in color equal to Step 4 on the ISO Gray Scale. Where one standard shows a color change greater than Step 4 and the next higher number standard shows less than a Step 4 color change, an intermediate or half-grade rating may be used.

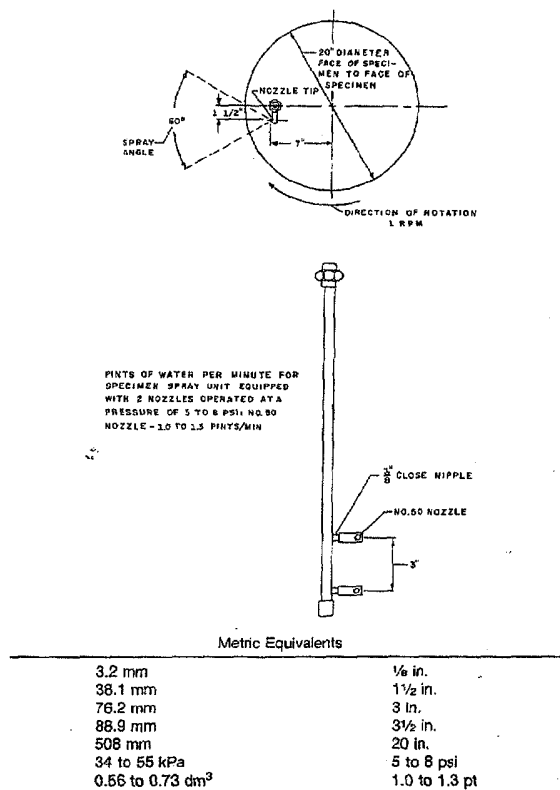


FIG. 6 Specimen Spray Arrangement for Types H and HH Single Enclosed Carbon-Arc Lamp Apparatus

8.3.3 *Versus Other Standard Sample*—Expose the test specimen and any mutually agreed-upon standard sample until either shows an agreed-upon amount of change when measured by any mutually agreed-upon method. Report the results on the basis of a comparison of the specimen with the standard sample.

METHOD 4—ALTERNATE EXPOSURE TO LIGHT AND DARKNESS WITHOUT WATER SPRAY

9. Apparatus

9.1 The apparatus shall be Type H or HH programmed to a predetermined cycle of light and darkness by turning off

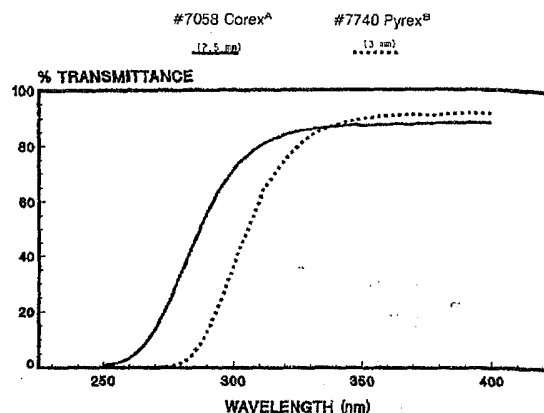


FIG. 7 Representative UV Transmission Curves for 2.5 mm and 3 mm Filter Glasses^A

^A # 7058 Corex is a tradename for a potash lithia borosilicate glass.

^B #7740 Pyrex is a tradename for a soda borosilicate glass.

the carbon arc.

9.2 The procedure for Method 4 is the same as specified for Method 3 except that the following light-dark interval shall replace the continuous light condition of Method 3.

9.2.1 Unless otherwise specified by the detailed requirements for the type of material being tested, adjust the controls of the apparatus so that it produces a cycle of 1 h of darkness followed by 3.8 h of light. Repeat this cycle for the duration of the test. During the light-on period the black-panel temperature, at equilibrium, shall be $63 \pm 2.5^\circ\text{C}$ ($145 \pm 5^\circ\text{F}$). During the period while the light is off, the black-panel temperature will progressively drop until it is in equilibrium with the air in the test chamber. The relative humidity of the air at the face of the test specimens will progressively rise during the light-off period until equilibrium is reached.

10. Report

10.1 The report shall include the following:

- 10.1.1 Type and model of exposure device,
- 10.1.2 Type of light source,
- 10.1.3 Type, thickness, and age of filters,
- 10.1.4 Elapsed exposure time,
- 10.1.5 Light and dark-water-humidity cycle employed,
- 10.1.6 Operating black-panel temperature,
- 10.1.7 Operating relative humidity,
- 10.1.8 Type of spray water,
- 10.1.9 Type of spray nozzle, and
- 10.1.10 Specimen relocation procedure.

APPENDIX

(Nonmandatory Information)

X1. OBSOLETE APPARATUS

X1.1 The following types of apparatus are no longer commercially available and should be considered obsolete. Although existing units may be operated in accordance with

Methods 1 and 3 of this practice, results may not be comparable with those obtained in superseding models.

X1.1.1 *Type A*—Single enclosed carbon-arc lamp apparatus*

with a 762 mm (30 in.) diameter specimen drum rotating at 1 rpm, automatic control of temperature and cycle, and no automatic control of the humidity.

X1.1.2 *Type AH*—Same as Type A, except with automatic control of humidity.

X1.1.3 *Type B*—Single enclosed carbon-arc lamp apparatus, 3 RPH, 762 mm (30 in.) diameter specimen drum, automatic temperature control, no automatic humidity con-

trol. Last manufactured in 1955.

X1.1.4 *Type C*—Same as Type B but without automatic temperature control. Last manufactured in 1960.

X1.1.5 *Type F*—Single open flame carbon arc lamp apparatus, 0.5 rph, 960 mm (37.75 in.) diameter specimen rack with vertical specimen mounting. No automatic control of temperature or humidity. Last manufactured in 1950.

X1.1.6 *Type G*—Same as Type F but with inclined specimen rack.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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Standard Practice for Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials¹

This standard is issued under the fixed designation G 26; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the basic principles and operating procedure for water- or light-exposure apparatus, or both, employing a xenon-arc light source.

NOTE 1—This practice combines the practices previously referred to as G 26 and G 27. Practice G 27, for Operating Xenon-Arc Type Apparatus for Light Exposure of Nonmetallic Materials², has been discontinued since it is now covered in this edition of G 26 under Test Methods C and D.

1.2 This practice does not specify the exposure conditions best suited for the material to be tested, but is limited to the method of obtaining, measuring, and controlling the conditions and procedures of the exposure. Sample preparation, test conditions, and evaluation of results are covered in ASTM methods or specifications for specific materials.

1.3 This practice includes four test methods:

1.3.1 *Test Method 1*—Continuous exposure to light and intermittent exposure to water spray.

1.3.2 *Test Method 2*—Alternate exposure to light and darkness and intermittent exposure to water spray.

1.3.3 *Test Method 3*—Continuous exposure to light without water spray. Exposure conditions are characteristic of those specified by AATCC Test Method 16E 1976.

1.3.4 *Test Method 4*—Alternate exposure to light and darkness without water spray. Exposure conditions characteristic of those natural conditions experienced when exposing are in accordance with Practice G 24.

1.4 *This practice may involve hazardous operations and equipment. This practice does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this practice to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 The values stated in SI units are to be regarded as the standard. The inch-pound equivalents of the SI units may be approximate.

2. Referenced Documents

2.1 ASTM Standard:

G 24 Practice for Conducting Natural Light Exposures Under Glass²

2.2 AATCC Standard:

Method 16E 1976 Colorfastness to Light, Water, and Xenon-Arc Lamp Continuous Light³

2.3 CIE Standard:

No. 20 Recommendations for the Integrated Irradiance and the Spectral Distribution of Simulated Solar Radiation for Testing Purposes⁴

2.4 ISO Standard:

Gray Scale

3. Significance and Use

3.1 Several types of apparatus with different exposure conditions are available for use. No single operating procedure for light-exposure apparatus with and without water can be specified as a direct simulation of natural exposure. This practice does not imply expressly or otherwise an accelerated weathering test.

3.2 Since the natural environment varies with respect to time, geography, and topography, it may be expected that the effects of natural exposure will vary accordingly. All materials are not affected equally by the same environment. Results obtained by use of this practice should not be represented as equivalent to those of any natural weathering test until the degree of quantitative correlation has been established for the material in question.

3.3 Variations in results may be expected among instruments of different types or when operating conditions among similar type instruments vary within the accepted limits of this practice. Therefore, no reference should be made to results from use of this practice unless accompanied by the report form shown in Fig. 1 or unless otherwise specified in referenced procedure.

4. Apparatus

4.1 Water-Cooled Type:⁵

4.1.1 The apparatus employed should utilize a water-cooled xenon-arc lamp as the source of radiation and should be one of the following general types, or their equivalent:

4.1.1.1 *Type A*—An exposure apparatus in which the source of radiant energy shall be a water-cooled xenon-arc lamp vertically located at the central axis of either a 508-mm (20-in.) diameter vertical specimen rack, or of a 648-mm (25.5-in.) diameter inclined rack. Means shall be provided for automatic programming of temperature and cycles.

¹ This practice is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.03 on Simulated and Controlled Environmental Tests.

Current edition approved Jan. 26, 1990. Published March 1990. Originally published as E 239 - 64 T. Redesignated G 26 in 1970. Last previous edition G 26 - 88¹.

² Annual Book of ASTM Standards, Vol 14.02.

³ Available from The Secretary, American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

⁴ Available from Secretary, U.S. National Committee, CIE, National Institute of Standards and Technology, Gaithersburg, MD 20899.

⁵ Available from Atlas Electric Devices Co., 4114 N. Ravenswood Ave., Chicago, IL 60613.

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Laboratory _____
Material _____
G 26 Test Method No. _____
Reference Standard Used: _____
Other ASTM Test No. _____ Method No. _____
Exposure Apparatus: ASTM Type _____
Mfr. Model _____ Serial No. _____
Light Source: Water-Cooled _____ W, Air-Cooled _____ W
Method Used to Regulate Wattage to Lamp _____
Optical Filters: Type _____ Age _____ h
Spectral Irradiance: _____ W/m² at _____ nm
Irradiation Units: _____ kJ/m² How Monitored: _____
Elapsed Exposure Time: _____ h
Exposure Conditions: Program _____ h
Light _____ min Dark _____ min
Black-Panel Temperature _____ °C (°F)
Air Temperature _____ °C (°F)
Dry Bulb Temperature _____ °C (°F) _____ °C (°F)
Relative Humidity _____ %
Specimen Water Spray _____ min cycle _____ min cycle
Specimen Nonspray _____ min cycle _____ min cycle
Specimen Spray Water Type: _____
Specimen Spray Nozzle Type: Mfg. Designation _____
Specimen Relocation Procedure During Exposure: _____
Identify properties to be determined on test specimens and identify test procedures or methods used for property measurement.
Operator/Date: _____
Supervisor/Date: _____
Company: _____

FIG. 1 Report Form

Means shall be provided for adjustment of relative humidity. The specimen rack shall rotate at 1 ± 0.1 rpm.

NOTE 2—In the commercial descriptions of the four types, the term "cycle" is defined as each time interval of light, darkness, and water spray that is specified differently in accordance with the different testing methods.

4.1.1.2 Type AH—The exposure apparatus shall be identical to Type A except it shall have automatic humidity control.

4.1.1.3 Type B—An exposure apparatus in which the source of radiant energy shall be a water-cooled xenon-arc vertically located at the central axis of a 960-mm (37.75-in.) diameter specimen rack. Means shall be provided for automatic programming of temperature and cycles. Means may be provided for adjustment of humidity. The specimen rack shall rotate at 1 ± 0.1 rpm.

4.1.1.4 Type BH—The exposure apparatus shall be similar to Type B except it shall have automatic humidity control.

4.1.2 The xenon-arcs employed shall be of the "long arc" water-cooled type operated through suitable reactance transformers and electrical equipment from a 50 or 60-Hz power supply. They shall employ cylindrical inner and outer optical filters to direct the flow of cooling water and to simulate a designed spectral energy distribution.

4.1.2.1 For the purpose of this practice, the xenon-arc lamp shall consist of a quartz xenon burner tube and one of the following optical filter combinations:^{6,7}

(a) Borosilicate glass inner and outer optical filter to simulate the spectral power distribution (SPD) of natural daylight throughout the actinic region.

(b) Infrared absorbing glass inner optical filter with quartz outer optical filter to simulate the SPD of natural daylight (300 to 1000 nm).

(c) Borosilicate glass inner optical filter with soda lime glass outer optical filter to simulate the SPD of natural daylight (actinic wavelengths) filtered through window glass.

(d) Infrared absorbing glass inner and outer optical filter to simulate the SPD of natural daylight (310 to 1000 nm) filtered through window glass.

(e) Quartz inner and outer optical filter to approximate sunlight intensities unfiltered by the earth's atmosphere.⁶

4.1.2.2 To prevent loss in levels of irradiance due to excessive solarization and to prevent possible breakage caused by stresses in optical filters exposed to high-intensity UV energy, inner optical filters⁸ shall be replaced periodically. Suggested intervals are 300 and 400 h for Types A and B, respectively. The outer optical filter shall be replaced after 1500 and 2000 h for Types A and B, respectively.

4.1.3 Distilled or deionized water should be recirculated past the burner at a flow rate sufficient to remove excess heat. Passing water through a cartridge demineralizer installed in the recirculation line just ahead of the lamp minimizes contamination of the quartz envelope of the burner. A heat-exchange unit should be used to cool the recirculated lamp water.

4.1.4 Since xenon-arc lamps, like all gas discharge lamps, will have a progressive drop in radiation output with continued use, and since optical filters will change in their

⁶ Norton, Kuntke, and Connor, *Canadian Textile Journal*, CTJOA, May 1969.

⁷ Corning 7740 Pyrex® (borosilicate), Corning 4601 IR absorbing visible transmitting, Kimble R6 soda lime glass tubing, and General Electric GA-204 quartz tubing of proper dimensions have been found suitable for use as xenon-arc optical filters. Available from Atlas Electric Devices Co., 4114 N. Ravenswood Ave., Chicago, IL 60613.

⁸ Kishii, T., and Ooka, K., *Symposium on Coloured Glasses*, International Comm. on Glass of Czech. Sci. & Tech. Soc. of the Silicate Industry, 1967.

transmission characteristics, provision shall be made in the apparatus (automatically or manually) for progressively increasing the wattage of the lamp to minimize changes in the intensity of the radiation at the face of the sample. The greatest change in both the xenon burner tube and optical filters occurs in the first 20 h of use. For this reason, burners and optical filters preaged by the instrument manufacturer are recommended for critical testing when a more rapidly changing rate of intensity is undesirable. For routine testing on a comparative basis, the 20 h of preaging may be omitted.

4.1.5 Many nonmetallic materials are selectively absorbing. The energy that a molecule absorbs depends upon the wavelength of the incident radiation.⁹ It is desirable to monitor the level of irradiance of the photochemically effective wavelengths if intercomparisons are to be made among samples not exposed simultaneously. Samples may then be exposed to known amounts of irradiation, the time integral of irradiance.

4.1.5.1 Where radiometers capable of monitoring discrete portions of a continuous spectrum are available, exposure to a mutually agreed-upon level of irradiation may be specified as the exposure interval in place of a time interval.

(a) When using the optical filter combination described in 4.1.2.1 (a), the suggested minimum spectral irradiance levels are:

- .2 W/m²/nm band at 320 nm
- .35 W/m²/nm band at 340 nm
- .5 W/m²/nm band at 380 nm
- .75 W/m²/nm band at 420 nm

(b) When using the optical filter combination described in 4.1.2.1 (c), the suggested minimum spectral irradiance levels are:

- .2 W/m²/nm band at 340 nm
- .45 W/m²/nm band at 380 nm
- .7 W/m²/nm band at 420 nm

(c) Operating at the suggested minimum levels approximates the average daily solar irradiance under ideal conditions. It is about half of the maximum values for total irradiance on a horizontal plane when the sun is at 90° altitude as reported in CIE No. 20. When irradiance is monitored and periodic manual adjustment of wattage is made to compensate for changes of intensity, no lamp assembly should be used that cannot maintain the minimum level of irradiance within a 10 % tolerance at the monitored wavelength. Irradiation expressed as joules per square metre is the product of irradiance × exposure time in seconds.

4.1.5.2 Xenon-arc type equipment not having a radiometer shall be operated with periodic increases in wattage to minimize any drift in levels of irradiation. Such intervals shall be established by the parties concerned or follow the suggested schedule below in 4.1.5.2 (a).

NOTE 3—The use of suggested wattage steps does not imply that irradiance will be maintained at levels equivalent to those obtained when employing a light monitor. They are intended as a guide to minimize the reduction of UV intensity with maximum lamp longevity when using either Test Method A or C for 2.5, 6, or 6.5-kW lamps, respectively.

⁹ Hawkins, W.L., ed., "Polymer Stabilization," Wiley-Interscience, 1972. p. 166.

(a) The following are the suggested wattage settings for each exposure interval of a 2500-W xenon burner tube based upon the average performance of xenon burner tubes with borosilicate filters:

Time, h	Power, W
0 to 20	1600
20 to 200	1800
200 to 400	2000
400 to 600	2250
600 to 800	2500
800 to 1000	2600
1000 to 1200	2800
1200 to 1500	3000

(b) The following are the suggested wattage settings for each exposure interval of a 6000-W xenon burner tube based upon the performance of xenon burner tubes with borosilicate filters in Test Method A:

Time, h	Power, W
0 to 20	min
20 to 100	4750
100 to 250	5000
250 to 400	5250
400 to 550	5500
550 to 700	6000
700 to 850	6500
850 to 1000	7000

(c) The following are the suggested wattage settings for each exposure interval of a 6500-W xenon burner tube based upon the average performance of xenon burner tubes with borosilicate filters in Test Method A:

Time, h	Power, W
0 to 20	min
20 to 100	5500
100 to 200	6000
200 to 500	6200
500 to 1000	6500
1000 to 1500	7000
1500 to 2000	7500
2000 and over	8500

4.1.6 Specimens should be mounted on a rack rotating about the lamp at a distance such that the location of each specimen assures that the irradiance incident on its surface does not vary by more than 5 % from the average. Figure 4 illustrates a rack meeting this condition.

4.1.7 Any apparatus that does not comply with the condition of 4.1.6 shall not have a variation in irradiance exceeding 10 % of the average, and may require specimen rearrangement during exposure, in accordance with the test procedure, in order to minimize the variability in radiant exposure.

4.1.8 Testing temperatures should be measured and regulated on the basis of a black panel thermometer unit mounted on the specimen rack so that the face of the unit is in the same relative position and is subjected to the same influences as the test specimens. The black panel thermometer unit should consist of a stainless steel panel about 70 by 50 by 0.95 mm (2.750 by 5.875 by 0.0375 in.), to which a stainless steel bimetallic dial-type thermometer is mechanically fastened. This thermometer should have a stem approximately 4 mm (0.16 in.) in diameter with a 44.5-mm (1.75-in.) dial. The sensitive portion extending about 38 mm (1.5 in.) from the end of the stem should be located in the center of the panel approximately 64 mm (2.5 in.) from the top and approximately 48 mm (1.875 in.) from the bottom

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of the panel. The face of the panel with the thermometer stem attached should be finished with a baked-on black infrared-absorbing coating having good resistance to light.

4.1.8.1 A thermocouple or resistance bulb thermometer mounted at the center face of the black panel, which provides temperature values equivalent to the dial thermometer, may be substituted.

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4.1.9 A blower unit in the base of the apparatus should provide a flow of air through the test chamber and over the test specimens. Control of specimen and black-panel temperature should be accomplished by thermostatic control of the temperature of the constant volume of air from the blower. Black-panel temperatures should be read through the window in the test chamber door without opening the door.

4.1.10 Apparatus operated as a light- and water-exposure test shall be equipped with a specimen spray unit as illustrated in Figs. 2 and 3. All components of the specimen spray unit should be fabricated from stainless steel, plastic, or other material that does not contaminate the water. Apparatus operated only as a light-exposure test are not required to have the specimen spray unit or, if present, it should not be used.

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4.1.11 Types A, AH, BH, and some Type B apparatus are equipped with an electrically operated vaporizing unit for adding moisture to the air as it passes through the conditioning chamber in the base section prior to its entry into the test chamber of the apparatus.

4.1.11.1 In Types A and B, the vaporizing unit, when manually turned on, may operate continuously while the apparatus is in operation. Type A and some Type B apparatus may be programmed to operate the vaporizer during a dark cycle only. The temperature of the water, with which the vaporizer is supplied, is not controlled. In Type B apparatus not supplied with a vaporizing unit, the relative humidity within the test chamber is governed by evaporation of water in the bottom of the chamber and from water emitted through the specimen spray unit.

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4.1.11.2 In Types AH and BH apparatus, operation of the vaporizer is controlled automatically by a wet-bulb thermostat. The temperature of the water supplied to the vaporizer is regulated automatically by thermostatically-actuated electric immersion heaters. Control automatically shifts between two separate sets of thermostats as the arc lamp is turned on and off by the program control unit.

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4.1.12 In Types A, B, AH, and BH apparatus containing vaporizing units, relative humidity is determined from wet- and dry-bulb thermometers located in the air stream at the vaporizer's point of exit from the test chamber.

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4.1.12.1 Determine the relative humidity for Type B apparatus not equipped with a vaporizing unit from wet- and dry-bulb thermometers mounted in holders on the specimen rack so that their sensitive portions are in the same relative position as the face of the test specimen but shielded from the radiation.

