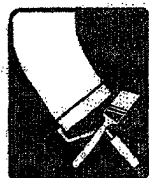


1992



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SECTION

6

Paints, Related Coatings, and Aromatics

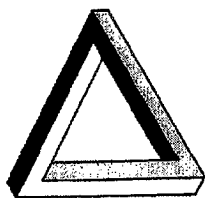


VOLUME

Paint—Pigments, Resins

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Polymers, Composites, and Fibers



06 02 and Polymers, Composites

includes standards of the following organizations:

American Society of Mechanical Engineers (ASME)

1992



ANNUAL BOOK OF ASTM STANDARDS



Paints, Related Coatings, and Aromatics



VOLUME
06.02 Paint—Pigments, Resins,
and Polymers; Cellulose

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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. 561

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— ϵ_1 for the first change, ϵ_2 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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ASTM Membership Application	

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 06.02

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. 519 and 527
A complete Subject Index begins on p. 561

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) ^{§1}	Barium Sulfate
D 1199 - 86 (1991) ^{§1}	Calcium Carbonate
D 604 - 81 (1989)	Diatomaceous Silica
§D 605 - 82 (1989)	Magnesium Silicate (Talc)
D 867 - 81 (1986) ^{§1}	Pumice
§D 607 - 82 (1987) ^{§1}	Wet Ground Mica

White Pigments

D 4288 - 83 (1989) ^{§1}	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) ^{§1}	Bone Black
D 561 - 82 (1989)	Carbon Black
D 769 - 87 (1991) ^{§1}	Iron Oxide, Synthetic Black
D 209 - 81 (1989)	Lampblack

Bronze and Miscellaneous Metallic Pigments

D 962 - 81 (1986) ^{§1}	Aluminum Powder and Paste
D 267 - 82 (1987) ^{§1}	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) ^{§1}	Copper Phthalocyanine Blue
D 261 - 75 (1987) ^{§1}	Iron Blue
D 262 - 81 (1987) ^{§1}	Ultramarine Blue

Green Pigments

§D 212 - 87	Chrome Green
D 263 - 75 (1987)	Chrome Oxide Green
D 3021 - 82 (1987) ^{§1}	Phthalocyanine Green

Yellow, Orange, Brown Pigments

D 1649 - 82 (1987) ^{§1}	Chromate, Strontium
D 478 - 86 (1991) ^{§1}	Chromate, Zinc Yellow
D 211 - 67 (1989) ^{§1}	Chrome Yellow and Chrome Orange
§D 768 - 81 (1987) ^{§1}	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
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D 1214 - 89	Glass Spheres, Sieve Analysis
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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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Standard Terminology Relating to Paint, Varnish, Lacquer, and Related Products¹

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 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.

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.

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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

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, $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$, 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} $\text{m}^2 \text{s}^{-1}$) 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} $\text{m}^2 \text{s}^{-1}$) 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} $\text{m}^2 \text{s}^{-1}$) 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

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.



Standard Test Methods for Sampling and Testing Lac Resins¹

This standard is issued under the fixed designation D 29; the number immediately following the designation indicates the year of original adoption 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 in October 1987.

1. Scope

1.1 These test methods cover procedures for sampling and testing orange shellac, button lac, garnet lac, and bleached lac.

1.2 The sampling procedures and test methods appear in the following order:

	Sections
Sampling:	
Orange Shellac, Button Lac, and Garnet Lac	3
Bleached Lac	4
Identification of Samples	5
General Requirements for Test Methods:	
Reagents	6
Source and Preparation of Portions of Sample for Tests	7
Insoluble Matter	8 to 9.5
Iodine Value	10 to 10.3
Purity	11 to 13.2
Volatile Matter (Moisture)	14 to 15.2
Wax	16 to 16.4
Matter Soluble in Water	17 to 17.2
Ash	18 to 18.2
Color	19 to 19.4.3
Color of Orange Shellac	20 to 20.4.4
Acid Value	21 to 21.4
Orpiment	22 to 22.4
Saponification Value	23 to 23.4

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. Specific hazard statements are given in Note 1.