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4.1.12.2 Any apparatus with a vaporizing unit operated with the unit and immersion heaters turned off will provide essentially the same conditions of relative humidity as are produced in Type B apparatus without a vaporizing unit.

4.1.13 The apparatus shall include means for measuring the following:

- 4.1.13.1 Wattage of the xenon-arc lamp,
- 4.1.13.2 Irradiance at the specimen rack,

- 4.1.13.3 Black-panel temperature,
- 4.1.13.4 Dry-bulb temperature (test chamber),
- 4.1.13.5 Wet-bulb temperature (test chamber),
- 4.1.13.6 Exposure interval, and
- 4.1.13.7 Water spray pressure when applicable.
- 4.1.14 Where specified, the apparatus shall include means for regulating or controlling the following:
 - 4.1.14.1 Wattage of the xenon lamp,
 - 4.1.14.2 Irradiance or irradiation, or both, at the specimen rack,
 - 4.1.14.3 Temperature (test chamber),
 - 4.1.14.4 Relative humidity (test chamber),
 - 4.1.14.5 Light-dark-spray-humidity-temperature cycles, and
 - 4.1.14.6 Water spray pressure when applicable.

4.2 Air-Cooled Type.¹⁰

4.2.1 The apparatus employed shall use an air-cooled xenon-arc lamp as the source of radiation and should be one of the following types, or their equivalent:

4.2.1.1 Type C—Air-cooled xenon-arc apparatus, 1500 W, 158-mm (6.2-in.) diameter specimen rack, with automatic programming of cycles and humidity. The specimen rack should rotate at 5.2 ± 0.1 rpm.

4.2.1.2 Type D—Air-cooled xenon-arc apparatus, 4500 W, 360-mm (14.2-in.) diameter specimen rack, with automatic programming of temperature, cycles, and humidity. The specimen rack should rotate at 3.7 ± 0.1 rpm.

4.2.1.3 Type E—Air-cooled xenon-arc apparatus, three lamps operating simultaneously at 4500 W each, 610-mm (24.1-in.) diameter specimen rack, with automatic programming of temperature, cycles, and humidity. The specimen rack should rotate at 2.0 ± 0.1 rpm.

4.2.2 The xenon arcs employed in Types C and D should be of the medium-pressure, air-cooled type and one or more optical filters shall be fitted between the light source and the samples to filter out undesired wavelengths of radiation. A combination of optical filters recommended by the manufacturer shall be used to simulate (1) sunlight in the open, (2) sunlight behind window glass, or (3) any other simulated solar irradiation condition desired.

4.2.3 All gas discharge lamps have a progressive drop in radiant output with continued use. For Types C and D, it is recommended to exchange the IR-optical filters for new ones after 3500 h of use. Answering the purpose, not all seven IR-optical filters of the filter lantern should be exchanged at the same time but each should be exchanged after it has been running for 3500 h. For Type E which has three lamps, one lamp should be replaced every 500 hours to minimize the overall drop in radiant output. For Type E, the three partial preaged filters should be cleaned monthly and replaced after five years of normal usage.

4.2.4 Specimen holders should rotate around the arc, describing a cylindrical surface, so that specimens may face, or be turned opposite to, the arc. No part of the specimens shall be above or below the ends of the arc. Repositioning of specimens in upper, center, or lower positions can improve the uniformity of intensity of the exposure.

¹⁰ Available from DSET Labs, Box 1850, Black Canyon Stage 1, Phoenix, AZ 85027.

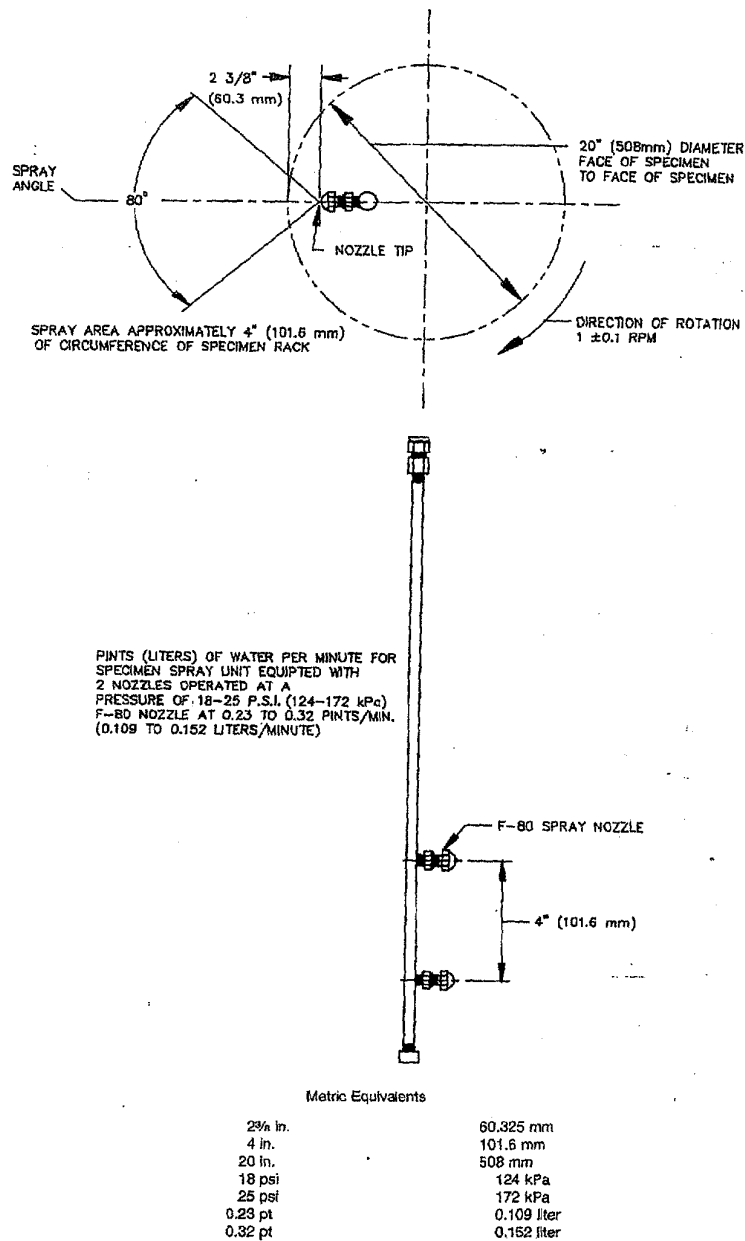


FIG. 2 Specimen Spray Arrangement for Type A and AH Apparatus

4.2.5 Testing temperatures should be measured and regulated on the basis of a black-panel thermometer unit that is mounted so that the face of the unit is in the same relative position and is subjected to the same influences as the test

specimens. The black-panel thermometer unit should consist of a stainless steel panel approximately 48 by 200 by 1 mm (1.9 by 8.0 by 0.04 in.), to which a stainless steel bimetallic dial-type thermometer is mechanically fastened. The temper-

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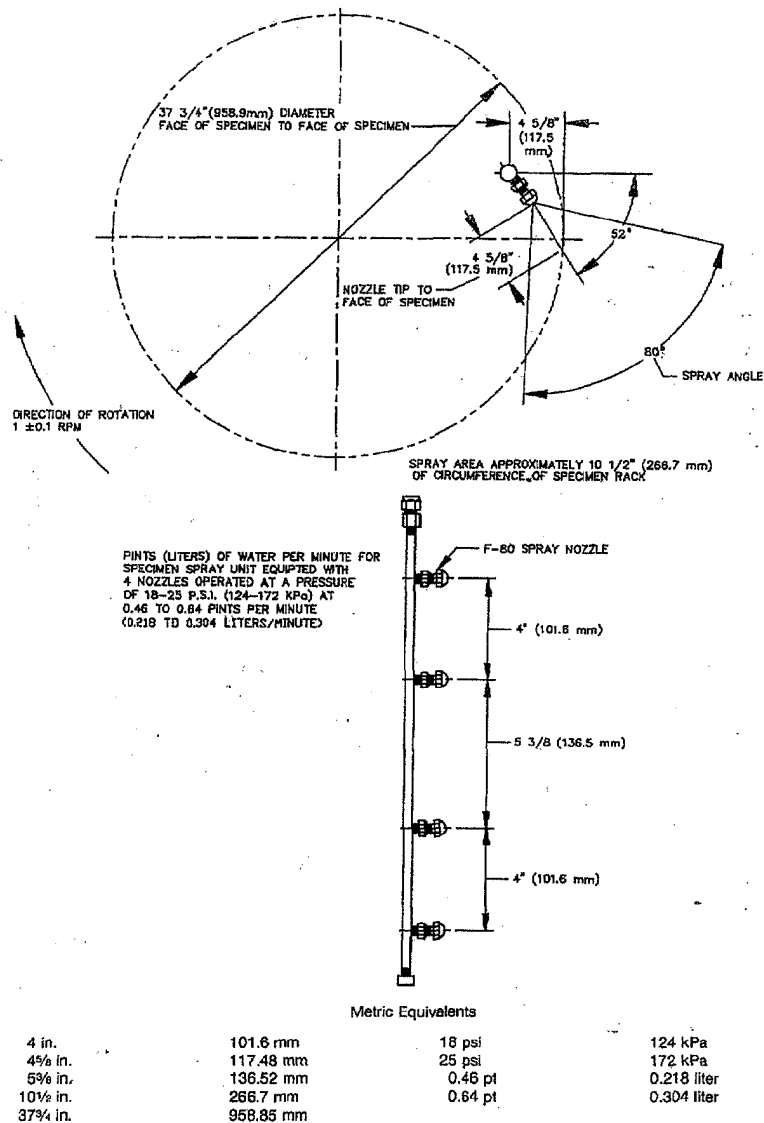


FIG. 3 Specimen Spray Arrangement for Type B and BH Apparatus

ature at the center of the panel should be sensed by the thermometer. The face of the panel should be finished with a baked-on black glossy enamel having good stability to light.

4.2.6 A blower unit in the base of the apparatus shall provide a flow of air through the test chamber and over the test specimens. Control of the specimen and the black-panel temperature should be accomplished by thermostatic control of the temperature of the constant volume of air from the blower. Black-panel temperature should be read through the window in the test chamber door without opening the door.

4.2.7 The apparatus should be equipped with a system to

spray the specimens uniformly with water. This system should be of stainless steel, plastic, or other material that does not react with or contaminate the water employed.

4.2.8 Relative humidity in the test chamber should be measured and controlled by a contact hygrometer. Water should be vaporized and diffused to enrich the air with moisture and produce the required humidity.

4.2.9 The apparatus shall include equipment necessary for measuring and controlling the same parameters as listed for water-cooled xenon arcs.

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5. General Procedure

5.1 Check to be sure the apparatus is operating properly at the start of each test. Check the lamp operation at 100-h intervals to be sure the burner tube and optical filters are clean and that they have not exceeded the maximum recommended period of use.

5.2 Program the instrument to operate in the continuous light-on mode without water spray. Fill the specimen rack with blanks and the black-panel thermometer. Operate the instrument in this mode while regulating the chamber dry-bulb temperature to provide the desired black-panel temperature of $63 \pm 3^\circ\text{C}$ ($145 \pm 5^\circ\text{F}$).

5.3 When the chamber dry-bulb temperature has been regulated, adjust instruments with automatic humidity control to the desired relative humidity (Types C, D, and E) or to the chamber wet-bulb temperature (Types A and B) at the level that will ensure the desired relative humidity.

5.4 In Types A, B, C, D, and E apparatus, use the type and number of spray nozzles (Figs. 2 and 3) recommended by the manufacturer and operate at a pressure of 124 to 172 kPa (18 to 25 psi), measured at the nozzle unless otherwise recommended. The water shall have a pH of 6.0 to 8.0, contain less than 20 ppm solids, and leave no deposit or stain on the specimens after continued exposure in the apparatus. The temperature of the water should be $16 \pm 5^\circ\text{C}$ ($60.8 \pm 9^\circ\text{F}$), and recirculation of the water shall not be permitted unless the recirculated water meets the above requirements. Pass the specimens through the spray once in each minute or revolution of the rack during the spray cycle. Allow the water to strike the test specimens in the form of a fine spray equally distributed over the specimens.

5.5 The temperature of the rack spray water should be sufficiently low to reduce the specimen temperature below the dewpoint when the specimen is continuously sprayed during a dark cycle.

5.6 Reference Standards:

5.6.1 AATCC Blue Wool Lightfastness Standards.³

NOTE 4—The eight standards L-2 to L-9 have been made by blending wool dyed with the fugitive dye Erio Chrome Azurole B and wool dyed with the fast dye Indigosol Blue AGG in different proportions. The blended wools were spun into yarns and the yarns woven into cloths. Each standard is approximately twice as fast as the next lower numbered standard. It has been found that when new lots of the standards are produced, the amounts of the dyes required and the properties of the two wools in the blends are often different from those used originally. The dyeing strengths and blending proportions would therefore be misleading, and they are intentionally not specified.

5.6.2 Any standard sample established by mutual agreement between the purchaser and the supplier.

5.6.3 ISO Gray Scale for Assessing Change in Color.¹¹

5.7 When the material to be evaluated is suspected of being very sensitive to variations in radiant exposure, and the apparatus does not meet the requirement of 4.1.6, it is the responsibility of the concerned parties to mutually agree upon a rearrangement schedule for specimens as part of the

exposure program in order to minimize any effect that variations in irradiance might have as a function of specimen position.

TEST METHOD A—CONTINUOUS EXPOSURE TO LIGHT AND INTERMITTENT EXPOSURE TO WATER SPRAY

6. Procedure

6.1 Apparatus shall be Type A, B, AH, BH, C, D, or E light-exposure device.

6.2 Program the instrument for continuous light and intermittent water spray in accordance with the manufacturer's instructions. Choice of the program selected will be by mutual agreement among the interested parties. Historical convention has established a cycle of 102 min of light followed by a cycle of 18 min of light and water spray as a commonly accepted program that permits the attainment of the maximum black-panel temperature during the light-only portion of the cycle.

6.3 In Types AH and BH apparatus, adjust dry- and wet-bulb temperature controls, humidifier, and immersion heater controls to maintain the desired conditions as specified in Section 5. In Types C, D, and E, adjust the chamber temperature and humidity controls to maintain desired conditions as specified in Section 5.

6.4 In Types A and B with humidifier, humidity may be adjusted but not controlled.

6.4.1 Type B instruments without a humidifier must operate with whatever humidity occurs from the evaporation of water in the bottom of the test chamber and from specimen spray.

TEST METHOD B—ALTERNATE EXPOSURE TO LIGHT AND DARKNESS AND INTERMITTENT EXPOSURE TO WATER SPRAY

7. Procedure

7.1 Apparatus shall be Type A, B, AH, BH, or E light-exposure device.

7.1.1 Types AH, BH, and E apparatus with automatic humidity controls may be operated on alternate light and dark cycles.

7.1.1.1 Operation during the light-on cycle shall be as described in Section 6.

7.1.1.2 Separate controls for temperatures and humidification may be adjusted during the dark cycle for automatic control as the program alternates from light to dark. In Type BH and E apparatus, a rack spray to cool the specimens by wetting the unexposed back surface can result in development of condensation on the exposed specimen surface during the dark cycle.

7.1.2 Types A, B, AH, BH, and E may be programmed to operate on alternate light and dark cycles without control of relative humidity.

TEST METHOD C—CONTINUOUS EXPOSURE TO LIGHT WITHOUT WATER SPRAY

8. Procedure

8.1 Apparatus shall be Types A, B, AH, BH, C, D, or E light-exposure devices programmed for continuous light only, in accordance with the manufacturer's instructions. Optical filters C or D described in 4.1.2.1 should be used.

¹¹ Reference Draft ISO Recommendation No. 177, Document ISO/TC-38 (Secretariat 102) 186. Scales with instructions for use are available from ISO member bodies in the various countries of the world. In the USA they may be obtained from The Secretary, American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

8.1.1 Adjust the controls on the apparatus so that the black-panel temperature is $63 \pm 3^\circ\text{C}$ ($145 \pm 5^\circ\text{F}$) and the relative humidity is $30 \pm 5\%$ when the xenon lamp is operated at a wattage that provides 20 AATCC Fading Units exposure in about 15 to 25 clock hours. Check at regular intervals and, when necessary, readjust the controls to maintain the specified values for these variables. Target values for black panel temperature, 20 AATCC Fading Units, and relative humidity should be 63°C , 20 clock hours, and 30 %, respectively.

NOTE 5—20 AATCC Fading Units is equivalent to a 1.5 ± 0.2 NLAB(40) unit change in Blue Wool L-4 (#4 on the ISO Gray Scale). Radiation of about 110 kJ/m^2 ($1.5 \text{ W/m}^2 \cdot 20\text{-h}$ exposure) at 420 nm has been found sufficient to produce the specified condition.

8.1.2 Expose the material to be tested as determined by mutual agreement among the concerned parties or, when not otherwise specified, in accordance with one of the following:

8.1.2.1 *Versus One AATCC Blue Wool Lightfastness Standard*—Expose the test specimen and any mutually agreed-upon AATCC Blue Wool Lightfastness Standard until the selected standard between its exposed and unexposed portion exhibits a color change equal to Step 4 of the ISO Gray Scale. Report the results by any mutually agreed-upon method of measuring change in the test specimen.

8.1.2.2 *Versus Set of AATCC Blue Wool Lightfastness Standards*—Expose the test specimen and a set of AATCC Blue Wool Lightfastness Standards until a maximum permissible amount of change, as determined by any specified or mutually agreed-upon method of measurement, occurs in the test specimen. Assign it a classification number equal to that of the numbered standard that most nearly exhibits a change in color equal to Step 4 on the ISO Gray Scale between its exposed and unexposed portion. Where one standard shows a color change greater than Step 4 and the next higher number standard shows less than a Step 4 color change, an intermediate or half-grade rating may be used.

8.1.2.3 *Versus Other Standard Sample*—Expose the test specimen and any mutually agreed-upon standard sample until either, by any specified or mutually agreed-upon method of measurement, shows an agreed-upon amount of change. Report the results on the basis of a comparison of

the specimen with the standard sample.

TEST METHOD D—ALTERNATE EXPOSURE TO LIGHT AND DARKNESS WITHOUT WATER SPRAY

9. Procedure

9.1 Apparatus shall be Types A, B, AH, BH, or E programmed to predetermined cycles of light and darkness by turning off the xenon arc.

NOTE 6—Type B instruments not equipped with a vaporizing unit do not meet the requirements of Test Method D. In Type A and Type B apparatus with intermittent blower operation during the light-off cycle and the atomizer in continuous operation, the test chamber air temperature will drop to that of room air less the heat removed by vaporization of water from wicks and atomizer.

9.1.1 Unless otherwise specified, exposure conditions shall be identical to Test Method B except that during the light-on cycle the equilibrium condition for relative humidity shall be $35 \pm 5\%$ and during periods of darkness equilibrium conditions shall be $90 \pm 5\%$ relative humidity at a dry-bulb temperature of $35 \pm 3^\circ\text{C}$ ($95 \pm 5^\circ\text{F}$).

10. Report

10.1 Report the following information:

10.1.1 Type and model of exposure device,

10.1.2 Type of light source and wattage,

10.1.3 Type and age of filters,

10.1.4 Spectral irradiance at sample location, $\text{W/m}^2 \cdot \text{nm}$ (see Note 9),

10.1.5 Irradiation, kJ/m^2 ,

10.1.6 Elapsed exposure time, h,

10.1.7 Light- or dark-water-humidity program employed,

10.1.8 Operating black-panel temperature,

10.1.9 Operating relative humidity,

10.1.10 Type of spray water,

10.1.11 Type of spray nozzle, and

10.1.12 Specimen relocation procedure.

NOTE 7—When direct measurement of irradiance and spectral irradiance cannot be made, data supplied by the manufacturer shall be substituted. Irradiation in kilojoules per square metre may be calculated by multiplying the product of the irradiance in watts per square metre and the exposure time in hours by the factor 3.6.

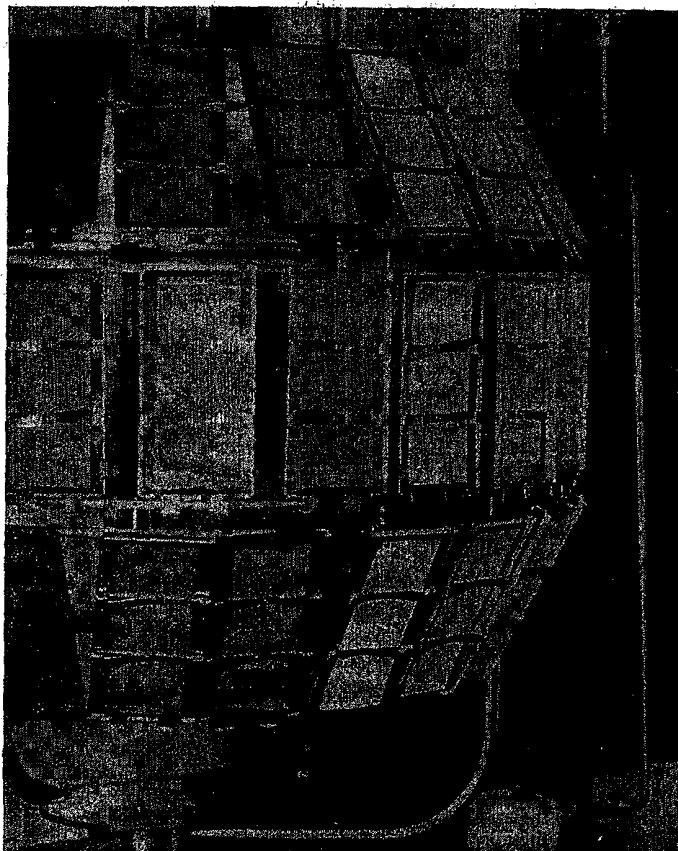


FIG. 4 Three-Tiered Specimen Rack

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.



Standard Test Method for Cathodic Disbonding of Pipeline Coatings Subjected to Elevated Temperatures¹

This standard is issued under the fixed designation G 42; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This test method describes an accelerated procedure for determining comparative characteristics of insulating coating systems applied to steel pipe exterior for the purpose of preventing or mitigating corrosion that may occur in underground service where the pipe will be exposed to high temperatures and is under cathodic protection. This test method is intended for use with samples of coated pipe taken from commercial production and is applicable to such samples when the coating is characterized by function as an electrical barrier.

1.2 This test method is intended for testing coatings submerged or immersed in the test solution at elevated temperature. When it is impractical to submerge or immerse the test specimen, Test Method G 95 may be considered where the test cell is cemented to the surface of the coated pipe specimen. If room temperatures are required, see Test Method G 8. If a specific test method is required with no options, see Test Method G 80.

1.3 The values stated in SI units to three significant decimals are to be regarded as the standard.

1.4 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 ASTM Standards:

G 8 Test Methods for Cathodic Disbonding of Pipeline Coatings²

G 12 Test Method for Nondestructive Measurement of Film Thickness of Pipeline Coatings on Steel²

G 80 Test Method for Specific Cathodic Disbonding of Pipeline Coatings³

G 95 Test Method for Cathodic Disbondment Test of Pipeline Coatings (Attached Cell Method)³

3. Summary of Test Method

3.1 This test method subjects the coating on the test specimen to electrical stress in a highly conductive electrolyte. The coating is artificially perforated before starting the

test. The electrical stress is produced by connecting the test specimen to the negative terminal of a source of direct current and by connecting an anode to the positive terminal. Electrical instrumentation is provided for measuring the current flowing in the cell. The electrical potential is also measured and the specimen is physically examined at intervals during the test period and upon conclusion of the test.

3.1.1 The cathodic stress is applied under conditions of a constant-elevated temperature.

4. Significance and Use

4.1 Damage to pipe coating is almost unavoidable during transportation and construction. Breaks or holidays in pipe coatings may expose the pipe to possible corrosion since, after a pipe has been installed underground, the surrounding earth will be moisture-bearing and will constitute an effective electrolyte. Applied cathodic protection potentials may cause loosening of the coating, beginning at holiday edges. Spontaneous holidays may also be caused by such potentials. This test method provides accelerated conditions for cathodic disbondment to occur and provides a measure of resistance of coatings to this type of action.

4.2 The effects of the test are to be evaluated by physical examinations and monitoring the current drawn by the test specimen. Usually there is no correlation between the two methods of evaluation, but both methods are significant. Physical examination consists of assessing the effective contact of the coating with the metal surface in terms of observed differences in the relative adhesive bond. It is usually found that the cathodically disbonded area propagates from an area where adhesion is zero to an area where adhesion reaches the original level. An intermediate zone of decreased adhesion may also be present.

4.3 Assumptions associated with test results include:

4.3.1 Maximum adhesion, or bond, is found in the coating that was not immersed in the test liquid, and

4.3.2 Decreased adhesion in the immersed test area is the result of cathodic disbondment.

4.4 Ability to resist disbondment is a desired quality on a comparative basis, but disbondment in this test method is not necessarily an adverse indication of coating performance. The virtue of this test method is that all dielectric-type coatings now in common use will disbond to some degree, thus providing a means of comparing one coating to another.

4.5 The current density appearing in this test method is much greater than that usually required for cathodic protection in natural environments.

4.6 That any relatively lesser bonded area was caused by electrical stressing in combination with the elevated and or

¹ This method is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials and is the direct responsibility of Subcommittee G03.06 on Durability of Pipeline Coatings and Linings.

Current edition approved May 25, 1990. Published July 1990. Originally published as G 42-75T. Last previous edition G 42-85.

² Annual Book of ASTM Standards, Vols 06.01 and 14.02.

³ Annual Book of ASTM Standards, Vol 14.02.

depressed temperature and was not attributable to an anomaly in the application process. Ability to resist disbondment is a desired quality on a comparative basis, but most insulating materials will disbond to some extent under the accelerated conditions of this test. Bond strength is more important for proper functioning of some coatings than others and the same measured disbondment for two different coating systems may not represent equivalent loss of corrosion protection.

4.6.1 The amount of current flowing in the test cell is a relative indicator of the extent of areas requiring protection against corrosion; however, the current density appearing in this test is much greater than that usually required for cathodic protection in natural, inland soil environments.

4.6.2 Test voltages higher than those recommended may result in the formation of chlorine gas. The subsequent chemical effects on the coating could cast doubt on the interpretation of the test results.

5. Apparatus

5.1 *Test Vessel*—A suitable nonreactive vessel shall be used, capable of withstanding internal heating at not less than 60°C and suitable for continuous circulation of the electrolyte. A 19-L (5-gal) cylindrical glass vessel has been found suitable, having an approximate diameter of 300 mm (12 in.) and a depth of 300 mm. A flat bottom is required for operation of a magnetic stirring rod. Dimensions of the vessel shall permit the following requirements:

5.1.1 Test specimens shall be suspended vertically in the vessel with at least 25 mm (1 in.) clearance from the bottom.

5.1.2 Test specimens shall be separated by not less than 38 mm (1½ in.), and a vertically suspended anode can be placed at an equal distance from each specimen not less than the separation distance.

5.1.3 Test specimens shall be separated from any wall of the vessel by not less than 13 mm (½ in.).

5.1.4 Depth of electrolyte shall permit the test length of the specimen to be immersed as required in 7.4.

5.1.5 The reference electrode may be placed anywhere in the vessel, provided it is separated from the specimen and from the anode by not less than 38 mm (1½ in.).

5.2 *Anode*—The anode shall be provided with a factory-sealed, insulated copper wire lead.⁴

5.3 *Connectors*—Wiring from anode to test specimen shall be 4107 cmil (14-gage Awg), minimum, insulated copper. Attachment to the test specimen shall be by soldering or brazing to the nonimmersed end, and the place of attachment shall be coated with an insulating material. A junction in the connecting wire is permitted, provided that it is made by means of a bolted pair of terminal lugs soldered or mechanically crimped to clean wire ends.

5.4 *Holiday Tools*—Holidays shall be made with conventional drills of the required diameter. For use in preparing small-diameter pipe specimens such as 19-mm (¾-in.) nominal diameter pipe, the use of a drill modified by substantially grinding away the sharp cone point has been found effective in preventing perforation of the metal wall of the pipe. A

sharp-pointed knife with a safe handle is required for use in making physical examinations.

5.5 Multimeters:

5.5.1 *Multimeter*, for direct current, having an internal resistance of not less than 10 MΩ and having a range from 0.01 to 5 V for measuring potential to the reference electrode.

5.5.2 *Multimeter*, for direct current, having an internal resistance of not less than 11 MΩ and capable of measuring as low as 10 μV potential drop across a shunt in the test cell circuit.

5.5.3 *Multimeter*, for initial testing of apparent coating resistance.

5.6 *Reference Electrode*—Saturated Cu/CuSO₄ electrode having a potential of -0.316 V with respect to the standard hydrogen electrode shall be the standard of reference in these test methods. Other electrodes may be used but measurements thus obtained shall be converted to the Cu/CuSO₄ reference for reporting by making the proper correction.

NOTE 1—A saturated Cu/CuSO₄ electrode reading -1.50 V at 25°C will read -1.53 V at 60°C, a scale increase of 0.03 V.

5.6.1 A saturated calomel electrode at 25°C is converted to Cu/CuSO₄ by adding -0.07 V to the observed reading. If the saturated calomel electrode reads -1.43 V at 25°C, it will read -1.46 V at 60°C, a scale increase of 0.03 V. It follows that a saturated calomel electrode reading of -1.46 V at 60°C is equal to a saturated Cu/CuSO₄ reading of -1.50 V at 25°C.

5.6.2 A 0.1 normal calomel electrode at 25°C is converted to Cu/CuSO₄ by subtracting -0.02 V from the observed reading. Since the potential change due to an increase from 25°C to 60°C is negligible, it follows that a 0.1 normal calomel electrode reading -1.52 V at 60°C is equal to a saturated Cu/CuSO₄ reading of -1.50 V at 25°C.

5.7 *Thermometers*, two, mercury-filled type, accurate to ±1°C. One shall be of the full-immersion type for measuring temperature near the bottom of the vessel, and a second thermometer shall be of the partial-immersion type for measuring temperature near the top of the vessel.

5.8 *Combination Heater Plate*, with built-in magnetic stirrer, or equivalent, shall be used for heating and stirring the electrolyte. The heater shall be adjustable to produce and control a temperature of 60 ± 1°C in the test vessel.

5.9 *Direct-Current Rectifier*, capable of supplying constant current at a voltage of 1.50 ± 0.01 V, as measured between the specimen and reference cell.

5.10 *Thickness Gage*, for measuring coating thickness in accordance with Test Method G 12.

5.11 *Precision Resistor*, 1Ω ± 1%, 1 W (min), to be used in the test cell circuit as a shunt for current.

5.12 *Carbon or Stainless Steel Electrode*, used temporarily with the volt-ohm-meter to determine apparent initial holiday status of the test specimen.

5.13 *Additional Connecting Wires*, 4107 cmil (14-gage Awg), minimum, insulated copper.

5.14 *Brass Snids*, used at a terminal board, together with alligator clips or knife switches, for making and breaking circuits. Alligator clips shall not be used to connect the electrodes or specimens at the top location of test cells.

6. Reagents and Materials

6.1 The electrolyte shall consist of potable tap water or

⁴ Duriron, a material found suitable for this purpose is available from Duriron Co., Inc., Dayton, OH.

higher purity water (distilled or demineralized water is satisfactory) with the addition of 1 weight % of each of the following technical-grade salts, calculated on an anhydrous basis: sodium chloride, sodium sulfate, and sodium carbonate.

NOTE 2—The resulting solution has a pH of 10 or higher and a resistivity of 25 to 50 $\Omega \cdot \text{cm}$ at room temperature.

6.2 Materials for sealing the ends of coated pipe specimens may consist of bituminous products, wax, epoxy, or other materials, including molded elastomeric or plastic end caps, capable of withstanding the test temperature.

6.3 Plywood has been found suitable for the construction of nonconductive test vessel covers and for the support through apertures of test specimens and electrodes. Wood dowels introduced through holes in the top ends of test specimens have been found suitable for suspending test specimens from the vessel cover.

7. Test Specimen

7.1 The test specimen shall be a representative piece of production-coated pipe. One end shall be plugged, sealed, or capped.

7.2 One holiday shall be made in the middle of the immersed length by drilling a radial hole through the coating so that the angular cone point of the drill will fully enter the steel where the cylindrical portion of the drill meets the steel surface. The drill diameter shall be not less than three times the coating thickness, but it shall never be less than 6 mm ($\frac{1}{4}$ in.) in diameter. The steel wall of the pipe shall not be perforated. With small-diameter pipes, where there is danger of perforating the pipe, the holiday shall be started with a standard 60° cone point and finished with a drill that has had a substantial portion of the cone point ground away.

NOTE 3—Before making the holiday, see 7.5.

7.3 The end of the pipe which will protrude above the

immersion line shall be provided with suitable supporting means and a separate wire connection for electrical purposes, soldered, or brazed to the pipe. The protruding end, including hanger and wire connections, shall be protected and sealed with an insulating coating material.

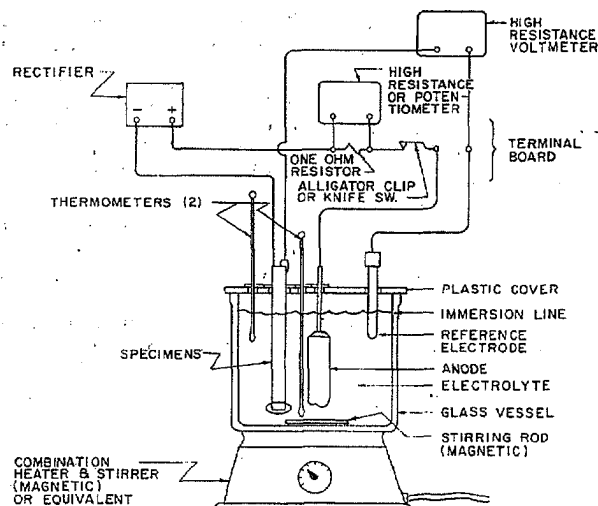
7.4 The specimen test area shall consist of the area between the edge of the bottom end seal and the immersion line. The bottom end seal area shall not be considered part of the area tested. Coated specimens of any suitable diameter and length of pipe may be used, but the immersed area shall be not less than 23 200 mm^2 (36 in.^2). An area of 92 900 mm^2 (1 ft^2) has been found preferable when convenient.

7.5 The continuity of the coating and efficiency of the end seal shall be tested before making artificial holidays as follows:

7.5.1 Immerse the test specimen and a carbon or stainless steel electrode in the electrolyte. Connect one terminal of the ohmmeter to the test specimen and the other terminal to the carbon or stainless steel electrode. Measure the apparent resistance in ohms, making two determinations: one with the specimen connected to the positive terminal of the ohmmeter, and one with the specimen connected to the negative terminal. The lowest of the two readings should be not less than 1000 $\text{M}\Omega$, however, the test may be conducted with a low reading less than 1000 $\text{M}\Omega$, provided that the condition is taken into account in evaluating results.

8. Procedure

8.1 Immerse the test specimen in the electrolyte and connect it to the anode as shown in Fig. 1. Adjust the rectifier or voltage divider so that the potential between specimen and reference cell is -1.50 ± 0.01 V at 25°C (-1.53 ± 0.01 V at 60°C) (see Fig. 2). Position the holiday to face toward the anode. Space the anode with respect to test specimens as required in 5.1. Mark the correct immersion level on the exterior of the test vessel and maintain by daily



NOTE—For multiple specimens in the same test vessel, see Fig. 2, for circuit diagram using voltage dividers.

FIG. 1 Test Set-Up for Cathodic Disbonding Test at Elevated Temperature

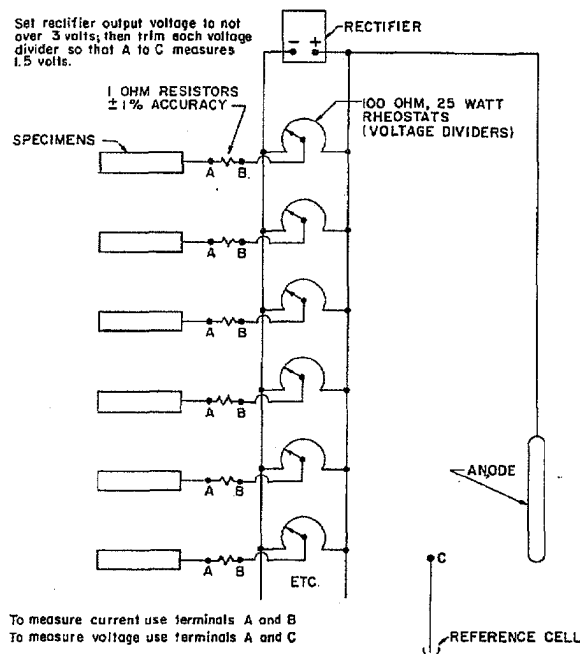


FIG. 2 Circuit Diagram for Cathodic Disbonding Test

additions of preheated, distilled, or demineralized water as required.

8.2 The temperature of the electrolyte surface to bottom shall be not less than $60 \pm 3^\circ\text{C}$. The electrolyte shall be continuously circulated by means of the stirring apparatus or equivalent. The maximum temperature shall be attained 4 ± 1 h after starting the heater and immersing the specimen.

NOTE 4—Throughout the test period, ejected sealant or salt deposits, or both, may form near, or partially obstruct the intentional coating holiday described in 7.2. No attempt shall be made to clear the holiday while the test is in progress.

8.3. Electrical Monitoring Schedule:

8.3.1 Electrical measurements shall be made on the start-up day and on each normal working day thereafter for the duration of the test. A maximum of three consecutive nonworking days shall be preceded by at least two working days; one non-working day or two consecutive non-working days shall be preceded by at least one working day, except at start-up and termination when three and two working days are required, respectively.

8.3.2 Electrical measurements characterizing the start of the test are defined as the average of measurements taken on the second and third days after immersion.

8.3.3 Electrical measurements characterizing intermediate and terminal time spans shall be taken on two successive days prior to and including the target date.

8.4 Electrical measurements and adjustments made each normal working day:

8.4.1 Measure E_2 , the stress potential in volts between the test specimen and the reference electrode, without disconnecting the energized anode or specimen from the circuit.

Use the high-resistance voltmeter described in 5.5. The stress potential, E_2 , should measure -1.50 ± 0.01 V at 25°C (-1.53 ± 0.01 V at 60°C); if it does not, adjust the rectifier or voltage divider accordingly.

8.4.2 After E_2 has been measured and adjusted, if necessary, measure I_1 , the current demand in amperes, by determining the potential drop across the 1- Ω precision resistor permanently installed in the test cell circuit with the high-resistance voltmeter described in 5.2. The voltage reading will be numerically equal to amperes.

NOTE 5—An alternative method of measuring current demand utilizes a zero-resistance ammeter. In this method, the wire connection between the test specimen and anode is temporarily broken and a zero-resistance ammeter temporarily interposed between the specimen and the anode. Reconnect the specimen to the anode with the connector wire as soon as this measurement is completed.

8.4.3 Measure E_1 , the polarized potential, in volts, using the high-resistance voltmeter described in 5.5 connected between the test specimen and the reference electrode as follows:

8.4.3.1 Disconnect the anode from the test specimen while closely observing the high-resistance voltmeter. As the instrument pointer falls, it will dwell significantly at the polarized potential before recording further. The dwell point is E_1 .

8.5 Standard duration of the test period shall be 30 days (720 h) in the energized test vessel including warm-up and cool-down times, unless longer test periods are specified.

8.6 A physical examination shall be performed immediately upon termination of the test period as follows:

8.6.1 Before examination, wash the test specimen care-

under gently flowing tap water without disturbing destructive effects. Adjust the temperature of the tap water so that it approximates that of the room temperature.

7.2 Examine the entire immersed area visually for any evidence of new holidays and loosening of coating at the cut end, including the artificial holiday.

7.3 Drill a new test hole in the coating in an area that is not immersed, staying away from the immersion line and the cut end. Recommended distances are given in Fig. 3. Use the same drilling procedure described in 7.2.

7.4 In order to gage or calibrate the lifting technique, attempt to lift the coating at the new test hole with the point of a sharp knife after making cuts through the coating meeting at the center of the hole. Inability or relative resistance to lifting or disbonding the coating shall be considered the adhered or bonded condition of the untested coating with respect to the lifting technique used.

7.5 Determine if the coating has been loosened at the immersed test hole by attempting to lift the coating with the point of a sharp knife after making cuts through the coating

intersecting at the holiday or point of inspection using the same technique applied in 8.6.4. Classify coating that can be lifted or disbonded more readily than at the new test hole as unsealed area. Measure the unsealed area.

NOTE 6—The use of a transparent film having a grid laid out in small squares such as 2.54 mm (0.1 in.) on a side has been found useful. The film is placed against the unsealed area and the boundary of the unsealed area is traced on the grid. The area is then obtained by counting the squares within the bounded area.