2. Referenced Documents

2.1 ASTM Standards:

- D 304 Specification for *n*-Butyl Alcohol (Butanol)²
- D 331 Specification for 2-Ethoxyethanol²
- D 1193 Specification for Reagent Water³
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)⁴

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 D 01.33 on Varnish and Resins, Including Shellac. Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 29 - 14 T. Last previous edition D 29 - 73.

² Annual Book of ASTM Standards, Vol 06.03.

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

⁴ Annual Book of ASTM Standards, Vols 06.01, 06.02 and 06.03.

D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁴

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

SAMPLING

3. Orange Shellac, Button Lac, and Garnet Lac

3.1 **Lot Size**—For the purpose of sampling, the quantity of a lot of any one of these types of lac resin shall not exceed 500 bags or packages. The net weight of lac resin in each bag or package shall not exceed 164 lb (75 kg).

3.2 **Source and Number of Samples**—Only original unopened bags or packages shall be sampled. Ten percent of the containers in every lot of lac resin shall be taken at random, but not less than 5 nor more than 25 containers shall be taken.

3.3 **Free-Flowing Lac Resins**—In sampling free-flowing lac resins, samples shall be drawn from different places in each container in double handfuls or by means of a suitable sampler.⁶ A total of approximately 6 lb (2.7 kg) shall be taken.

3.4 **Blocked or Matted Lac Resin**—Pieces of blocked or matted lac resin shall be chipped with an axe, pick, or other suitable instrument from each container taken for sampling. Approximately the same amount shall be taken from each container and the total amount taken shall be about 6 lb (2.7 kg). The pieces of lac resin shall then be ground to pass a No. 4 (4.75-mm) sieve.

3.5 **Preparation of Samples for Observation or Analysis**—Whether free-flowing or rough ground, as in the case of blocked lac resin, the entire sample representing any lot shall be thoroughly mixed and divided into halves. The use of a mechanical mixer⁷ is recommended for mixing the resin and a riffle sampler⁸ for dividing it into quarters. When these devices are not available for use, the entire sample shall be

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ A grain sampler (Catalog No. 14-208) by the Fisher Scientific Co., 635 Greenwich St., New York, NY 10014, has been found satisfactory for this purpose.

⁷ The MacLellan mixer available from the Anglo-American Mill Corp., 75-1000 Kennaday St., Owensboro, KY, has been found satisfactory for this purpose.

⁸ The Jones sampler (Catalog No. 4-941) available from the Fisher Scientific Co., 635 Greenwich St., New York, NY 10014, has been found satisfactory for this purpose.

mixed, heaped, and quartered along two diameters that intersect at right angles and the opposite quarters combined. One half the sample, thus obtained, shall then be mixed and divided into quarters as before. Each quarter shall be placed in an airtight container, sealed, labeled (Section 5), and sent to the interested parties as the "original observation sample." When agreed upon between the seller and the purchaser, the "original observation sample" shall be used for the determination of volatile matter (moisture) (Sections 14 to 15, as applicable). The other half of the sample shall be ground to pass a No. 10 (2.00-mm) sieve,⁵ mixed thoroughly, and divided into two equal portions A and B. Portion A shall be labeled the "reserve sample." Portion B shall then be ground to pass a No. 25 (710- μ m) sieve,⁵ mixed thoroughly, and quartered as described above. Each quarter shall be packaged in an airtight container, sealed, labeled "prepared sample," and sent to the testing laboratory for analysis.

4. Bleached Lac

4.1 *Lot Size*—For the purpose of sampling, the quantity of a lot shall not exceed 200 packages.

4.2 *Source and Number of Samples*—Only original unopened packages shall be sampled. Twenty percent of the containers in every lot shall be taken at random, but not less than two containers in any lot shall be taken, except in the case where the entire lot is packaged in a single container.

4.3 *Dry Bleached Lac (Free-Flowing)*—Samples shall be drawn with a scoop or suitable sampler⁶ from different parts of each container directly after the packages are opened or bored. Approximately 1 lb (450 g) shall be drawn from each container. The samples shall be combined, mixed thoroughly, and where larger than 3 lb (1.4 kg), shall be reduced by quartering as prescribed in 3.5 to a sample of this size.