8.7 Determine and record the resistivity and pH of the electrolyte at the beginning and end of the test period.

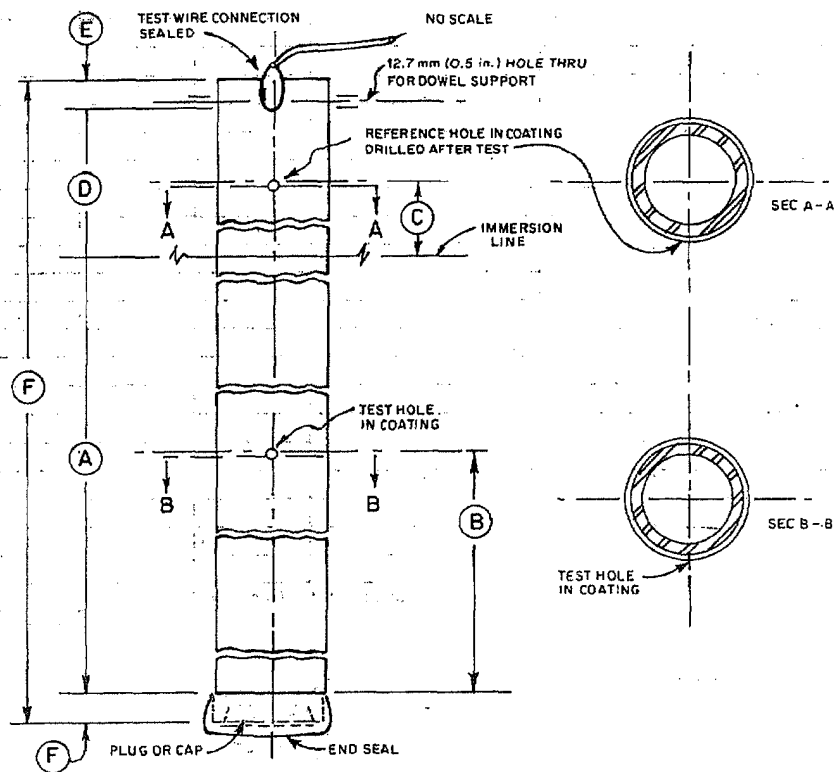
9. Report

9.1. The report shall include the following (see Fig. 4):

9.1.1 Complete identification of the test specimen, including:

9.1.1.1 Name and code number of the coating,

9.1.1.2 Size of pipe,



DIMENSION	MILLIMETRES	INCHES
A	490.22 ± 12.7	(19.300 ± 0.500)
B	245.11 ± 12.7	(9.650 ± 0.500)
C	114.3 ± 6.35	(4.500 ± 0.250)
D	233.363 MIN	(9.1875, min)
E	19.05 MIN	(0.750, min)
F	762 MIN	(30, min)

FIG. 3 Recommended Dimensions for Specimen

DATA SHEET AND REPORT

Cathodic Disbonding of Pipeline Coatings Subjected to Elevated Temperatures

Specimen _____	Production Date _____	Target, °C Electrolyte _____
Coating _____	Production Run No. _____	
Applied by _____		
Thickness, mm (in.): Max _____ Min _____	At Holiday (1) _____ (2) _____	
Date Started _____	pH @ 25°C _____	Ohm-cm @ 25°C _____
Date Finished _____	pH @ 25°C _____	Ohm-cm @ 25°C _____
Immersed Area, mm ² (in. ²) _____		Initial Ohms: + _____
Holiday Diameter, mm (in.) _____		

Report No. _____
Page _____ of _____
Initials _____

[illegible]

Change: Start to Termination:

Comments:

Physical Examination at Termination:

	mm ² (in. ²)	Circ. D ^a mm (in.)
Area Disbonded		
Less Initial Holiday		
Total Unsealed Area (net)		

^a Equivalent Circle Diameter.

FIG. 4 Suggested Form for Use in Presenting Data

9.1.1.3 Source, production date, and production run number,

9.1.1.4 Minimum-maximum coating thickness.

9.1.1.5 Immersed area,

9.1.1.6 Size and number of initial holidays, and

9.1.1.7 Dates of starting and terminating test.

9.1.2 The relative resistances of the test specimen, in ohms, before the artificial holiday was made as described in 7.5.

9.1.3 Tally of areas that have been found unsealed on the terminal date. Areas may be reported in square millimetres (or square inches) or millimetres (or inches) of equivalent circle diameter of the area, or both.

9.1.4 The results of starting, intermediate, and terminal electrical measurements. Report the following measurements:

9.1.4.1 Current demand in microamperes or negative characteristic of the logarithm of the current in amperes, in both,

9.1.4.2 The value of $\Delta E = E_2 - E_1$, in volts, and

9.1.4.3 Change from start to termination for values in 9.1.4.1 and 9.1.4.2.

9.1.5 Temperature of the electrolyte at approximately the same hour each working day.

9.1.6 Other information that may be pertinent.

10. Precision and Bias

10.1 Precision data are limited to two adjacent specimens taken from the same production-coated pipe and assumed that the production process was uniform with respect to pipe

surface condition and coating material. Specimens that were not adjacent in the as-produced condition or were taken from different lengths of pipe may represent differing process conditions. The following data should be used for judging the acceptability of results. (These precision data are approximations based on limited data, but they provide a reasonable basis for judging the significance of results.)

10.2 *Repeatability*—Duplicate results obtained within a laboratory should be acceptable unless they differ by more than 25 mm (1 in.) in value D in accordance with the following equation:

$$D = (A/0.785)^{1/2}$$

where:

A = unsealed area developed from one artificial holiday, mm² (or in.²)

or if they differ by more than unity in the negative characteristic of the logarithm of the current demand in amperes.

10.3 *Reproducibility*—The results reported by one laboratory should be acceptable unless they differ from those of another laboratory by more than 25 mm (1 in.) for the value D in the equation given in 10.2, and by more than unity in the negative characteristic of the logarithm of the current demand in amperes.

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Standard Practice for Operating Light- and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Nonmetallic Materials¹

This standard is issued under the fixed designation G 53; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice covers the basic principles and operating procedures for using fluorescent ultraviolet (UV) and condensation apparatus to simulate the deterioration caused by sunlight and water as rain or dew.

1.2 This practice is limited to the method of obtaining, measuring, and controlling the conditions and procedures of exposure. It does not specify the exposure conditions best suited for the material to be tested. Specimen preparation and evaluation of the results are covered in ASTM test methods or specifications for specific materials.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

E 220 Method for Calibration of Thermocouples by Comparison Techniques²

G 7 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials³

2.2 CIE Standard:

No. 20 Recommendations for the Integrated Irradiance and the Spectral Distribution of Simulated Solar Radiation for Testing Purposes⁴

3. Description of Terms Specific to This Standard

3.1 *irradiance*—the radiation incident on a surface expressed in W/m^2 . Irradiance is the total of the incident radiation at all wavelengths. Forty watt fluorescent lamps of the UV-B and UV-A types generate similar amounts of

irradiance. However, since this irradiance is distributed at different wavelengths, the photochemical effects caused by these different lamps vary greatly. Therefore, irradiance should not be used to compare UV light sources.

3.2 *spectral irradiance*—distribution of irradiance as a function of wavelength. It is expressed in W/m^2 per wavelength band. The spectral irradiance of sunlight is often shown as W/m^2 per 10 nm band. The spectral irradiance of fluorescent UV lamps should be shown in bands 1 or 2 nm wide. Spectral irradiance is the proper method for comparing sources with different energy distributions.

3.3 *spectral energy distribution (SED)*—general term for the characterization of the amount of radiation present at each wavelength. SEDs can be expressed by power in watts, irradiance in W/m^2 , or energy in joules. The shape of the SED would be identical in all of these units. Fluorescent lamps are frequently described by relative SEDs which show the amount of radiation at each wavelength as a percentage of the amount of radiation at the peak wavelength. Fig. 1 is a relative SED.

3.4 *ultraviolet regions*—CIE Publication No. 20 (1972) divides the ultraviolet spectrum into three regions: UV-A, radiation in wavelengths between 315 nm and 400 nm; UV-B, radiation in wavelengths between 280 nm and 315 nm; and UV-C, radiation in wavelengths shorter than 280 nm.

3.5 *fluorescent UV lamp*—lamp in which radiation at 254 nm from a low-pressure mercury arc is transformed to longer wavelength UV by a phosphor. The spectral energy distribution of a fluorescent UV lamp is determined by the emission

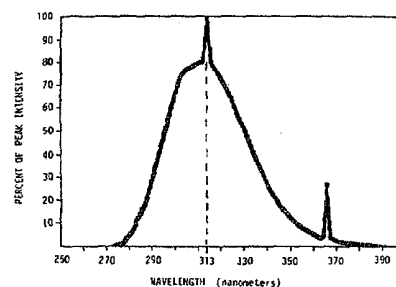


FIG. 1 Typical Relative SED of UV-B Lamp

¹ This practice is under the jurisdiction of ASTM Committee G-3 on Durability of Nonmetallic Materials, and is the direct responsibility of Subcommittee G03.03 on Simulated and Controlled Environmental Tests.

Current edition approved July 29, 1988. Published September 1988. Originally published as G 53 - 77. Last previous edition G 53 - 84.

² Annual Book of ASTM Standards, Vol 14.03.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Available from Secretary, U.S. National Committee, CIE, National Bureau of Standards, Gaithersburg, MD 20899.

spectrum of the phosphor and the UV transmission of the glass tube.

Summary of Practice

4.1 Specimens are alternately exposed to ultraviolet light alone and to condensation alone in a repetitive cycle.

4.2 The UV source is an array of fluorescent lamps, with lamp emission concentrated in the UV range.

4.3 Condensation is produced by exposing the test surface to a heated, saturated mixture of air and water vapor, while the reverse side of the test specimen is exposed to the cooling influence of ambient room air.

4.4 The exposure condition may be varied by selection of the fluorescent UV lamp; the timing of the UV and condensation exposure; the temperature of UV exposure; and the temperature of condensation exposure.

5. Significance and Use

5.1 The use of the apparatus under this practice is intended to simulate the deterioration caused by water as rain or dew and the ultraviolet energy in sunlight. It is not intended to simulate the deterioration caused by localized weather phenomena, such as atmospheric pollution, biological attack, and salt water exposure.

5.2 Variation in results may be expected when operating conditions are varied within the accepted limits of this practice. Therefore, no reference shall be made to results from use of this practice unless accompanied by a report detailing the specific operating conditions in conformance with Section 11.

5.3 Any report correlating results from use of this practice to results from a period of natural weathering shall specify in detail the conditions of natural exposure, since sunlight and water effects upon materials exposed to the weather will vary from year to year and also vary with location, latitude, time of year, temperature, proximity to water sources, etc.

NOTE 1—Practice G 7 lists the information required to describe a particular condition of outdoor exposure.

5.4 Correlations established and reported in conformance with 5.2 and 5.3 shall not be extrapolated to other test conditions permitted by this practice, to other conditions of natural exposure, or to materials other than those tested.

6. Apparatus⁵

6.1 *Test Chamber*, (see Fig. 2) constructed of corrosion-resistant materials enclosing eight fluorescent UV lamps, a heated water pan, test specimen racks, and provisions for controlling and indicating operating times and temperatures.

6.2 *Lamps*, rapid start, medium bipin fluorescent UV lamps with a length of 1220 mm, and a nominal rating of 40 W when operated from a ballast providing a controlled current of 430 mA at 102V.

6.2.1 Unless otherwise specified, the lamps shall be UV-B lamps with a peak emission at 313 nm and a spectral energy distribution as shown in Fig. 1.

⁵ Apparatus and lamps from Q-Panel Co., 26200 First St., Cleveland, OH 44145, and from Atlas Electric Devices Co., 4114 N. Ravenswood Ave., Chicago, IL 60613, have been found satisfactory.

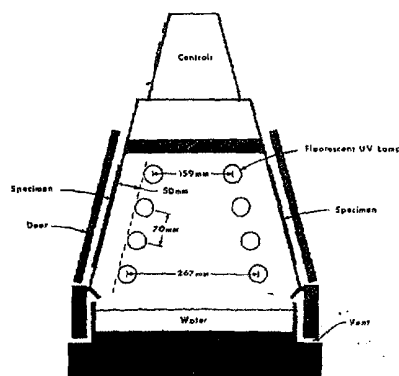


FIG. 2 Apparatus Schematic Cross Section

6.2.2 Other fluorescent UV lamps meeting the size and electrical characteristics in 6.2 may be used, provided that the lamp and spectral energy distribution are reported in conformance with Section 11.

6.3 *Lamp Spacing and Arrangement*—The lamps shall be mounted in two banks of four lamps each as shown in Fig. 2. The lamps in each bank shall be mounted parallel in a flat plane on 70-mm centers.

6.4 *Specimen Mounting and Arrangement*—The test specimens shall be mounted in stationary racks with the plane of the test surface parallel to the plane of the lamps at a distance of 50 mm from the nearest surface of the lamps, as shown in Fig. 2.

6.4.1 The test specimens shall be exposed within an area 210 mm in height by 900 mm wide on each side of the apparatus located as shown in Fig. 4.

NOTE 2—It is possible to mount specimens above, below, and beside the 210 by 900 mm area, but specimens so mounted will be exposed to lower UV intensities.

6.5 *Condensation Mechanism*—Water vapor shall be generated by heating a water pan extending under the entire sample area and containing a minimum water depth of 25 mm. Specimen racks and the test specimens themselves shall constitute the side walls of the chamber. The back side of the

LAMP ROTATION

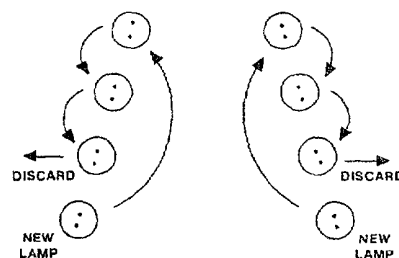


FIG. 3 Lamp Rotation

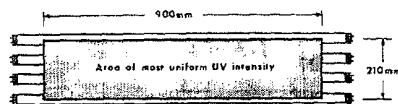


FIG. 4 Limits of Area of Uniform Intensity

specimens shall be exposed to cooling effects of ambient room air. The resulting heat transfer causes water to condense on the test surface.

6.5.1 The specimens shall be arranged so that condensate runs off the test surface by gravity and is replaced by fresh condensate in a continuous process. Vents along the bottom of the test chamber shall be provided to permit an exchange of ambient air and water vapor to prevent oxygen depletion of the condensate.

6.6 *Water Supply*, with an automatic control to regulate the level in the water pan shall be provided. Distilled, deionized, or potable tap water are equally acceptable for purposes of the test, since the condensation process itself distills water onto the test surface.

6.7 *Cycle Timer*, a continuously operating cycle time, for programming the selected cycle of UV periods and condensation periods.

6.7.1 Hour meters shall be provided to record total time of operation and total time of UV exposure.

6.8 *Specimen Temperature Measurement:*

6.8.1 Specimen temperature shall be measured by a thermometer with a remote sensor attached to a black aluminum panel 75 by 100 by 2.5 mm thick. The thermometer shall be precise to $\pm 1^\circ\text{C}$ through a range from 30° to 80°C . The indicator dial shall be located outside the test chamber.

6.8.2 The black aluminum panel with the thermometer sensor shall be positioned in the center of the exposure area so that the sensor is subject to the same conditions as the specimens.

6.9 *Specimen Temperature Control:*

6.9.1 During UV exposure, the selected equilibrium temperature shall be maintained within $\pm 3^\circ\text{C}$ by supplying heated air to the test chamber.

6.9.2 During condensation exposure, the selected equilibrium temperature shall be maintained within $\pm 3^\circ\text{C}$ by heating the water in the water pan.

6.9.3 The UV and condensation temperature controls shall be independent of each other.

6.9.4 Doors shall be located on the room air side of the specimen racks to act as insulation during the UV exposure and to minimize drafts. Such doors shall not interfere with the room air cooling of the specimen during the condensation exposure.

6.10 *Test Chamber Location:*

6.10.1 The apparatus shall be located in an area maintained at a temperature between 20°C and 30°C . The room temperature shall be measured by thermometers mounted on interior walls or column approximately 1500 mm above the floor level and at least 300 mm from any heated apparatus. Three or more thermometers located at various points will show any temperature variation in the area.

6.10.2 It is recommended that the apparatus be located at least 300 mm from walls or other apparatus. Nearby heat

sources, such as ovens or heated test apparatus, should be avoided or shielded, because such heat sources can reduce the cooling required for condensation.

6.10.3 The room where the apparatus is located shall be ventilated to remove the heat and moisture produced and to maintain the temperatures specified in 6.10.1. Two to four air changes per hour will normally provide sufficient ventilation.

7. Test Specimens

7.1 Replicate specimens are desirable to provide a record of degradation at different time intervals. Retention of an unexposed specimen is recommended as it is difficult to mask a specimen to prevent exposure to condensation.

7.2 For specimens of insulating materials, such as wood, plastic, or porous laminates, maximum specimen thickness should be 20 mm to allow adequate heat transfer for condensation.

7.3 To provide rigidity, flexible specimens may be attached to a backing panel made of aluminum or other noncorrosive heat conductive material.

7.4 Cut edges of coated steel specimens shall be protected so that rust does not contaminate the test surface.

7.5 Holes in specimens and any openings larger than 1 mm around irregularly shaped specimens shall be sealed to prevent loss of water vapor. Porous specimens, such as textiles or wood shall be backed with a vapor barrier such as metal or plastic.

8. Calibration and Standardization

8.1 The thermometer or thermocouple which indicates test temperature shall be calibrated by immersing the sensing element and a liquid-in-glass thermometer in water heated to approximately 70°C and comparing the two temperatures as in Method E 220.

8.2 The formation of condensation may be observed by using clear glass or plastic blanks in the specimen holders or rack. One large sheet of plastic may also be used for this purpose. The condensate that forms on a given area during a period of condensation may be collected and then measured.

8.3 The fading and other changes caused by the fluorescent UV lamps may be observed by exposing lightfastness standards in the apparatus using UV alone without condensation.

8.3.1 The AATCC Blue Wool Lightfastness reference materials⁶ may be used to measure the changes caused by UV. The L2 Blue Wool has been found satisfactory for evaluating fluorescent UV lamps.

8.3.2 Other lightfastness reference materials may be used by agreement.

8.4 Reference standards for calibrating the operation of the apparatus in alternate exposure to UV light and condensation may be prepared from painted metal, plastics, or other materials. Painted metal specimens produced by the coil-coating process have been found suitable for such reference standards.

9. Procedure

9.1 Mount the test specimens in the specimen racks with

⁶ Available from American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709.

the test surfaces facing the lamp. When the test specimens do not completely fill the racks, fill the empty spaces with blank panels to maintain the test conditions within the chamber.

9.2 Program the selected test conditions. Operate continuously, repeating the cycle, except for servicing the instrument and inspection of specimens.

9.3 Various test conditions may be used. If no conditions are specified, the following cycle and temperatures are suggested: 4 h UV at 60°C, 4 h Condensation at 50°C.

NOTE 3—Prior versions of this practice recommended a condensation temperature of 40°C in 9.3. When operating in room conditions that do not comply with those set forth in 6.10, a 40°C condensation temperature can result in inadequate condensation. Therefore, a 50°C condensation temperature is now suggested.

9.3.1 Any test temperature that can be maintained within the limits specified in 6.9.1 and 6.9.2 may be used. UV test temperatures of 50°C, 60°C, and 70°C are widely used. A condensation test temperature of 50°C is commonly used.

9.3.2 The following time cycles are widely used: 4 h UV/4 h CON, and 8 h UV/4 hr CON. Use UV and condensation periods of at least 2-h duration to allow sufficient time to reach equilibrium.

9.3.3 The severity of the UV exposure is influenced by test temperatures and time cycles. Photochemical reaction begins as soon as the UV lamps are turned on. The rate of UV degradation is proportional to the time of UV exposure or the temperature of UV exposure, or both. UV exposures at temperatures higher than those expected in the service environment can cause abnormal thermal degradation.

9.3.4 Water reactions during condensation exposure are affected by the permeability of the specimen and require time to initiate. The rate of water degradation is increased by increased temperature. However, long hot condensation exposures can cause abnormal degradation. Four-hour condensation exposures are often used for paints on metals, while condensation exposures of 20 h duration may be used on wood.

9.4 Maintenance—Periodic maintenance is required to maintain uniform UV and condensation exposure conditions.

9.4.1 After 400 to 450 h of lamp operating time, replace one lamp in each bank of lamps, and rotate the other lamps as shown in Fig. 3. This procedure provides a useful lamp life of 1600 to 1800 h.

9.4.2 Drain water and clean water pan when conducting lamp replacement and rotation procedure. Scum on the top of the water can inhibit water vaporization.

9.5 Sample Rotation—The samples' positions within the test area should be rotated on a regular basis in order to minimize any effects from horizontal or vertical variations in UV or temperature. The sample rotation sequence should provide that all samples spend approximately the same amount of exposure time near the center of the exposure area and near the edges of the exposure area. The following rotation sequence is recommended:

9.5.1 Horizontal Rotation—Once a day rotate the extreme left hand and extreme right hand samples into the center as shown in Fig. 6.

9.5.2 Vertical Rotation—Rotate the samples vertically so that each sample spends the same amount of exposure time in each vertical position in the sample holder. For instance, if two samples are stacked vertically in each sample holder, then the top and bottom sample should switch places halfway through the test method. If four samples are stacked vertically, then the samples should be repositioned vertically three times during the test method.

9.5.3 Other sample rotation sequences may be used by agreement.

9.6 Inspect specimens daily in tests with a length of 1 week or less. Tests for longer periods should be inspected weekly.

9.6.1 A permanent record of degradation at various times may be obtained by using three replicate specimens and

REPORT FORM		ASTM PRACTICE G-53		FLUORESCENT-UV / CONDENSATION TEST	
TEST No.:		Date:		By:	
APPARATUS Type and Model				TEST TIME _____ hrs.	
LAMP	MANUFACTURER	TYPE	PEAK EMISSION _____ nm	LOW CUT-OFF (1% of peak) _____ nm	
CYCLE _____ hrs UV at _____ °C. / _____ hrs COND at _____ °C.					
SPECIAL TEST CONDITIONS _____ _____ _____					
START DATE:	END DATE:	TIMER HRS. START	TIMER HRS. STOP	LAMP SERVICE at _____ hrs.	
SPECIMEN SIZE _____				Rotated ☐ Yes ☐ No	
MATERIAL _____					
PROPERTIES TO BE MEASURED			MEASURING PROCEDURE (ASTM Spec., etc.)		
1. _____					
2. _____					
3. _____					

FIG. 5 Sample Report Form

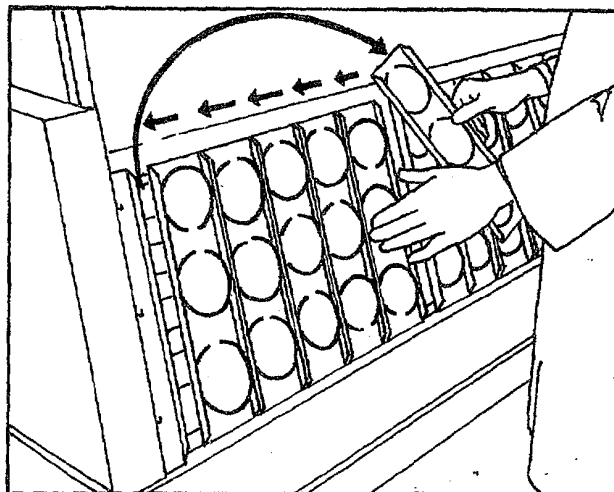


FIG. 6 Daily Horizontal Sample Rotation

removing the replicates sequentially at intervals equal to one third of the test length.

9.7 Conclude the test when either a mutually agreed-upon number of total test hours or a mutually agreed-upon change has occurred in the test specimen or a standard test specimen. For convenience in inspecting and concluding tests during normal working hours, the following test durations are recommended:

96 h = 4 days	672 h = 4 weeks
168 h = 1 week	1008 h = 6 weeks
336 h = 2 weeks	1512 h = 9 weeks
504 h = 3 weeks	2016 h = 12 weeks

10. Interpretation of Results

10.1 This practice is intended to simulate the deterioration caused by natural weathering. However, the rate of degradation in natural weathering varies from year to year. Laboratory accelerated weathering is more consistent in rate. Therefore, in comparing natural and laboratory weathering one should not attempt to predict the number of hours of laboratory exposure that might equal a year of natural weathering. Even if the details of the natural exposure are fully reported as required by 5.3, any time to time comparison would apply only to a given year at a particular site.

10.2 It is possible to correlate the deterioration resulting from a period of natural weathering, such as 1 year, to the deterioration resulting from a period of laboratory accelerated weathering. Spearman rank correlation, which is dis-

cussed in most basic statistical texts, has proved to be a useful method for this. A minimum of seven variables must be ranked by both test methods to obtain statistical validity from Spearman rank correlation. Ranking 10 to 15 variables by both methods will improve the reliability of such correlations.

10.3 It is frequently not possible to predict how many hours or weeks of accelerated testing will yield the best correlation to some period of outdoor testing. Therefore, laboratory tests should be measured periodically, such as weekly or biweekly, so that the correlation coefficient may be calculated at several points during the accelerated test.

11. Report

11.1 The report shall include the following:

11.1.1 Manufacturer and model of fluorescent UV/condensation apparatus,

11.1.2 Manufacturer's designation for the fluorescent UV lamp, the wavelength (nm) at which peak emission occurs, and the short wavelength at which 1 % of peak emission occurs (for example, FS-40 313/280 nm),

11.1.3 Cycle of UV exposure time and temperature, condensation time and temperature (for example 4 h UV/60°C, 4 h CON/50°C),

11.1.4 Total exposure time,

11.1.5 Special conditions of test, such as rotation of test specimens, and

11.2 A sample report form is shown in Fig. 5.

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RELATED MATERIAL

Any documents included in this section that are marked "proposed" are published for information only. They have received the approval of the sponsoring technical committee for publication as "proposed" but have not been officially accepted by the Society. Comments are solicited and should be addressed to ASTM, 1916 Race St., Philadelphia, Pa. 19103.



List by Subjects

1992 ANNUAL BOOK OF ASTM STANDARDS, VOLUME 06.02

PAINT—PIGMENTS, RESINS, AND POLYMERS; CELLULOSE

Since the standards in this book are arranged in alphanumerical sequence, no page numbers are given in this list. The standards listed in *italics* are related documents included for information only and do not appear in this volume.

For List by Subjects of Volumes 06.01 and 06.03, see pp. xxiv and 1100
A complete Subject Index begins on p. 1133

PIGMENT SPECIFICATIONS

Inert or Low Hiding Pigments

D 3619-77 (1989)	Aluminum Silicate (Anhydrous)
D 603-66 (1989)	Aluminum Silicate (Hydrous)
D 602-81 (1991) ^{e1}	Barium Sulfate
D 1199-86 (1991) ^{e1}	Calcium Carbonate
D 604-81 (1989)	Diatomaceous Silica
D 605-82 (1989)	Magnesium Silicate (Talc)
D 867-81 (1986) ^{e1}	Pumice
§D 607-82 (1987) ^{e1}	Wet Ground Mica

White Pigments

D 4288-83 (1989) ^{e1}	Calcium Borosilicate
§D 476-84 (1989)	Titanium Dioxide
D 81-87	White Basic Lead Carbonate
D 4462-85 (1989)	Zinc Hydroxy Phosphite
D 79-86	Zinc Oxide

Black Pigments

D 210-81a (1991) ^{e1}	Bone Black
D 561-82 (1989)	Carbon Black
D 769-87 (1991) ^{e1}	Iron Oxide, Synthetic Black
D 209-81 (1989)	Lampblack

Bronze and Miscellaneous Metallic Pigments

D 962-81 (1986) ^{e1}	Aluminum Powder and Paste
D 267-82 (1987) ^{e1}	Bronze Powder, Gold
D 964-65 (1989)	Copper Powder, Antifouling
§D 912-81 (1986)	Cuprous Oxide, Antifouling
D 911-87	Mercuric Oxide, Antifouling
D 520-84 (1989)	Zinc Dust

Blue Pigments

D 963-81 (1986) ^{e2}	Copper Phthalocyanine Blue
D 261-75 (1987) ^{e1}	Iron Blue
D 262-81 (1987) ^{e1}	Ultramarine Blue

Green Pigments

§D 212-87	Chrome Green
D 263-75 (1987)	Chrome Oxide Green
D 3021-82 (1987) ^{e1}	Phthalocyanine Green

Yellow, Orange, Brown Pigments

D 1649-82 (1987) ^{e1}	Chromate, Strontium
D 478-86 (1991) ^{e1}	Chromate, Zinc Yellow
D 211-67 (1989) ^{e1}	Chrome Yellow and Chrome Orange
§D 768-81 (1987) ^{e1}	Iron Oxide, Hydrated Yellow

§ Approved for use by agencies of the Department of Defense and, if indicated on the standard, replaces corresponding Federal or Military document. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

LIST BY SUBJECTS, VOLUME 06.02

D 3722 - 82 (1991) [†]	Iron Oxide, Natural Red and Brown
D 3724 - 82 (1987) [†]	Iron Oxide, Synthetic Brown
§D 2218 - 67 (1989) [†]	Molybdate Orange
D 85 - 87 (1991) [†]	Ochre
D 765 - 87 (1991) [†]	Sienna, Raw and Burnt
D 1648 - 86	Silicochromate, Basic Lead
D 763 - 81 (1988) [†]	Umber, Raw and Burnt

Red Pigments

D 3722 - 82 (1987) [†]	Iron Oxides, Natural Red and Brown
D 3721 - 83 (1991) [†]	Iron Oxide, Synthetic Red
D 475 - 67 (1989)	Para, Pure Red Toner
D 83 - 84 (1989)	Red Lead
§D 656 - 87	Toluidine, Pure Red Toner

PIGMENT TEST METHODS

Analytical Tests

Test Methods for:

D 305 - 84 (1990) [†]	Acetone Extract in Black Pigments, Determination of
§D 480 - 88	Aluminum Powders and Pastes, Flaked, Sampling and Testing
D 718 - 86 (1991) [†]	Aluminum Silicate Pigment, Analysis of
§D 2350 - 90	Antimony Oxide in White Pigment Separated from Solvent-Reducible Paints, Determination of
D 715 - 86 (1991) [†]	Barium Sulfate Pigment, Analysis of
§D 1135 - 86 (1991) [†]	Blue Pigments, Chemical Analysis of
D 4487 - 90	Calcium Borosilicate, Analysis of
§D 126 - 87 (1991) [†]	Chromium, Yellow, Orange, and Green Pigments, Analysis of
§D 185 - 84 (1989) [†]	Coarse Particles in Pigments, Pastes, and Paints
§D 1208 - 84 (1989) [†]	Common Properties of Certain Pigments
§D 283 - 84 (1990) [†]	Copper and Dry Cuprous Oxide Pigments, Chemical Analysis of
D 3872 - 86 (1991) [†]	Ferrous Iron in Iron Oxides, Determination of
§D 280 - 81 (1987)	Hygroscopic Moisture (and Other Volatile Matter) in Pigments
D 4358 - 84 (1990) [†]	Lead and Chromium in Air Particulate Filter Samples of Lead Chromate Pigment Dusts
§D 717 - 86 (1991) [†]	Magnesium Silicate Pigment, Analysis of
D 284 - 88	Mercuric Oxide Pigment, Chemical Analysis of
D 716 - 86 (1991) [†]	Mica Pigment, Evaluating
D 3256 - 86 (1991) [†]	Phthalocyanine Blue and Green Pigments, Chemical Analysis of
D 49 - 83 (1990) [†]	Red Lead, Chemical Analysis of
§D 970 - 86 (1991) [†]	Red Pigments, Para and Toluidine
D 719 - 86	Silica, Diatomaceous Pigment, Analysis of
D 1844 - 86 (1991) [†]	Silicochromate, Basic Lead, Chemical Analysis of
D 1845 - 86 (1991) [†]	Strontium Chromate Pigment, Chemical Analysis of
D 2351 - 90	Sulfide Sulfur in White Pigment Separated from Solvent-Reducible Paints, Determination of
D 2352 - 85 (1990) [†]	Sulfur Dioxide in White Pigment Separated from Solvent-Reducible Paints, Determination of
D 3720 - 84	Titanium Dioxide Pigments, Ratio of Anatase to Rutile by X-Ray Diffraction
D 3926 - 80 (1991) [†]	Titanium Dioxide Slurries, Percent Solids in
D 2742 - 79	Tribasic Lead Phosphosilicate, Chemical Analysis of (Discontinued (1990)†)
D 2448 - 85 (1989)	Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment
§D 1301 - 91	White Lead Pigments, Chemical Analysis of
§D 1394 - 76 (1991) [†]	White Titanium Pigments, Chemical Analysis of
§D 3280 - 85 (1990) [†]	White Zinc Pigments, Chemical Analysis of
§D 50 - 90	Yellow, Orange, Red, and Brown Pigments, Chemical Analysis of
D 521 - 81	Zinc Dust, Chemical Analysis of
D 4450 - 85 (1990) [†]	Zinc Hydroxy Phosphite, Analysis of
§D 444 - 88	Zinc Yellow (Zinc Chromate), Chemical Analysis of

Practice for:

§D 34 - 91	White Pigments, Chemical Analysis of
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Guide for:

D 4139 - 82 (1991) [†]	Volatile and Nonvolatile Content of Pigments, Determining
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General Physical Tests

Test Methods for:

D 1155 - 89	Glass Spheres, Roundness
D 1214 - 89	Glass Spheres, Sieve Analysis
D 1483 - 84 (1989) [†]	Oil Absorption of Pigments by Gardner-Coleman Method

† Although this standard has been officially withdrawn from Society approval, a brief description is included for information only.

LIST BY SUBJECTS, VOLUME 06.02

Test Methods for:

§D 281 - 84 (1989)
D 3360 - 80 (1989)
§D 153 - 84 (1989)⁶¹

Oil Absorption of Pigments by Spatula Rub-Out
Particle Size Distribution of Common White Extender Pigments
Specific Gravity of Pigments

Practice for:

D 1366 - 86 (1991)⁶¹

Particle Size Characteristics of Pigments, Reporting

Color and Opacity Tests

Test Methods for:

§D 279 - 87 (1991)⁶¹
D 3022 - 84 (1989)⁶¹
§D 387 - 86
D 2745 - 89
§D 332 - 87 (1991)⁶¹

Bleeding of Pigments
Color and Strength of Color Pigments by Use of a Miniature Sandmill
Color and Strength of Color Pigments with a Mechanical Muller
Tinting Strength of White Pigments, Relative, by Reflectance Measurements
Tinting Strength of White Pigments, Relative, by Visual Observation

RESINS AND POLYMERS

Specifications for:

§D 207 - 55 (1987)
D 784 - 83 (1987)
D 237 - 91
D 360 - 89

Lac, Dry Bleached
Orange Shellac and Other Indian Lacs for Electrical Insulation
Orange Shellac and Other Lacs
Shellac Varnishes

General

Guide for:

D 4277 - 83 (1988)⁶¹
D 4142 - 89
D 4143 - 89
D 4368 - 89

Amino Resins, Testing
Epoxy Resins, Testing
Latex Vehicles, Testing
Poly(Vinyl Chloride) Resins, Testing

Test Methods for:

D 1979 - 91
§D 29 - 81 (1987)⁶¹
D 365 - 84 (1989)⁶¹
D 411 - 83 (1987)
D 1650 - 91
D 1439 - 83a (1989)⁶¹
D 509 - 70 (1987)

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1992 ANNUAL BOOK OF ASTM STANDARDS, VOLUME 06.03

PAINT—FATTY OILS AND ACIDS, NAVAL STORES, SOLVENTS, MISCELLANEOUS; AROMATIC HYDROCARBONS

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§ Approved for use by agencies of the Department of Defense, and if indicated on the standard, replaces corresponding Federal or Military document. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

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† Although this standard has been officially withdrawn from Society approval, a brief description is included for information only.

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D 1728 - 83	Glycerin, Phthalate Ester Color of High Gravity (Discontinued 1991†)
D 3804 - 86 (1991) ⁶¹	Iron in Paint Driers by EDTA Method
D 2374 - 85 (1990) ⁶¹	Lead in Paint Driers by EDTA Method
D 2375 - 85 (1990) ⁶¹	Manganese in Paint Driers by EDTA Method
D 3125 - 83 (1987)	Monomethyl Ether of Hydroquinone in Colorless Monomeric Acrylate Esters and Acrylic Acid
D 2999 - 85	Monopentaerythritol in Commercial Pentaerythritol by Gas Chromatography (Discontinued 1989†— Replaced by Test Method D 2195)
§D 564 - 87 (1991) ⁶¹	Paint Driers, Liquid
D 2195 - 84	Pentaerythritol
D 3989 - 81a (1990) ⁶¹	Rare Earth Metals, Total, in Paint Driers by EDTA Method
D 3962 - 80 (1984) ⁶¹	Styrene Analysis by Gas Chromatography
D 2119 - 87	Styrene Monomer, Aldehydes in
D 2120 - 87	Styrene Monomer, <i>p</i> -tert-Butylcatechol Inhibitor in
D 2340 - 82 (1987) ⁶¹	Styrene Monomer, Peroxides in
D 2121 - 84	Styrene Monomer, Polymer Content of
D 1721 - 84 (1988) ⁶¹	Tricresyl Phosphate, Permanganate Time of
D 1399 - 90	Tricresyl Phosphate, Unsaponifiable Content of
§D 1468 - 84 (1988) ⁶¹	Tricresyl Phosphate, Volatile Matter in
§D 1638 - 74 ⁶¹	Urethane Foam Isocyanate Raw Materials (Discontinued 1991†)
D 3988 - 85 (1990) ⁶¹	Vanadium in Paint Driers by EDTA Method
D 2191 - 84	Vinyl Acetate, Acetaldehyde Content of
D 2086 - 84	Vinyl Acetate and Acetaldehyde, Acidity in
D 2193 - 84	Vinyl Acetate, Hydroquinone in
D 1631 - 85	Water in Phenol and Related Materials by the Iodine Reagent Method
D 3969 - 85 (1990) ⁶¹	Zirconium in Paint Driers by EDTA Method

Terminology Relating to:

D 16 - 91	Paint, Varnish, Lacquer, and Related Products
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NAVAL STORES

Rosin

Test Methods for:

D 465 - 82 (1987) ⁶¹	Acid Number of Rosin
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Test Methods for:

D 1063 - 51 (1987)	Ash in Rosin
D 1585 - 82	Fatty Acids Content of Tall Oil Rosin
§D 1064 - 58 (1981)	Iron in Rosin
D 3008 - 90	Resin Acids in Rosin by Gas Chromatography
D 1240 - 82	Rosin Acids in Fatty Acids
D 509 - 70 (1987)	Sampling and Grading Rosin
D 464 - 91	Saponification Number of Rosin
§E 28 - 67 (1982) ^{ei}	Softening Point by Ring-and-Ball Apparatus
D 269 - 52 (1987) ^{ei}	Toluene-Insoluble Solid Matter in Rosin (Chiefly Sand, Chips, Dirt, and Bark)
D 1065 - 82	Unsaponifiable Matter in Rosin
D 889 - 58 (1987)	Volatile Oil in Rosin

AROMATIC HYDROCARBONS AND RELATED MATERIALS

(see gray-edge pages 523 to 797 of Volume 06.03)

Specifications for:

D 3264 - 86	Aniline, Industrial Grade
D 836 - 84	Benzene, Industrial Grade (Discontinued 1991†)
D 835 - 90	Benzene-485, Refined (Nitration Grade)
D 2359 - 90	Benzene-535, Refined
D 4734 - 90	Benzene-545, Refined
D 3055 - 90	Cyclohexane 995
D 4077 - 91	Isopropylbenzene (Cumene)
D 3504 - 87	Maleic Anhydride
D 2439 - 91	Phenol, Refined
D 2403 - 91	Phthalic Anhydride-1308, Refined
D 2323 - 84 (1989) ^{ei}	Pyridine, Refined
D 2827 - 88 ^{ei}	Styrene Monomer 996
§D 362 - 84	Toluene, Industrial Grade (Discontinued 1991†)
D 841 - 90	Toluene, Nitration Grade
D 5211 - 91	Xylene, Feedstock for <i>p</i> -Xylene
D 843 - 90	Xylene, Nitration Grade
D 846 - 84	Xylene, Ten-Degree (Discontinued 1991†)
D 4076 - 86 (1990)	<i>o</i> -Xylene 950
D 5136 - 90	<i>p</i> -Xylene, High Purity

Test Methods for:

§D 847 - 91	Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons
D 848 - 81 (1989) ^{ei}	Acid Wash Color of Industrial Aromatic Hydrocarbons
D 2119 - 87	Aldehydes in Styrene Monomer
§D 2935 - 91	Apparent Density of Industrial Aromatic Hydrocarbons
D 4492 - 85 (1989) ^{ei}	Benzene Analysis by Gas Chromatography
D 4534 - 89	Benzene Content of Cyclic Products by Gas Chromatography
D 1492 - 91	Bromine Index of Aromatic Hydrocarbons by Coulometric Titration
D 2324 - 81 (1989) ^{ei}	Carbon Disulfide in Benzene
D 5194 - 91	Chloride, Trace, in Liquid Aromatic Hydrocarbons
D 3627 - 82 (1987) ^{ei}	Color of Cresylic Acids ("C" Series Standards)
D 4789 - 88	Color of 4,4'-Isopropylidenediphenol (Bisphenol A) in Solution
D 3366 - 90	Color of Maleic Anhydride and Phthalic Anhydride in the Molten State and After Heating
D 1686 - 81 (1990)	Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale)
D 4590 - 86	Colorimetric Determination of <i>p-tert</i> -Butylcatechol in Styrene Monomer by Addition of Alcoholic NaOH
§D 849 - 88	Copper Corrosion of Industrial Aromatic Hydrocarbons
D 3439 - 89	Cresylate Solutions, Alkaline, from Petroleum Sources
§D 3505 - 84 ^{ei}	Density or Relative Density of Pure Liquid Chemicals
§D 850 - 91	Distillation of Industrial Aromatic Hydrocarbons and Related Materials
D 2232 - 81 (1986) ^{ei}	Evaporating Residue of Naphthalene
D 1015 - 89	Freezing Points of High-Purity Hydrocarbons
D 2870 - 86	Gel Time of Tar Acids
§D 853 - 91	Hydrogen Sulfide and Sulfur Dioxide Content (Qualitative) of Industrial Aromatic Hydrocarbons
D 5060 - 90	Impurities in High-Purity Ethylbenzene by Gas Chromatography, Determining
D 4961 - 89	Impurities, Major Organic, in Phenol Produced by the Cumene Process by Gas Chromatographic Analysis
D 2120 - 87	Inhibitor, <i>p-tert</i> -Butylcatechol, in Styrene Monomer
D 3760 - 79 (1984)	Isopropylbenzene (Cumene) by Gas Chromatography
D 2930 - 80 (1989) ^{ei}	Maleic Acid in Maleic Anhydride by Potentiometric Titration
§D 2360 - 82 (1987) ^{ei}	Monocyclic Aromatic Hydrocarbons, Trace Impurities in, by Gas Chromatography
D 4589 - 91	Nitrobenzene in Aniline
D 2340 - 82 (1987) ^{ei}	Peroxides in Styrene Monomer
D 2121 - 90	Polymer Content of Styrene Monomer

LIST BY SUBJECTS, VOLUME 05.03

Test Methods for:

- D 3054 - 81 (1985) Purity and Benzene Content of Cyclohexane by Gas Chromatography
- D 1016 - 89 Purity of Hydrocarbons from Freezing Points
- D 4471 - 85 (1989)^{el} Pyridine Bases in Cresylic Acid by Direct Titration
- D 2031 - 84 (1989)^{el} Reducing Substances in Refined Pyridine
- D 852 - 87 (1991) Solidification Point of Benzene
- D 1493 - 90 Solidification Point of Industrial Organic Chemicals
- D 4493 - 89 Solidification Point of 4,4'-Isopropylidenediphenol (Bisphenol A)
- D 5135 - 90 Styrene, Analysis of, by Capillary Gas Chromatography
- D 3962 - 80 (1989)^{el} Styrene, Analysis of, by Gas Chromatography
- D 3799 - 89 Styrene Purity by Freezing Point Method
- D 3961 - 89 Sulfur, Trace Quantities, in Liquid Aromatic Hydrocarbons by Oxidative Microcoulometry
- D 3626 - 85 (1990)^{el} Tar Acid Composition by Gas Liquid Chromatography
- D 1685 - 86 (1990) Thiophene in Benzene, Traces of, Using Isatin and Spectrophotometry
- D 4735 - 87 (1991)^{el} Thiophene in Refined Benzene, Trace, by Gas Chromatography
- §D 1555 - 91 Volume and Weight of Industrial Aromatic Hydrocarbons, Calculation of
- D 1631 - 85 (1989)^{el} Water in Phenol and Related Materials by the Iodine Reagent Method
- D 2030 - 84 (1989)^{el} Water Solubility of Refined Pyridine
- D 2306 - 81 (1985) Xylene Isomer Analysis by Gas Chromatography
- D 3797 - 88 o-Xylene by Gas Chromatography
- D 3798 - 89 p-Xylene by Gas Chromatography
- D 1217 - 86 Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer (see Vol 05.01)
- D 1218 - 87 Refractive Index and Refractive Dispersion of Hydrocarbon Liquids (see Vol 05.01)

Practices for:

- D 3436 - 91 Aniline, Sampling and Handling
- D 4297 - 89 4,4'-Isopropylidenediphenol (Bisphenol A), Sampling and Handling
- D 3437 - 89 Liquid Cyclic Products, Sampling and Handling
- D 3438 - 89 Naphthalene, Maleic Anhydride, and Phthalic Anhydride, Sampling and Handling
- D 3852 - 90 Phenol and Cresylic Acid, Sampling and Handling

Guide for:

- D 4588 - 87 p-Xylene, Analysis of (Discontinued 1992+—Replaced by Specification D 5136)

Terminology of:

- D 4790 - 89a Aromatic Hydrocarbons and Related Chemicals

GENERAL STANDARDS

Specifications for:

- D 1193 - 91 Reagent Water
- E 100 - 81 (1986) ASTM Hydrometers (see Vol 14.03)
- E 1 - 90 ASTM Thermometers (see Vol 14.03)
- E 133 - 86 Distillation Equipment (see Vol 14.02)

Test Methods for:

- E 299 - 90 Peroxides in Organic Solvents, Trace Amounts
- E 70 - 90 pH of Aqueous Solutions with the Glass Electrode (see Vol 15.05)
- E 200 - 86 Standard Solutions for Chemical Analysis, Preparation, Standardization, and Storage of (see Vol 15.05)

Practices for:

- E 300 - 86 Industrial Chemicals, Sampling
- E 691 - 87 Interlaboratory Study to Determine the Precision of a Test Method, Conducting

Definitions of Terms Relating to:

- E 12 - 70 (1986) Density and Specific Gravity of Solids, Liquids, and Gases (see Vols 04.02 and 15.05)

METRIC PRACTICE

Practice for:

- §E 380 - 91 Use of the International System of Units (SI) (the Modernized Metric System) (Excerpts) (see Related Material section)

Excerpts from Standard Practice for Use of the International System of Units (SI) (the Modernized Metric System)¹

Following are excerpts from *Standard Practice for Use of the International System of Units (SI) the Modernized Metric System E 380*, which is available as a separate publication and which appears in its entirety in Volume 14.02. Deleted are Appendixes X1, X2, X3, and X4. Added is a table of selected conversion factors from Appendix X3.

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1. Scope

1.1 This standard gives guidance for application of The International System of Units (the modernized metric system) developed and maintained by the General Conference on Weights and Measures (abbreviated CGPM from the official French name *Conférence Générale des Poids et Mesures*). The name International System of Units and the international abbreviation SI² were adopted by the 11th CGPM in 1960.

1.2 Information is included on SI, a limited list of non-SI units recognized for use with SI units, and a list of conversion factors from non-SI to SI units, together with general guidance on proper style and usage.

1.3 It is hoped that an understanding of the system and its characteristics, and careful use according to this standard,

will help to avoid the degradation that has occurred in all older measurement systems.

2. SI Units and Symbols

2.1 *Classes of Units*—SI units are divided into three classes:

base units
supplementary units
derived units

2.2 *Base Units*—SI is based on seven well-defined units (see Table 1) which by convention are regarded as dimensionally independent.

2.3 *Supplementary Units*—This class contains two units, the radian and the steradian (see Table 2). At the time of the introduction of the International System, the 11th CGPM left open the question of the nature of these supplementary units. Considering that plane angle is generally expressed as the ratio between two lengths and solid angle as the ratio between an area and the square of length, the CIPM (1980) specified that in the International System the quantities plane angle and solid angle should be considered as dimen-

¹ This standard is under the jurisdiction of ASTM Committee E-43 on Metric Practice and is the direct responsibility of Subcommittee E43.10 on Standards.

Current edition approved Oct. 3, 1991. Published December 1991. Originally published in 1964 without designation as ASTM *Metric Practice Guide*, revised 1966. Adopted as standard 1968. Last previous edition E 380 - 89.

² From the French name, *Le Système International d'Unités*.

TABLE 1 Base SI Units

Quantity ³	Unit	Symbol
length	metre	m
mass	kilogram	kg
time	second	s
electric current	ampere	A
thermodynamic temperature ⁴	kelvin	K
amount of substance	mole	mol
luminous intensity	candela	cd

⁴ For a discussion of Celsius temperature see 3.4.2.

TABLE 2 Supplementary SI Units

Quantity ³	Unit	Symbol
plane angle	radian	rad
solid angle	steradian	sr

sionless derived quantities. Therefore, the supplementary units radian and steradian are to be regarded as dimensionless derived units which may be used or omitted in the expressions for derived units.

2.4 Derived Units:

2.4.1 Derived units are formed by combining base units, supplementary units, and other derived units according to the algebraic relations linking the corresponding quantities. The symbols for derived units are obtained by means of the mathematical signs for multiplication, division, and use of exponents. For example, the SI unit for velocity is the metre per second (m/s or $\text{m}\cdot\text{s}^{-1}$), and that for angular velocity is the radian per second (rad/s or $\text{rad}\cdot\text{s}^{-1}$).

2.4.2 Those derived SI units which have special names and symbols approved by the CGPM are listed in Table 3.

2.4.3 It is frequently advantageous to express derived units in terms of other derived units with special names; for example, the SI unit for electric dipole moment is usually expressed as $\text{C}\cdot\text{m}$ instead of $\text{A}\cdot\text{s}\cdot\text{m}$.

2.4.4 Some common derived units are listed in Table 4.

2.5 SI Prefixes (see 3.2 for application):

2.5.1 The prefixes and symbols listed in Table 5 are used to form names and symbols of the decimal multiples and submultiples of the SI units except for kilogram.

2.5.2 Unit of Mass—Among the base and derived units of SI, the unit of mass (kilogram) is the only one whose name, for historical reasons, contains a prefix. Names of decimal multiples and submultiples of the unit of mass are formed by attaching prefixes to the word *gram* (g).

2.5.3 These prefixes or their symbols are directly attached to names or symbols of units, forming multiples and submultiples of the units. In strict terms these must be called "multiples and submultiples of SI units," particularly in discussing the coherence of the system (see Section 5). In common parlance, the base units and derived units, along with their multiples and submultiples, are all called SI units.

3. Application of the Metric System

3.1 General—SI is the form of the metric system that is preferred for all applications. It is important that this modernized form of the metric system be thoroughly under-

³ "Quantity" as used in the headings of the tables of this standard means measurable attribute of phenomena or matter.

TABLE 3 Derived SI Units with Special Names

Quantity ³	Unit	Symbol	Formula
frequency (of a periodic phenomenon)	hertz	Hz	1/s
force	newton	N	$\text{kg}\cdot\text{m/s}^2$
pressure, stress	pascal	Pa	N/m^2
energy, work, quantity of heat	joule	J	$\text{N}\cdot\text{m}$
power, radiant flux	watt	W	J/s
quantity of electricity, electric charge	coulomb	C	$\text{A}\cdot\text{s}$
electric potential, potential difference, electromotive force	volt	V	W/A
electric capacitance	farad	F	C/V
electric resistance	ohm	Ω	V/A
electric conductance	siemens	S	A/V
magnetic flux	weber	Wb	$\text{V}\cdot\text{s}$
magnetic flux density	tesla	T	Wb/m^2
inductance	henry	H	Wb/A
Celsius temperature	degree Celsius ⁴	$^{\circ}\text{C}$	K [see 3.4.2]
luminous flux	lumen	lm	$\text{cd}\cdot\text{sr}$
illuminance	lux	lx	lm/m^2
activity (of a radionuclide)	becquerel	Bq	1/s
absorbed dose ⁵	gray	Gy	J/kg
dose equivalent	sievert	Sv	J/kg

⁴ Inclusion in the table of derived SI units with special names approved by CIPM in 1978.

⁵ Related quantities using the same unit are: specific energy imparted, kerma, and absorbed dose index.

stood and properly applied. Obsolete metric units and practices are widespread, particularly in those countries that long ago adopted the metric system, and much usage is improper. This section gives guidance concerning the limited number of cases in which units outside SI are appropriate, used, and makes recommendations concerning usage and style.

3.2 Application of SI Prefixes:

3.2.1 General—In general the SI prefixes (2.5) should be used to indicate orders of magnitude, thus eliminating nonsignificant digits and leading zeros in decimal fractions, and providing a convenient alternative to the powers-of-ten notation preferred in computation. For example:

12 300 mm becomes 12.3 m
 12.3×10^3 m becomes 12.3 km
 $0.00123 \mu\text{A}$ becomes 1.23 nA

3.2.2 Selection—When expressing a quantity by a numerical value and a unit, a prefix should preferably be chosen so that the numerical value lies between 0.1 and 1000. To minimize variety, it is recommended that prefixes representing 1000 raised to an integral power be used. However, three factors may justify deviation from the above:

3.2.2.1 In expressing area and volume, the prefixes hecto-, deka-, deci-, and centi- may be required, for example, square hectometre, cubic centimetre.

3.2.2.2 In tables of values of the same quantity, or in a discussion of such values within a given context, it is generally preferable to use the same unit multiple throughout.

3.2.2.3 For certain quantities in particular applications, one particular multiple is customarily used. For example, the millimetre is used for linear dimensions in mechanical engineering drawings even when the values lie far outside the range 0.1 to 1000 mm; the centimetre is often used for body

absorbed dose
 acceleration
 angular accel.
 angular veloc.
 area
 concentration
 current dens.
 density, mas.
 electric char.
 electric field
 electric flux
 energy dens.
 entropy
 exposure
 heat capac.
 heat flux
 irradiance
 luminance
 magnetic
 molar ene.
 molar ent.
 molar heat
 moment
 permeabil.
 permittivi.
 power
 radianc.
 radiant
 specific
 specific
 specific
 surface
 thermal
 velocity
 viscosi.
 viscosi.
 volume
 wave

See 3.4.4.

measurements
 3.2.3 Prefixe
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TABLE 4 Some Common Derived Units of SI

Quantity ^a	Unit	Symbol
absorbed dose rate	gray per second	Gy/s
acceleration	metre per second squared	m/s ²
angular acceleration	radian per second squared	rad/s ²
angular velocity	radian per second	rad/s
area	square metre	m ²
concentration (of amount of substance)	mole per cubic metre	mol/m ³
current density	ampere per square metre	A/m ²
density, mass	kilogram per cubic metre	kg/m ³
electric charge density	coulomb per cubic metre	C/m ³
electric field strength	volt per metre	V/m
electric flux density	coulomb per square metre	C/m ²
energy density	joule per cubic metre	J/m ³
entropy	joule per kelvin	J/K
exposure (X and gamma rays)	coulomb per kilogram	C/kg
heat capacity	joule per kelvin	J/K
heat flux density	watt per square metre	W/m ²
irradiance		
luminance	candela per square metre	cd/m ²
magnetic field strength	ampere per metre	A/m
molar energy	joule per mole	J/mol
molar entropy	joule per mole kelvin	J/(mol·K)
molar heat capacity	joule per mole kelvin	J/(mol·K)
moment of force ⁴	newton metre	N·m
permeability (magnetic)	henry per metre	H/m
permittivity	farad per metre	F/m
power density	watt per square metre	W/m ²
radiance	watt per square metre steradian	W/(m ² ·sr)
radiant intensity	watt per steradian	W/sr
specific heat capacity	joule per kilogram kelvin	J/(kg·K)
specific energy	joule per kilogram	J/kg
specific entropy	joule per kilogram kelvin	J/(kg·K)
specific volume	cubic metre per kilogram	m ³ /kg
surface tension	newton per metre	N/m
thermal conductivity	watt per metre kelvin	W/(m·K)
velocity	metre per second	m/s
viscosity, dynamic	pascal second	Pa·s
viscosity, kinematic	square metre per second	m ² /s
volume	cubic metre	m ³
wave number	1 per metre	1/m

^a See 3.4.4.

measurements and clothing sizes.

3.2.3 *Prefixes in Compound Units*⁴—It is recommended that only one prefix be used in forming a multiple of a compound unit. Normally the prefix should be attached to a unit in the numerator. One exception to this is when the kilogram occurs in the denominator.

Examples:

V/m, not mV/mm, and MJ/kg, not kJ/g

3.2.4 *Compound Prefixes*—Compound prefixes, formed by the juxtaposition of two or more SI prefixes are not to be used. For example, use

1 nm, not 1 mμm
1 pF, not 1 μμF

If values are required outside the range covered by the prefixes, they should be expressed by using powers of ten applied to the base unit.

3.2.5 *Powers of Units*—An exponent attached to a symbol containing a prefix indicates that the multiple or submultiple of the unit (the unit with its prefix) is raised to the power expressed by the exponent. For example:

⁴ A compound unit is a derived unit that is expressed in terms of two or more units rather than by a single special name.

$$\begin{aligned}
 1 \text{ cm}^3 &= (10^{-2} \text{ m})^3 = 10^{-6} \text{ m}^3 \\
 1 \text{ ns}^{-1} &= (10^{-9} \text{ s})^{-1} = 10^9 \text{ s}^{-1} \\
 1 \text{ mm}^2/\text{s} &= (10^{-3} \text{ m})^2/\text{s} = 10^{-6} \text{ m}^2/\text{s}
 \end{aligned}$$

3.2.6 *Calculations*—Errors in calculations can be minimized if the base and the coherent derived SI units are used and the resulting numerical values are expressed in powers-of-ten notation instead of using prefixes.

3.3 Other Units:

3.3.1 *Units from Different Systems*—To assist in preserving the advantage of SI as a coherent system, it is advisable to minimize the use with it of units from other systems. Such use should be limited to units listed in this section.

3.3.2 Units in Use with SI (see Table 6):

3.3.2.1 *Time*—The SI unit of time is the second. This unit is preferred and should be used if practical, particularly when technical calculations are involved. In cases where time relates to life customs or calendar cycles, the minute, hour, day, and other calendar units may be necessary. For example, vehicle speed will normally be expressed in kilometres per hour.

3.3.2.2 *Plane Angle*—The SI unit for plane angle is the radian. Use of the degree and its decimal submultiples is permissible when the radian is not a convenient unit. Use of

TABLE 5 SI Prefixes

Multiplication Factor	Prefix	Symbol
1 000 000 000 000 000 000 = 10^{18}	exa	E
1 000 000 000 000 000 = 10^{15}	peta	P
1 000 000 000 000 = 10^{12}	tera	T
1 000 000 000 = 10^9	giga	G
1 000 000 = 10^6	mega	M
1 000 = 10^3	kilo	k
100 = 10^2	hecto ^A	h
10 = 10^1	deka ^A	da
0.1 = 10^{-1}	deci ^A	d
0.01 = 10^{-2}	centi ^A	c
0.001 = 10^{-3}	milli	m
0.000 001 = 10^{-6}	micro	μ
0.000 000 001 = 10^{-9}	nano	n
0.000 000 000 001 = 10^{-12}	pico	p
0.000 000 000 000 001 = 10^{-15}	femto	f
0.000 000 000 000 000 001 = 10^{-18}	atto	a

^A To be avoided where practical, except as noted in 3.2.2.

the minute and second is discouraged except for special fields such as cartography.

3.3.2.3 Area—The SI unit of area is the square metre (m^2). The hectare (ha) is a special name for square hectometre (hm^2). Large land or water areas are generally expressed in hectares or in square kilometres (km^2).

3.3.2.4 Volume—The SI unit of volume is the cubic metre. This unit, or one of the regularly formed multiples such as the cubic centimetre, is preferred. The special name *litre* (L)⁶ has been approved for the cubic decimetre, but use of this unit is restricted to volumetric capacity, dry measure, and measure of fluids (both gases and liquids). No prefix other than milli- or micro- should be used with litre.

3.3.2.5 Mass—The SI unit of mass is the kilogram. This unit, or one of the multiples formed by attaching an SI prefix to *gram* (g), is preferred for all applications. The megagram (Mg) is the appropriate unit for measuring large masses such as have been expressed in tons. However, the name *ton* has been given to several large mass units that are widely used in commerce and technology—the long ton of 2240 lb, the short ton of 2000 lb, and metric ton of 1000 kg (also called the *tonne*). None of these terms are SI. The term *metric ton* should be restricted to commercial usage, and no prefixes should be used with it. Use of the term *tonne* is deprecated.

⁵ See Appendix XI.11.1.

⁶ The CGPM in October 1979 approved L and l as alternative symbols for litre. Since the letter symbol l can easily be confused with the numeral 1, only the symbol L is recommended for USA use.

TABLE 6 Units in Use with SI

Quantity ³	Unit	Symbol	Definition
time	minute	min	1 min = 60 s
	hour	h	1 h = 60 min = 3600 s
	day	d	1 d = 24 h = 86 400 s
	week, month, etc.	...	...
plane angle	degree	°	1° = ($\pi/180$) rad
	minute ⁴	'	1' = ($1/60$)°
	second ⁴	"	1" = ($\pi/10 800$) rad
volume	litre ⁵	L	1 L = ($\pi/648 000$) rad
	metric ton	t	1 t = 1 dm ³ = 10^{-3} m ³
	area	ha	1 ha = 10 ⁴ m ²

⁴ Use discouraged except for special fields such as cartography.

⁵ See 3.3.2.4.

3.3.3 Units in Use with SI Temporarily (see Table 7):

3.3.3.1 Energy—The SI unit of energy, the joule, together with its multiples, is preferred for all applications. The kilowatthour is widely used, however, as a measure of electric energy. This unit should not be introduced into any new areas, and eventually it should be replaced by the megajoule.

3.3.3.2 Pressure and Stress—The SI unit of pressure and stress is the pascal (newton per square metre) and with proper SI prefixes is applicable to all such measurements. Old metric gravitational units for pressure and stress such as kilogram-force per square centimetre (kgf/cm^2) shall not be used. Widespread use has been made of other non-SI units such as bar and torr for pressure, but this use is strongly discouraged. The millibar is widely used in meteorology; this usage will continue for the present in order to permit meteorologists to communicate easily within their profession. The kilopascal should be used in presenting meteorological data to the public.

3.3.4 Units and Names to Be Abandoned—A great many metric units other than those of the SI have been defined over the years. Some of these are used only in special fields; others have found broad application in countries that adopted the metric system early. Except for the special cases discussed in the previous sections, non-SI units (as well as special names for multiples or submultiples of SI units) are to be avoided. Various categories of deprecated units are discussed in 3.3.4.1 to 3.3.4.4. The lists are not intended to be complete, but only to indicate more or less prominent examples of each category.

3.3.4.1 Cgs Units—All units peculiar to the various cgs systems (measurement systems constructed by using the centimetre, gram, and second as base units) are to be avoided. Among these units are the following, defined for mechanics, fluid mechanics, and photometry: the erg, dyne, gal, poise, stokes, stilb, phot, and lambert. Further use of the cgs units of electricity and magnetism is deprecated. This statement applies to the units designated by the general abbreviations "esu" (for electrostatic cgs unit) and "emu" (for electromagnetic cgs unit), including those units that have been given special names—the gauss, oersted, maxwell, gilbert, biot, and franklin. It also applies to the unit names formed with the prefixes ab- and stat-, for example, the abampere, statvolt, etc.

3.3.4.2 Decimal Multiples of SI Units—Those multiples of SI units that cannot be handled by using the SI prefixes are deprecated. Many such examples are covered in subsection 3.3.4.1. An additional example is the angstrom (0.1 nm).