4.4 *Dry Bleached Lac (Blocked or Matted)*—Samples aggregating at least 1 lb (450 g) shall be chopped or chiseled from different parts of each container. The composite sample from all the containers shall be quickly crushed to lumps about 1 in. (25 mm) square or smaller. The crushed lac resin shall be well mixed and where the amount is larger than 3 lb (1.4 kg), it shall be reduced by quartering, as prescribed in 3.5, to a sample of approximately this size.

4.5 *Hanks, Bars, or Crushed Free-Ground Bleached Lac*—This material, which generally contains approximately 25 % moisture, shall be sampled by the procedures described in 4.3 or 4.4, as applicable.

4.6 *Preparation of Samples for Analysis*—The composite sample obtained as described in 4.3 or 4.4 shall be mixed thoroughly and divided into two equal portions A and B as prescribed in 3.5. Each portion shall be placed in a clean, dry glass jar provided with a rubber-sealed cap or an airtight friction-top tin can. Portion A shall be labeled "reserve sample." Portion B obtained in accordance with 4.3 or 4.4 shall be further ground to pass a No. 20 (850- μ m) sieve,⁵ thoroughly mixed and replaced in the jar, sealed and labeled "prepared sample." Portion B obtained in accordance with 4.5 shall be further ground to pass a No. 10 (2.00-mm) sieve,⁵ thoroughly mixed, replaced in the jar, sealed, labeled "prepared sample," and sent to the laboratory for analysis.

5. Identification of Samples

5.1 The following information shall be legibly placed on

the label, which shall be securely attached to each sample container: date of the sampling, number of bags, barrels or packages sampled, total number of containers in the lot, condition of the containers and their contents, manufacturer's name, lot and code numbers of the containers, and the purpose identification, namely "original observation sample" or "sample for determination of volatile matter (moisture)", "reserve sample" or "prepared sample" as may apply.

TEST METHODS

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 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.

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. Source and Preparation of Specimens for Tests

7.1 Each portion of sample for use in a given test shall be taken from the sample of lac resin only after it has been mixed, either by rolling on paper or by rolling and tumbling in its airtight container, as the condition of the sample requires, a sufficient number of times to ensure uniformity of the specimen taken. The test specimens shall be taken from the "prepared sample" (3.5 or 4.6), as received, except in the following cases:

7.1.1 When it has been previously agreed upon between the seller and the purchaser that the "original observation sample" shall be used for the determination of volatile matter (moisture). In this case, the "original observation sample" shall be mixed, quartered, ground, and sieved in accordance with the procedure described in 3.5 for obtaining the "prepared sample." All operations shall be done as expeditiously as possible and the test specimen taken immediately after the sieved sample has been thoroughly mixed, to avoid any possible loss by evaporation.

7.1.2 When the "prepared sample" is known to have a high moisture content, as in the case of certain forms of bleached lac (4.5), it shall be dried to a moisture content of 6 % before the test specimens are taken. The lac resin shall be dried by placing it in a thin layer in a flat-bottom dish (loosely covered to prevent dust contamination) and exposing it to the atmosphere at room temperature for 24 h and then desiccating it over anhydrous calcium chloride. The partially dried lac resin shall be kept in a clean, dry, airtight container, and shall be thoroughly mixed by rolling and tumbling in the container before the specimens are taken for analysis.

⁹ "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."

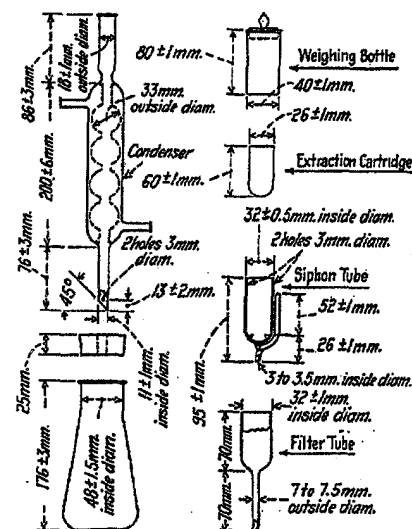


FIG. 1 Extraction Apparatus for Insoluble Matter, Test Method A

INSOLUBLE MATTER

8. Test Method A—For Orange Shellac, Button Lac, Garnet Lac, and Regular Bleached

8.1 Apparatus:

8.1.1 *Condenser*—A four-bulb Allihn condenser of the dimensions and design shown in Fig. 1.