3.3.4.3 Unit Names to Be Avoided—Special names for

TABLE 7 Units in Use with SI Temporarily

Quantity ³	Unit	Symbol	Definition
energy [see 3.3.3.1]	kilowatthour	kWh	1 kWh = 3.6 MJ
cross section	barn	b	1 b = 10^{-28} m ² = 100 fm ²
pressure [see 3.3.3.2]	bar	bar	1 bar = 10^5 Pa
activity (of a radio-nuclide)	curie	Ci	1 Ci = 3.7×10^{10} Bq
exposure (X and gamma rays)	roentgen	R	1 R = 2.58×10^{-4} C/kg
absorbed dose	rad	rd	1 rd = 0.01 Gy
dose equivalent	rem	rem	1 rem = 0.01 Sv = 10 mSv

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multiples and submultiples of SI units are to be avoided except for the *litre* (3.3.2.4), *metric ton* (3.3.2.5), and *hectare* (3.3.2.3). For example, do not use:

fermi	1 fermi	= 1 fm = 10^{-15} m
micron	1 micron	= 1 μ m = 10^{-6} m
millimicron	1 millimicron	= 1 nm = 10^{-9} m
are	1 are	= 1 dam ² = 100 m ²
gamma	1 gamma	= 1 nT
(magnetic flux density)		
γ (mass)	1 γ	= 1 μ g
λ (volume)	1 λ	= 1 μ L = 1 mm ³
mho	1 mho	= 1 S
candle	1 candle	= 1 cd
candlepower	1 candlepower	= 1 cd

3.3.4.4 *Miscellaneous Units*—Other non-SI units that are deprecated include the following:

calorie
grade [1 grade = $(\pi/200)$ rad]
kilogram-force
langley (= 1 cal/cm²)
metric carat
metric horsepower
millimetre of mercury
millimetre, centimetre, metre of water

standard atmosphere
(1 atm = 101.325 kPa)
technical atmosphere
(1 at = 98.0665 kPa)
torr

3.4 Other Recommendations Concerning Units:

3.4.1 Mass, Force, and Weight:

3.4.1.1 The principal departure of SI from the gravimetric system of metric engineering units is the use of explicitly distinct units for mass and force. In SI, the name kilogram is restricted to the unit of mass, and the kilogram-force (from which the suffix *force* was in practice often erroneously dropped) should not be used. In its place the SI unit of force, the newton, is used (see Fig. 1). Likewise, the newton rather than the kilogram-force is used to form derived units which include force, for example, pressure or stress ($\text{N/m}^2 = \text{Pa}$), energy ($\text{N} \cdot \text{m} = \text{J}$), and power ($\text{N} \cdot \text{m/s} = \text{W}$).

3.4.1.2 Considerable confusion exists in the use of the term *weight* as a quantity to mean either *force* or *mass*. In commercial and everyday use, the term *weight* nearly always means mass; thus, when one speaks of a person's weight, the quantity referred to is mass. This nontechnical use of the term *weight* in everyday life will probably persist. In science and technology, the term *weight of a body* has usually meant

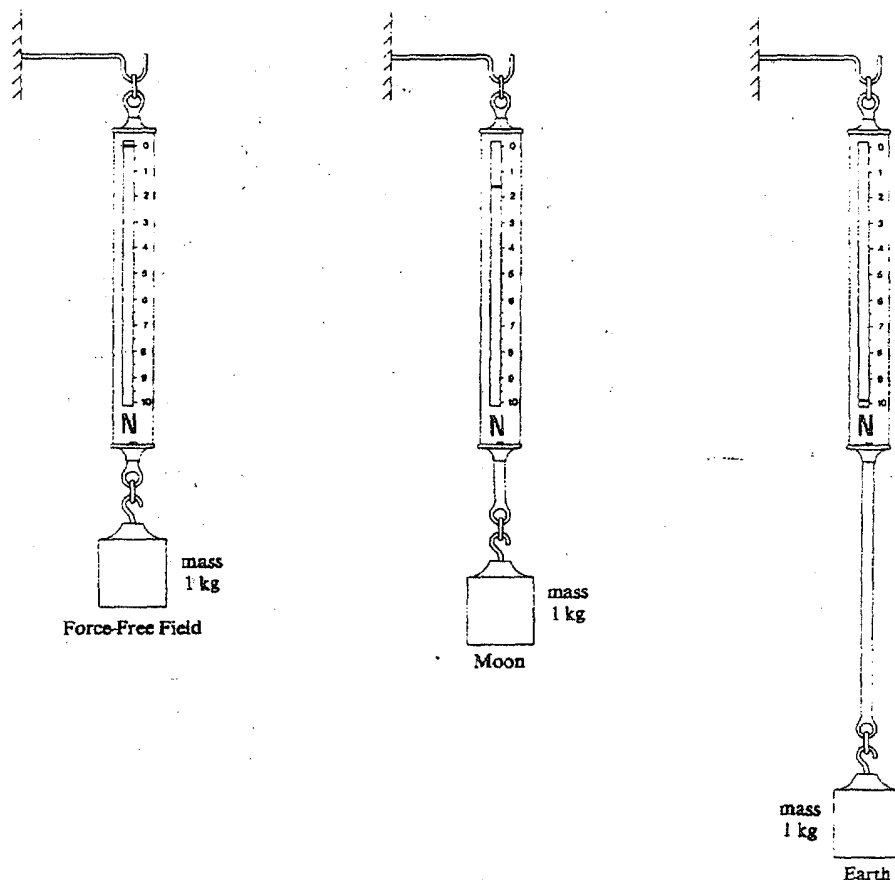


FIG. 1 Illustration of Difference Between Mass (Unit = kilogram = kg) and Force (Unit = newton = N) (see 3.4.1)

the force that, if applied to the body, would give it an acceleration equal to the local acceleration of free fall. The adjective "local" in the phrase "local acceleration of free fall" has usually meant a location on the surface of the earth; in this context the "local acceleration of free fall" has the symbol g (commonly referred to as "acceleration of gravity") with observed values of g differing by over 0.5 % at various points on the earth's surface. The use of *force of gravity* (mass times acceleration of gravity) instead of *weight* with this meaning is recommended. Because of the dual use of the term *weight* as a quantity, this term should be avoided in technical practice except under circumstances in which its meaning is completely clear. When the term is used, it is important to know whether mass or force is intended and to use SI units properly as described in 3.4.1.1, by using kilograms for mass or newtons for force.

3.4.1.3 Gravity is involved in determining mass with a balance or scale. When a standard mass is used to balance the measured mass, the effects of gravity on the two masses are equalized, but the effects of the buoyancy of air or other fluid on the two masses are generally not equalized. When a spring scale is used, the scale reading is directly related to the force of gravity. Spring scales graduated in mass units may be properly used if both the variation in acceleration of gravity and the buoyancy corrections are not significant in their use.

3.4.1.4 The use of the same name for units of force and mass causes confusion. When the non-SI units are used, a distinction should be made between *force* and *mass*, for example, lbf to denote force in gravimetric engineering units and lb for mass.

3.4.1.5 The term *load* means either mass or force, depending on its use. A load that produces a vertically downward force because of the influence of gravity acting on a mass may be expressed in mass units. Any other load is expressed in force units.

3.4.2 *Temperature*—The SI unit of thermodynamic temperature is the kelvin (K), and this unit is properly used for expressing thermodynamic temperature and temperature intervals. Wide use is also made of the degree Celsius ($^{\circ}\text{C}$), which is the SI unit used for expressing Celsius temperature and temperature intervals. The Celsius scale (formerly called centigrade) is related directly to thermodynamic temperature (kelvins) as follows:

The temperature interval one degree Celsius equals one kelvin exactly. Celsius temperature (t) is related to thermodynamic temperature (T) by the equation:

$$t = T - T_0$$

where $T_0 = 273.15 \text{ K}$ by definition.

The International Practical Temperature Scale (IPTS) must be recognized in temperature work of extreme precision. See *ASTM STP 565, Evolution of the International Practical Temperature Scale of 1968*.

3.4.3 Linear Dimensions:

3.4.3.1 Nominal dimensions name the item; no SI equivalent is required (see Section 5 for definition of "nominal value"). For example, there is nothing "1 in" about a nominal "1-in pipe," the dimensions of which should be converted as follows:

Nominal Size, inches	Outside Diameter, inches (mm)	Wall Thickness, inches (mm)		
		Sch 40	Sch 80	Sch 160
1	1.315 (33.40)	0.133 (3.38)	0.179 (4.55)	0.250 (6.35)

Likewise, a "2 by 4" is that in name only and refers to the approximate dimensions in inches of a rough-sawn piece of green lumber, the finished dimensions of which are considerably less. A $\frac{1}{4}$ -20 UNC screw thread should continue to be identified in this manner. However, the controlling dimensions of the part, such as the pitch, major, and minor diameters of a screw thread, should be converted to SI values in accordance with 4.1 and 4.2.

3.4.3.2 Surface texture should be expressed in micrometres.

3.4.4 Quantities and Units used in Rotational Mechanics

3.4.4.1 *Angle, Angular Velocity, and Angular Acceleration*. Their SI units are rad, rad/s, and rad/s^2 respectively. In accordance with Sec. 2.3, since the radian is here taken to be dimensionless, the units 1, 1/s, and $1/\text{s}^2$ are also used when appropriate.

3.4.4.2 *Moment of Force* (Torque or Bending Moment) is force times moment arm (lever arm). Its SI unit is $\text{N}\cdot\text{m}$.

3.4.4.3 *Moment of Inertia* (I) is a property of the mass distribution of a body about an axis ($I = \sum mr^2$). Its SI unit is $\text{kg}\cdot\text{m}^2$.

3.4.4.4 *Angular Momentum* (moment of momentum) is linear momentum ($\text{kg}\cdot\text{m/s}$) times moment arm (m). Its SI unit is $\text{kg}\cdot\text{m}^2/\text{s}$. For a rotating body the total angular momentum is equal to the moment of inertia I ($\text{kg}\cdot\text{m}^2$) times the angular velocity ω (rad/s or 1/s).

3.4.4.5 *Rotational Kinetic Energy* of a rotating body is equal to $\frac{1}{2}I\omega^2$. Its SI unit is J.

3.4.4.6 *Rotational Work* is equal to torque ($\text{N}\cdot\text{m}$) times angle of rotation (rad). Its SI unit is J.

3.4.4.7 *Torsional Stiffness* (torsion constant) of a body is applied torque ($\text{N}\cdot\text{m}$) divided by angle of twist (rad). Its SI unit is $\text{N}\cdot\text{m}/\text{rad}$.

3.4.4.8 *Centripetal Acceleration*, v^2/r or $\omega^2 r$, where v is the tangential linear velocity (m/s), r the radius (m), and ω the angular velocity (rad/s) is, like any other linear acceleration, measured in SI units m/s^2 .

NOTE—Centripetal Force, equal to mass times centripetal acceleration, is, like any force in SI, measured in newtons.

3.4.5 *Impact Energy Absorption*—This quantity, often incorrectly called impact resistance or impact strength, is measured in terms of the work required to break a standard specimen; the proper unit is joule.

3.4.6 *Pressure and Vacuum*—Gage pressure is absolute pressure minus ambient pressure (usually atmospheric pressure). Both gage pressure and absolute pressure are properly expressed in pascals, using SI prefixes as appropriate. Absolute pressure is never negative. Gage pressure is positive if above ambient pressure and negative if below. Pressure below ambient is often called vacuum; whenever the term *vacuum* is applied to a numerical measure it should be made clear whether negative gage pressure or absolute pressure is meant. See 3.5.5 for methods of designating gage pressure and absolute pressure.

3.4.7 Dimensionless Quantities:

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3.4.7.1 The values of so-called dimensionless quantities, as for example refractive index and relative permeability, are expressed by pure numbers. In these cases the corresponding SI unit is the ratio of the same two SI units and may be expressed by the number 1.

3.4.7.2 Terms such as percent, parts per thousand, and parts per million may also be used.

3.4.7.3 In all cases, the meaning must be unequivocal. Expressions like "The mole fraction of CO₂ in the sample was 1.2 parts per million" or "The mass fraction of CO₂ in the sample was 1.2 parts per million" are permissible, but would not be permissible if the word "mole" in the first expression or "mass" in the second expression were not present.

3.5 *Style and Usage*—Care must be taken to use unit symbols properly, and international agreement provides uniform rules. Handling of unit names varies because of language differences, but use of the rules included here will improve communications in the United States.

3.5.1 *Rules for Writing Unit Symbols:*

3.5.1.1 Unit symbols should be printed in upright type regardless of the type style used in the surrounding text.

3.5.1.2 Unit symbols are unaltered in the plural.

3.5.1.3 Unit symbols are not followed by a period except when used at the end of a sentence.

3.5.1.4 Letter unit symbols are written in lower-case (for example, cd) unless the unit name has been derived from a proper name, in which case the first letter of the symbol is capitalized (for example, W, Pa). The exception is the symbol for litre, L. Prefix symbols use either lower-case or upper-case letters as shown in 2.5.1. Symbols retain their prescribed form regardless of the surrounding typography. For symbols for use in systems with limited character sets, refer to ANSI X3.50 or ANSI/IEEE 260, as applicable. The symbols in ANSI X3.50 are intended for applications in the field of information processing, where unambiguous transmission of information between computers is required. The symbols in ANSI/IEEE 260 are generally consistent with those in ANSI X3.50 and are intended for communication between human beings. The symbols for limited character sets must never be used when the available character set permits the use of the proper general-use symbols as given in this standard.

3.5.1.5 When a quantity is expressed as a numerical value and a unit symbol, a space should be left between them. For example, use 35 mm, *not* 35mm, and 2.37 lm (for 2.37 lumens), *not* 2.37lm.

Exception: No space is left between the numerical value and the symbols for degree, minute, and second of plane angle, and degree Celsius. For example, use 45°, 20°C.

3.5.1.6 When a quantity expressed as a number and a unit is used in an adjectival sense, it is preferable to use a hyphen instead of a space between the number and the unit name or between the number and the symbol. Examples: A three-metre pole. . . The length is 3 m. . . A 35-mm film. . . The width is 35 mm. However, per 3.5.1.5 *Exception*, a 90° angle. . . an angle of 90°.

3.5.1.7 No space is used between the prefix and unit symbols.

3.5.1.8 Symbols, not abbreviations, should be used for units. For example, use "A" and not "amp" for ampere.

3.5.2 *Rules for Writing Names:*

3.5.2.1 Spelled-out unit names are treated as common nouns in English. Thus, the first letter of a unit name is not capitalized except at the beginning of a sentence or in capitalized material such as a title.

3.5.2.2 Plurals are used when required by the rules of English grammar and are normally formed regularly, for example, henries for the plural of henry. The following irregular plurals are recommended:

Singular	Plural
lux	lux
hertz	hertz
siemens	siemens

3.5.2.3 No space or hyphen is used between the prefix and unit name. There are three cases where the final vowel in the prefix is commonly omitted: *megohm*, *kilohm*, and *hectare*. In all other cases where the unit name begins with a vowel both vowels are retained and both are pronounced.

3.5.3 *Units Formed by Multiplication and Division:*

3.5.3.1 *With unit names:*

Product, use a space (preferred) or hyphen:

newton metre *or* newton-metre

In the case of the watt hour the space may be omitted, thus:

watthour

Quotient, use the word *per* and not a solidus:

metre per second, *not* metre/second

Powers, use the modifier *squared* or *cubed* placed after the unit name:

metre per second squared

In the case of area or volume, the modifier may be placed before the unit name:

square millimetre, cubic metre

This alternative is also allowed for derived units that include area or volume:

watt per square metre

NOTE—To avoid ambiguity in complicated expressions, symbols are preferred over words.

3.5.3.2 *With unit symbols:*

Product, use a raised dot:

N·m for newton metre

In the case of W·h, the dot may be omitted, thus:

Wh

An exception to this practice is made for computer printouts, automatic typewriter work, etc., where the raised dot is not possible, and a dot on the line may be used.

Quotient, use one of the following forms:

m/s *or* m·s⁻¹ *or* $\frac{m}{s}$

In no case should more than one solidus be used in the same

expression unless parentheses are inserted to avoid ambiguity. For example, write:

$$J/(\text{mol} \cdot \text{K}) \text{ or } J \cdot \text{mol}^{-1} \cdot \text{K}^{-1} \text{ or } (J/\text{mol})/\text{K},$$

but not

$$J/\text{mol/K}$$

3.5.3.3 Symbols and unit names should not be mixed in the same expression. Write:

$$\text{joules per kilogram or } J/\text{kg or } J \cdot \text{kg}^{-1}$$

but not

$$\text{joules/kilogram nor joules/kg nor joules} \cdot \text{kg}^{-1}$$

3.5.4 Numbers:

3.5.4.1 The recommended decimal marker is a dot on the line. When writing numbers less than one, a zero should be written before the decimal marker.

3.5.4.2 Outside the United States, the comma is often used as a decimal marker. In some applications, therefore, the common practice in the United States of using the comma to separate digits into groups of three (as in 23,478) may cause ambiguity. To avoid this potential source of confusion, recommended international practice calls for separating the digits into groups of three, counting from the decimal point toward the left and the right, and using a small space to separate the groups. In numbers of four digits on either side of the decimal point the space is usually not necessary, except for uniformity in tables.

Examples:

2.141 596 73 722 7372 0.1335

Where this practice is followed, the space should be narrow (approximately the width of the letter "i"), and the width of the space should be constant even if, as is often the case in printing, variable-width spacing is used between words. Exceptions: In certain specialized applications, such as engineering drawings and financial statements, the practice of using a space for a separator is not customary.

3.5.4.3 Because *billion* means a thousand million (prefix *giga*) in the United States but a million million (prefix *tera*) in most other countries, this term and others, such as trillion, should be avoided in technical writing.

3.5.4.4 Use of M to indicate thousands, as in MCF for thousands of cubic feet, or in MCM for thousands of circular mils, of MM to indicate millions, of C to indicate hundreds, etc., is deprecated because of obvious conflicts with the SI prefixes.

3.5.5 Attachment—Attachment of letters to a unit symbol as a means of giving information about the nature of the quantity under consideration is incorrect. Thus MWe for "megawatts electrical (power)," Vac for "volts ac," and kJt for "kilojoules thermal (energy)" are not acceptable. For this reason, no attempt should be made to construct SI equivalents of the abbreviations "psia" and "psig," so often used to distinguish between absolute and gage pressure. If the context leaves any doubt as to which is meant, the word *pressure* must be qualified appropriately. For example:

"... at a gage pressure of 13 kPa"

or

"... at an absolute pressure of 13 kPa"

Where space is limited, such as on gages, nameplates, graph labels, and in table headings, it is permissible to use the unit symbol followed by a space and the modifier in parentheses. For example: V (ac) and V (dc); kPa (gage) and kPa (absolute).

3.5.6 Pronunciation—Some recommended pronunciations in English are shown in Table 8.

4. Rules for Conversion and Rounding

4.1 General:

4.1.1 Conversion factors to change a value of a quantity expressed in non-SI units to the corresponding value of that quantity expressed in the International System of Units may be exact or approximations adequate for the particular task. The rules in this section are based on using either exact or approximate factors such as those of seven-digit factors listed in Appendix X3. In some cases the quantity is such that factors with fewer digits are appropriate.

4.1.2 Conversion of quantities should be handled with careful regard to the implied correspondence between the accuracy of the data and the given number of digits. In all conversions, the number of significant digits retained should be such that accuracy is neither sacrificed nor exaggerated. (For guidance concerning significant digits see 4.3.) For example, a length of 125 ft converts exactly to 38.1 m. If, however, the 125-ft length had been obtained by rounding to the nearest 5 ft, the conversion should be given as 38 m; and if it had been obtained by rounding to the nearest 25 ft, the conversion should be given as 40 m.

4.1.3 Proper conversion procedure is to multiply a value by a conversion factor that is more accurate than is required; the result is then rounded to the appropriate number of significant digits. For example, to convert 3 feet 2 7/16 inches to metres: $(3 \times 0.3048) + (2.5625 \times 0.0254) = 0.979\,487\,5$ m, which rounds to 0.979 m. Do not round either the conversion factor or the quantity before performing the multiplication, as accuracy may be reduced. After the conversion, the

TABLE 8 Recommended Pronunciation

Prefix	Pronunciation (USA) ^A
exa	ex' a (a as in about)
peta	pet' a (e as in pet, a as in about)
tera	as in terra firma
giga	jig' a (i as in jig, a as in about)
mega	as in megaphone
kilo	kill' oh
hecto	heck' toe
déka	deck' a (a as in about)
deci	as in decimal
centi	as in centipede
milli	as in military
micro	as in microphone
nano	nan' oh (an as in ant)
pico	peek' oh
femto	fem' toe (fem as in feminine)
atto	as in anatomy
Selected Units	Pronunciation
candela	can dell' a
joule	rhyme with tool
kilometre	kill' oh metre
pascal	rhyme with rascal
siemens	same as seamen's

^A The first syllable of every prefix is accented to assure that the prefix will retain its identity. Therefore, the preferred pronunciation of kilometre places the accent on the first syllable, not the second.

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possible to use the unit
differ in parentheses.
Pa (gage) and kPa

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SI value may be expressed by a multiple or submultiple unit of SI by the use of an appropriate prefix, for example, 979 mm.

4.2 Accuracy and Rounding—A conversion obtained by multiplying a value by a seven-digit factor usually gives a product with more digits than the original value. The converted value must be rounded to the proper number of significant digits commensurate with the intended accuracy. Conversions usually yield a product with more digits than the original value. The practical aspect of measuring must be considered when using SI equivalents. If a scale having division of 1/16 inch was suitable for making the original measurements, a metric scale having divisions of 1 mm is obviously suitable for measuring in SI units. Similarly, a gage or caliper graduated in divisions of 0.02 mm is comparable to one graduated in divisions of 0.001 in. Analogous situations exist in mass, force, and other measurements. Many techniques are used to guide the determination of the proper number of significant digits in the converted values. Two different approaches to rounding of quantities are here described—one for general use and the other for conversion of dimensions involving mechanical interchangeability.