8.1.2 *Siphon Tube*—A Knoefler siphon tube of the dimensions shown in Fig. 1.

8.1.3 *Filter Tube*—A carbon filter tube of the dimensions shown in Fig. 1.

8.1.4 *Flask*—A borosilicate glass Erlenmeyer flask 176 ± 3 mm in height and 48 ± 1.5 mm in inside diameter at the top.

8.1.5 *Flask Support*—A suitable ring support with an iron clamp and a Nichrome or iron wire gauze square without an asbestos center.

8.1.6 *Extraction Thimble*—Extraction thimble 26 ± 1 mm in diameter and 60 ± 1 mm in height.¹⁰

8.1.7 *Water Bath*—A metal container with cover of the size and design shown in Fig. 2. The container and cover shall be made of 26-oz copper sheet. The cover shall have a flanged hole 57 ± 1 mm in diameter for a 200-mL beaker and also a hole 35 ± 1 mm in diameter for the carbon filter tube. Directly below this hole in the bottom of the container shall be a flanged hole 25 ± 1 mm in diameter.

8.1.8 *Heating Device*—An electric hot plate or bunsen burner equipped with a draft shield.

8.1.9 *Weighing Bottle*—A glass-stoppered weighing bottle of the dimensions shown in Fig. 1.

8.2 *Solvent*—Specially denatured 95 % (190 proof) ethyl alcohol conforming to Formula No. 1 or No. 3A of the

¹⁰ No. 603 extraction thimbles, or equivalent, available from Schleicher & Schuell, 10 Optical Ave., Keene, NH 03431, have been found satisfactory for this purpose.

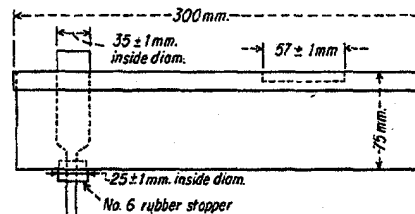


FIG. 2 Hot-Water Bath for Insoluble Matter, Test Method A

Alcohol, Tobacco and Firearms Division of Internal Revenue Service, U.S. Treasury Department.

NOTE 1: *Precautions*—The reagents and samples used in these methods may, under some conditions, be hazardous. Refer to the supplier's Material Safety Data Sheet for specific handling and safety precautions. Safe laboratory handling procedures and all applicable OSHA regulations are to be followed.

8.3 Preparation of Extraction Thimble:

8.3.1 Pass the stem of the condenser through a hole cut in the center of a cork stopper of such size that it will tightly fit the flask. Adjust the cork on the stem so that the bottom of the cork is just above the holes in the stem. Place an extraction thimble (use new thimbles only) in the siphon tube. Suspend the siphon tube from the stem of the condenser by passing a piece of copper wire through the holes in the stem and fastening the ends of the wire through the holes in the siphon tube. The wire shall be sufficiently long to leave about 6-mm space between the tip of the condenser and the top of the siphon tube.

8.3.2 Place 125 mL of ethyl alcohol in the flask and attach the flask to the condenser by means of the cork stopper. Place the flask on an electric hot plate or a flask support. Run a steady stream of cold water through the condenser. Adjust the flame of the burner or the hot plate setting so as to give a cycle of filling and emptying of the siphon tube every 2 min, and extract the thimble for 30 min. Remove the extraction thimble from the siphon tube and allow to drain and air-dry for several minutes.

8.3.3 Place the thimble in a weighing bottle and dry in an oven for 2 h at 105 ± 2°C. Remove and stopper the weighing bottle and cool in a desiccator. Weigh the bottle and thimble lifting the stopper momentarily before weighing. Continue drying and weighing as before after each hour of drying until the loss in weight between successive weighings does not exceed 2 mg.