4.2.1 General Conversion—This approach depends on first establishing the intended precision or accuracy of the quantity as a necessary guide to the number of digits to retain. This precision should relate to the number of digits in the original, but in many cases this is not a reliable indicator. A figure 1.1875 may be a very accurate decimalization of a noncritical 1 1/16 that should have been expressed 1.19. On the other hand, the value 2 may mean "about 2," or it may mean a very accurate value of 2 which should have been written 2.0000. It is therefore necessary to determine the intended precision of a quantity before converting. This estimate of intended precision should never be smaller than the accuracy of measurement and should usually be smaller than one tenth the tolerance if one exists. After estimating the precision of the dimension, the converted dimension should be rounded to a minimum number of significant digits (see 4.3) such that a unit of the last place is equal to or smaller than the converted precision. Examples:

1. A stirring rod 6 in. long. In this case, precision is estimated to be about 1/2 in (± 1/4 in). Converted, this is 12.7 mm. The converted dimension 152.4 mm should be rounded to the nearest 10 mm, or 150 mm.

2. 50 000 lbf/in² (psi) tensile strength. In this case, precision is estimated to be about ± 200 lbf/in² (± 1.4 MPa) based on an accuracy of ± 0.25 % for the tensile tester and other factors. Therefore, the converted dimension, 344.7379 MPa, should be rounded to the nearest whole unit, 345 MPa.

3. Test pressure 200 ± 15 lbf/in² (psi). Since one tenth of the tolerance is 3 lbf/in² (20.68 kPa), the converted dimension should be rounded to the nearest 10 kPa. Thus, 1378.9514 ± 103.421 35 kPa becomes 1380 ± 100 kPa.

4.2.2 Special Cases:

4.2.2.1 Converted values should be rounded to the minimum number of significant digits that will maintain the required accuracy, as discussed in 4.1.2. In certain cases deviation from this practice to make use of convenient or whole numbers may be feasible, in which case the word "approximate" must be used following the conversion. For example:

1 1/8 in = 47.625 mm, exact
47.6 mm, normal rounding
47.5 mm (approx) rounded to preferred num
48 mm (approx) rounded to whole number

4.2.2.2 A quantity stated as a limit, such as "less than" or "maximum," must be handled so that the limit is not violated. For example, a specimen "at least 101.6 mm wide" requires a width of at least 101.6 mm, or at least 102 mm.

4.3 Significant Digits:

4.3.1 When converting integral values of units, the precision must be given to the implied or required precision of the integral value to be converted. For example, the value "1 in" may be intended to represent 4, 4.0, 4.00, 4.0000 in, or even greater accuracy. Obviously, the converted value must be carried to a sufficient number of digits to maintain the accuracy implied or required in the original quantity.

4.3.2 Any digit that is necessary to define the value or quantity is said to be significant. When measuring the nearest 1 m, a distance may be recorded as 1500 m. This number has three significant digits. If the measurement had been made to the nearest 0.1 m, the distance may have been 157.4 m; this number has four significant digits.

4.3.3 Zeros may be used either to indicate a specific value like any other digit, or to indicate the order of magnitude of a number. The 1970 United States population was rounded to thousands was 203 185 000. The six left-hand digits of this number are significant; each measures a unit of the order of magnitude of the number rounded to the nearest thousand. The three right-hand digits are zeros which merely indicate the order of magnitude of the number rounded to the nearest thousand. The identification of significant digits is possible through knowledge of the circumstances. For example, the number 1000 may be rounded from 999.7, in which case only one zero is significant, or it may be rounded from 999.7, in which case all three zeros are significant.

4.3.4 Occasionally data required for an investigation are drawn from a variety of sources where they have been recorded with varying degrees of refinement. Special care must be observed when such data are to be added, subtracted, multiplied, or divided.

4.3.4.1 The rule for addition and subtraction is that the answer shall contain no significant digits farther to the right than occurs in the least precise number. Consider the addition of three numbers drawn from three sources, each of which reported data in millions, the second in thousands, and the third in units:

163 000 000
217 885 000
96 432 768
477 317 768

The total indicates a precision that is not valid. The numbers should first be rounded to one significant digit farther to the right than that of the least precise number, then the sum taken as follows:

163 000 000
217 900 000
96 400 000
477 300 000

The total is then rounded to 477 000 000 as called for by the rule. Note that if the second of the figures to be added had been 217 985 000, the rounding before addition would have produced 218 000 000, in which case the 0 following 218 would have been a significant digit.

4.3.4.2 The rule for multiplication and division is that the *product* or *quotient* shall contain no more significant digits than are contained in the number with the *fewest significant digits* used in the multiplication or division. The difference between this rule and the rule for addition and subtraction should be noted; the latter rule merely requires rounding of digits that lie to the right of the last significant digit in the least precise number. The following illustration highlights this difference:

Multiplication:

$113.2 \times 1.43 = 161.876$, rounded to 162

Division:

$113.2 \div 1.43 = 79.16$, rounded to 79.2

Addition:

$113.2 + 1.43 = 114.63$, rounded to 114.6

Subtraction:

$113.2 - 1.43 = 111.77$, rounded to 111.8

The above product and quotient are limited to three significant digits since 1.43 contains only three significant digits. In contrast, the rounded answers in the addition and subtraction examples contain four significant digits.

4.3.4.3 Numbers used in the above illustrations have all been estimates or measurements. Numbers that are exact counts are treated as though they consist of an infinite number of significant digits. More simply stated, when a count is used in computation with a measurement the number of significant digits in the answer is the same as the number of significant digits in the measurement. If a count of 40 is multiplied by a measurement of 10.2, the product is 408. However, if 40 were an estimate accurate only to the nearest 10, and hence contained but one significant digit, the product would be 400.

4.4 *Rounding Values*⁷:

4.4.1 When a figure is to be rounded to fewer digits than the total number available, the procedure should be as follows:

4.4.1.1 When the first digit discarded is less than 5, the last digit retained should not be changed. For example, 3.463 25, if rounded to four digits, would be 3.463; if rounded to three digits, 3.46.

4.4.1.2 When the first digit discarded is greater than 5, or if it is a 5 followed by at least one digit other than 0, the last digit retained should be increased by one unit. For example 8.376 52, if rounded to four digits, would be 8.377; if rounded to three digits 8.38.

4.4.1.3 When the first digit discarded is exactly 5, followed only by zeros, the last digit retained should be rounded upward if it is an odd number, but no adjustment made if it

is an even number. For example, 4.365, when rounded to three digits, becomes 4.36. The number 4.355 would also round to the same value, 4.36, if rounded to three digits.

4.5 *Conversion of Linear Dimensions of Interchangeable Parts*—The use of the exact relation 1 in = 25.4 mm generally produces converted values containing more decimal places than are required for the desired accuracy. It is therefore necessary to round these values suitably and at the same time maintain the degree of accuracy in the converted values compatible with that of the original values.

4.5.1 *General*—The number of decimal places given in Table 9 for rounding converted toleranced dimensions relates the degree of accuracy to the size of the tolerances specified. Two methods of using Table 9 are given: Method A, which rounds to values nearest to each limit, and Method B, which rounds to values always inside the limits.

In Method A, rounding is effected to the nearest rounded value of the limit, so that, on the average, the converted tolerances remain statistically identical with the original tolerances. The limits converted by this method, where acceptable for interchangeability, serve as a basis for inspection.

In Method B, rounding is done systematically toward the interior of the tolerance zone so that the converted tolerances are never larger than the original tolerances. This method must be employed when the original limits have to be respected absolutely, in particular, when components made to converted limits are to be inspected by means of original gages.

Method A—The use of this method ensures that even in the most unfavorable cases neither of the two original limits will be changed by more than 5 % of the value of the tolerance. Proceed as follows:

(a) Calculate the maximum and minimum limits in inches.

(b) Convert the corresponding two values exactly into millimetres by means of the conversion factor 1 in = 25.4 mm.

(c) Round the results obtained to the nearest rounded value as indicated in Table 9, depending on the original tolerance in inches, that is, on the difference between the two limits in inches.

Method B—This method must be employed when the original limits may not be violated, for instance, certain critical mating parts. In extreme cases, this method may increase the lower limit a maximum of 10 % of the tolerance and decrease the upper limit a maximum of 10 % of the tolerance.

(a) Proceed as in Method A step (a).

(b) Proceed as in Method A step (b).

(c) Round each limit toward the interior of the tolerance, that is, to the next lower value for the upper limit and to the next higher value for the lower limit.⁸

Examples:

⁷ Adapted from ISO R370 (7).

⁸ If the digits to be rounded are zeros, the retained digits remain unchanged.

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A dimension is expressed in inches as 1.950 $\pm$ 0.016
The limits are 1.934 and 1.966
Conversion of the two limits into millimetres
gives 49.1236 and 49.9364
Method A—The tolerance equals 0.032 in and
thus lies between 0.004 and 0.04 in (see Table
9). Rounding these values to the nearest 0.01
mm, the values in millimetres to be employed
for these two limits are 49.12 and 49.94
Method B—Rounding toward the interior of the
tolerance, millimetre values for these two
limits are 49.13 and 49.93
This reduces the tolerance to 0.80 instead of 0.82 mm given by Method A.

4.5.2 Special Method for Dimensions with Plus and Minus Deviations—In order to avoid accumulation of rounding errors, the two limits of size normally are converted separately; thus, they must first be calculated if the dimension consists of a basic size and two deviations. However (except when Method B is specified) as an alternative, the basic size may be converted to the nearest rounded value and each of the deviations converted toward the interior of the tolerance. This method, which sometimes makes conversion easier, gives the same maximum guarantee of accuracy as Method A, but usually results in smaller converted tolerances.

4.5.3 Special Methods for Limitation Imposed by Accuracy of Measurements—If the increment of rounding for the tolerances given in Table 9 is too small for the available accuracy of measurement, limits that are acceptable for interchangeability must be determined separately for the dimensions. For example, where accuracy of measurement is limited to 0.001 mm, study shows that values converted from 1.0000 $\pm$ 0.0005 in can be rounded to 25.413 and 25.387 mm instead of 25.4127 and 25.3873 mm with little disadvantage, since neither of the two original limits is exceeded by more than 1.2 % of the tolerance.

4.5.4 Positional Tolerance—If the dimensioning consists solely of a positional tolerance around a point defined by a nontoleranced basic dimension, the basic dimension must be converted to the nearest rounded value and the positional variation (radius) separately converted by rounding downward.

4.5.5 Toleranced Dimension Applied to a Nontoleranced Position Dimension—If the toleranced dimension is located in a plane, the position of which is given by nontoleranced basic or gage dimension, such as when dimensioning certain conical surfaces, proceed as follows:

(a) Round the converted reference gage arbitrarily, to the nearest convenient value.

(b) Calculate exactly, in the converted unit of measurement, new maximum and minimum limits of the specified tolerance zone, in the new plane defined by the new basic dimension.

(c) Round these limits in conformity with the rules in 4.4. For example, a cone of taper 0.05 in/in has a diameter of 1.000 $\pm$ 0.002 inch in a reference plane located by the nontoleranced dimension 0.9300 in. By virtue of the taper of the cone, the limits of the tolerance zone depend on the position of the reference plane. Consequently, if the dimension 0.9300 in = 23.6220 mm is rounded to 23.600 mm (that is, a reduction of 0.022 mm), each of the two original limits, when converted exactly into millimetres, must be corrected by 0.022 $\times$ 0.05 = 0.0011 mm, in the appropriate sense, before being rounded.

4.5.6 Consideration of Maximum and Minimum Material

Condition—The ability to assemble mating parts depends on a "go" condition at the maximum material limits of the parts. The minimum material limits, which are determined by the respective tolerances, are often not as critical from a functional standpoint. Accordingly, it may be desirable to employ a combination of Methods A and B in certain conversions by using Method B for the maximum material limits and Method A for the minimum material limits. Alternatively, it may be desirable to round automatically the converted minimum material limits outside the original limits to provide greater tolerances for manufacturing.

4.5.7 While the technique described in 4.5 provides good accuracy of conversion, it will often result in dimensions that are impractical for actual production use. For conversions intended for production, it is usually necessary to round to fewer decimal places and apply design judgment to each dimension to assure interchangeability.

4.6 Other Units:

4.6.1 Temperature—General guidance for converting tolerances from degrees Fahrenheit to kelvins or degrees Celsius is given below:

Conversion of Temperature
Tolerance Requirements

Tolerance, °F	Tolerance, K or °C
2 ($\pm$ 1)	1 ($\pm$ 0.5)
4 ($\pm$ 2)	2 ($\pm$ 1)
10 ($\pm$ 5)	6 ($\pm$ 3)
20 ($\pm$ 10)	11 ($\pm$ 5.5)
30 ($\pm$ 15)	17 ($\pm$ 8.5)
40 ($\pm$ 20)	22 ($\pm$ 11)
50 ($\pm$ 25)	28 ($\pm$ 14)

Normally, temperatures expressed in a whole number of degrees Fahrenheit should be converted to the nearest 0.5 kelvin (or degree Celsius). As with other quantities, the number of significant digits to retain will depend upon implied accuracy of the original dimension, for example:

100 $\pm$ 5°F; implied accuracy estimated to be 2°F.

37.7777 $\pm$ 2.7777°C rounds to 38 $\pm$ 3°C.

1000 $\pm$ 50°F; implied accuracy estimated to be 20°F.

537.7777 $\pm$ 27.7777°C rounds to 540 $\pm$ 30°C.

4.6.2 Pressure or Stress—As with other quantities, pressure or stress values may be converted by the principle given above. Values with an uncertainty of more than 2 % may be converted without rounding by approximate factors:

$$1 \text{ lbf/in}^2 (1 \text{ psi}) = 7 \text{ kN/m}^2 = 7 \text{ kPa}$$

5. Terminology

5.1 To help ensure consistently reliable conversion and rounding practices, a clear understanding of the related nontechnical terms is a prerequisite.

TABLE 9 Rounding Tolerances Inches to Millimetres

Original Tolerance, inches		Fineness of Rounding, mm
at least	less than	
0.000 04	0.000 4	0.0001
0.000 4	0.004	0.001
0.004	0.04	0.01
0.04	0.4	0.1
0.4		1

5.2 Certain terms used in this standard are defined as follows:

accuracy (as distinguished from precision)—the degree of conformity of a measured or calculated value to some recognized standard or specified value. This concept involves the systematic error of an operation, which is seldom negligible.

approximate value—a value that is nearly but not exactly correct or accurate.

coherent system of units—a system of units of measurement in which a small number of base units, defined as dimensionally independent, are used to derive all other units in the system by rules of multiplication and division with no numerical factors other than unity (see Appendix X1.9).

deviation—variation from a specified dimension or design requirement, usually defining upper and lower limits (see also tolerance).

digit—one of the ten arabic numerals (0 to 9).

dimension—a geometric element in a design, such as length or angle, or the magnitude of such a quantity.

feature—an individual characteristic of a part, such as screw-thread, taper, or slot.

figure (numerical)—an arithmetic value expressed by one or more digits.

inch-pound units—units based upon the yard and the pound commonly used in the United States of America and defined by the National Institute of Standards and Technology. Note that units having the same names in other countries may differ in magnitude.

nominal value—a value assigned for the purpose of convenient designation; existing in name only.

precision (as distinguished from accuracy)—the degree of mutual agreement between individual measurements, namely repeatability and reproducibility.

significant digit—any digit that is necessary to define a value or quantity (see 4.3).

tolerance—the total amount by which a quantity is allowed to vary; thus the tolerance is the algebraic difference between the maximum and minimum limits.

E 380 SELECTED CONVERSION FACTORS

such as	To convert from	to	multiply by
d by one	atmosphere (760 mm Hg)	pascal (Pa)	1.013 25 × 10 ⁵
and the	board foot	cubic metre (m ³)	2.359 737 × 10 ⁻³
area and	Btu (International Table)	joule (J)	1.055 056 × 10 ³
and Tech-	Btu (International Table)/h	watt (W)	2.930 711 × 10 ⁻¹
in other	Btu (International Table)·in./s·ft ² ·°F (<i>k</i> , thermal conductivity)	watt per metre kelvin [W/(m·K)]	5.192 204 × 10 ²
of con-	calorie (International Table)	joule (J)	4.186 800*
degree of	centipoise	pascal second (Pa·s)	1.000 000* × 10 ⁻³
rements,	centistokes	square metre per second (m ² /s)	1.000 000* 10 ⁻⁶
define a	circular mil	square metre (m ²)	5.067 075 × 10 ⁻¹⁰
ntity is	degree Fahrenheit	degree Celsius	°C = (°F - 32)/1.8
fference	foot	metre (m)	3.048 000* × 10 ⁻¹
	ft ²	square metre (m ²)	9.290 304* 10 ⁻²
	ft ³	cubic metre (m ³)	2.831 685 × 10 ⁻²
	ft·lbf	joule (J)	1.355 818
	ft·lbf/min	watt (W)	2.259 697 × 10 ⁻²
	ft/s ²	metre per second squared (m/s ²)	3.048 000* × 10 ⁻¹
	gallon (U.S. liquid)	cubic metre (m ³)	3.785 412 × 10 ⁻³
	horsepower (electric)	watt (W)	7.460 000* × 10 ⁺²
	inch	metre (m)	2.540 000* × 10 ⁻²
	in. ²	square metre (m ²)	6.451 600* × 10 ⁻⁴
	in. ³	cubic metre (m ³)	1.638 706 × 10 ⁻⁵
	inch of mercury (60°F)	pascal (Pa)	3.376 85 × 10 ³
	inch of water (60°F)	pascal (Pa)	2.488 4 × 10 ²
	kgf/cm ²	pascal (Pa)	9.806 650* × 10 ⁴
	kip (1000 lbf)	newton (N)	4.448 222 × 10 ³
	kip/in. ² (ksi)	pascal (Pa)	6.894 757 × 10 ⁶
	ounce (U.S. fluid)	cubic metre (m ³)	2.957 353 × 10 ⁻⁵
	ounce-force	newton (N)	2.780 139 × 10 ⁻¹
	ounce (avoirdupois)	kilogram (kg)	2.834 952 × 10 ⁻²
	oz (avoirdupois)/ft ²	kilogram per square metre (kg/m ²)	3.051 517 × 10 ⁻¹
	oz (avoirdupois)/yd ²	kilogram per square metre (kg/m ²)	3.390 575 × 10 ⁻²
	oz (avoirdupois)/gal (U.S. liquid)	kilogram per cubic metre (kg/m ³)	7.489 152
	pint (U.S. liquid)	cubic metre (m ³)	4.731 765 × 10 ⁻⁴
	pound-force (lbf)	newton (N)	4.448 222
	pound (lb avoirdupois)	kilogram (kg)	4.535 924 × 10 ⁻¹
	lbf/in ² (psi)	pascal (Pa)	6.894 757 × 10 ³
	lb/in. ³	kilogram per cubic metre (kg/m ³)	2.767 990 × 10 ⁴
	lb/ft ³	kilogram per cubic metre (kg/m ³)	1.601 846 × 10
	quart (U.S. liquid)	cubic metre (m ³)	9.463 529 × 10 ⁻⁴
	ton (short, 2000 lb)	kilogram (kg)	9.071 847 × 10 ²
	torr (mm Hg, 0°C)	pascal (Pa)	1.333 22 × 10 ²
	W·h	joule (J)	3.600 000* × 10 ³
	yard	metre (m)	9.144 000* × 10 ⁻¹
	yd ²	square metre (m ²)	8.361 274 × 10 ⁻¹
	yd ³	cubic metre (m ³)	7.645 549 × 10 ⁻¹

* Exact

Condensed Index of Committee D-1 Standards—1992¹

This index references all standards under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials plus several others of auxiliary usefulness. It attempts to include what is relevant and exclude what is incidental to the concept of each standard. Being designed for Committee D-1 usage it omits much of the detailed information provided in the regular more comprehensive index, the latter being intended for users with a broader range of product concern. Its structure employs cross-indexing from specific to general and vice-versa, with the objective of avoiding redundancy while guiding the user with dispatch to the desired and related material. The criterion for the selection of index terms is whether they might reasonably occur to an index user familiar with the technology of organic coatings. Index users encountering errors or other deficiencies should report them promptly by writing to ASTM Subcommittee D01.18 in care of ASTM Headquarters. Changes will be included in the next edition along with entries for new standards. Subcommittee chairmen should review the standards under their jurisdiction for that purpose.

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**Section 6****PAINTS, RELATED COATINGS, AND AROMATICS**

This index covers the standards and related material appearing in Volumes 06.01, 06.02, and 06.03. The boldface references are to the ASTM designations; the standards appear in each volume in alphanumeric order. Proposed methods and specifications carry the index reference, "(Proposed)"; they appear, as does other ancillary material, in the Related Material (gray-edged) section in the back of the book. A triangle (Δ) preceding the ASTM designation denotes that Adjunct Material for the standard is published separately; the Adjunct No. is given in the standard. A Combined Index, covering the standards appearing in all volumes of the 1992 *Annual Book of ASTM Standards*, is issued as Volume 00.01.

Alphabetization in the index is letter-for-letter, with no consideration to punctuation or word division. Initial prepositions of (indented) subentries are ignored for alphabetization.

In the preparation of indexes, every attempt has been made to index standards on three levels: (1) by main subject, using general and specific search terms; (2) by test methods or other significant sections of ASTM standards; and (3) by cross-references to locate main subject entry terms. (*See also* references are abbreviated as *Sa* and appear under main entry terms.) Specification F451, for Acrylic Bone Cements (Volume 13.01), illustrates ASTM's method of indexing.

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Methyl

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ext

weight

pig

white

m

Pigment

relative

D

Pigment

lead/c

cl

st

Pigment

volum

D

Pigment

alum

D

Pigment

alum

D

anhy

hydr

Pigment

bari

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Pigment

basic

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Pigment

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