8.3.4 A number of thimbles may be extracted and kept in weighing bottles or a desiccator until needed.

8.4. Procedure:

8.4.1 Weigh to 1 mg 5 ± 0.2 g of the mixed sample (Section 7) and place in a 200-mL tall-form beaker. Add 125 mL of ethyl alcohol to the beaker and place it in the hot-water bath (Fig. 2), which has been previously heated to not less than 90°C. Maintain the bath at this temperature, or above, during the solution and filtration of the sample. Boil the solution for 30 min, keeping the volume of alcohol constant to ensure complete solution of the lac resin.

8.4.2 Place an extracted, weighed extraction thimble (8.3) in the carbon filter tube (Fig. 2). Wet the thimble with hot

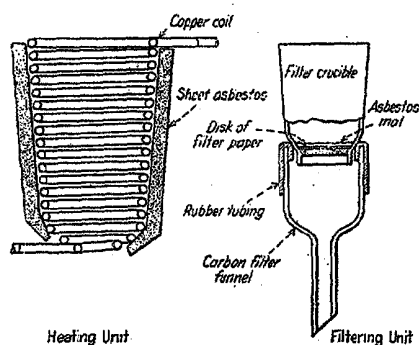


FIG. 3 Apparatus for Insoluble Matter, Test Method B

alcohol and decant the boiling solution into the warm thimble until the beaker is nearly empty. Wash the remaining solution and insoluble matter into the thimble with a stream of hot alcohol from a wash bottle using a "policeman" if necessary. Finally, wash the thimble from the top down. The transfer of the insoluble matter from the beaker and the washing down of the thimble will require at least 75 mL of hot alcohol.

8.4.3 Transfer the thimble with the insoluble matter to the siphon tube of the extraction apparatus (Fig. 1). Place 125 mL of alcohol in the flask and attach the condenser. Adjust the heating device so that a complete filling and emptying of the siphon tube with hot alcohol requires 2 min or 30 cycles per hour (Note 2), and extract for exactly 1 h (Note 3). Remove the thimble, and allow it to drain in an upright position.

NOTE 2—Occasionally, lac resins are encountered that will not yield the required 30 siphons per hour due to slow filtration. In these instances the extraction shall be continued until 30 cycles have occurred and the determination reported as abnormal or slow filtering.

NOTE 3—During the 1-h extraction, all the remaining soluble matter should be extracted by the hot alcohol, leaving only the insoluble matter in the thimble.

8.4.4 Place the thimble in the weighed weighing bottle and dry in an oven at $105 \pm 2^\circ\text{C}$ for 2 h. Remove the weighing bottle from the oven, insert its stopper, cool in a desiccator, and weigh, lifting the stopper momentarily to break the vacuum before weighing. Continue drying and weighing as before after each hour of heating until the loss in weight between successive weighings does not exceed 2 mg.

8.5 Calculation—Calculate the percent of matter insoluble in hot alcohol as follows:

$$\text{Insoluble matter, \%} = [R/S(1 - M)] \times 100$$

where:

R = insoluble matter obtained, g,

S = sample used, g, and

M = volatile matter (moisture) content of the sample, expressed as a decimal fraction.

9. Test Method B—For All Grades of Lac Resins Including Refined Bleached Lac¹¹

9.1 Apparatus (Fig. 3):

9.1.1 *Crucible*—A borosilicate glass Gooch crucible having a capacity of 30 mL with a fritted-glass filter disk having a medium porosity.

9.1.2 *Filter Tube*—A carbon filter tube made of borosilicate to fit the crucible.

9.1.3 *Heating Coil*—A heating coil made of 3-mm diameter copper tubing of such size and shape that the filter crucible and the large part of the carbon tube will fit snugly within it. The outside shall be insulated with sheet asbestos paper.

9.2 *Solvent*—Either of the following materials may be used as the solvent for the lac resin:

9.2.1 *2-Ethoxyethanol*, conforming to the requirements of Specification D 331.

9.2.2 *Normal Butyl Alcohol (n-Butanol)*, conforming to the requirements of Specification D 304.

9.3 Preparation of Filtering Unit

9.3.1 Cut a disk of rapid, ashless filter paper to fit inside the crucible and place it on top of the glass filter. Introduce upon the filter paper, in the customary manner, an asbestos mat approximately 3 mm in thickness. Dry the crucible at $105 \pm 2^\circ\text{C}$; then cool in a desiccator to constant weight. Weigh the prepared crucible and place it within the carbon tube, using thin rubber tubing to form an airtight connection. Place the combined filtering unit within the heating unit, attach to a suction flask, and pass a current of steam through the coil.

9.4 Procedure:

9.4.1 Weigh to $1 \text{ mg } 5 \pm 0.2$ of the mixed sample (Section 7 and Note 4) into a 200-mL beaker. Add 75 mL of the solvent (9.2) and bring the solution to boiling on an electric hot plate. Keep the solution boiling slowly for 5 min to ensure complete solution.

NOTE 4—For refined bleached shellac and other shellacs having a very low insoluble matter content take a 10 to 20-g specimen using proportionally more solvent.

9.4.2 Pour about 10 mL of the boiling solvent from a wash bottle into the heated crucible. Gently apply suction and immediately pour the boiling solution into the crucible so as to retain as much as possible of the insoluble residue in the beaker. Wash the insoluble residue successively with three 20-mL portions of the solvent, boiling the solution on the electric hot plate for about 1 min before each filtration.

9.4.3 Transfer the residue from the beaker to the crucible with a stream of the boiling solvent from a wash bottle, using a policeman when necessary. Wash down the inner walls of the crucible with the boiling solvent. The total volume of the solvent used will be approximately 175 mL. It is advisable to keep the crucible covered with a small watch glass at all times, except when actually transferring the solution from the beaker to crucible, or when washing down the inside walls of the crucible to maintain a higher temperature within the crucible. Allow the crucible to remain inside the heating

¹¹ Hartman, C. C., "Determination of Insoluble Matter in Shellac," *Journal of Research, Nat. Bureau Standards*, Vol 7, No. 6, 1931, p. 1105.

coil with the suction on for a few minutes, so as to suck it as dry as possible.

NOTE 5—The insoluble matter can be easily removed together with the asbestos mat and filler paper. The crucible may be used several times without further cleaning. Additional cleaning when necessary is easily accomplished by immersing the crucible in a hot mixture of sulfuric and nitric acids for a few minutes.

9.4.4 Remove the crucible and wash the outside with boiling solvent. Dry in an oven at $105 \pm 2^\circ\text{C}$ for 2 h, cool in a desiccator, and weigh.

9.5 Calculation—Calculate the percent of matter insoluble in the hot solvent as follows:

$$\text{Insoluble, \%} = [R/S(1 - M)] \times 100$$

where:

R = insoluble matter obtained, g,

S = sample used, g, and

M = volatile matter (moisture) content of the sample, expressed as a decimal fraction.

10. Iodine Value

10.1 Reagents:

10.1.1 *Potassium Iodide Solution* (100 g/L)—Dissolve 10 g of iodate-free potassium iodide (KI) in water and dilute to 100 mL.

10.1.2 *Reference Standard Shellac*—Pure shellac of known iodine value.¹²

10.1.3 *Sodium Thiosulfate, Standard Solution* (0.1 N)—Prepare and standardize in accordance with Section 5 of Method D 1959.

10.1.4 *Starch Indicator Solution*—Prepare in accordance with Test Method D 1959.

10.1.5 *Wijs Solution*—Prepare in accordance with Test Method D 1959. The Wijs solution should be tested against an orange shellac the iodine value of which is accurately known.¹¹ The iodine value thus obtained should be within ± 0.5 of the known iodine value.

10.2 Procedure:

10.2.1 Weigh to 0.1 mg about 0.2 g of the mixed sample (Section 7 and Notes 6 and 7) into a 250-mL dry, clear glass bottle having a ground-glass stopper. Add 20 mL of glacial acetic acid and heat at 65 to 70°C on a hot-water bath, gently swirling the contents of the bottle occasionally, until solution is complete, except for the wax. This should not require more than 15 min. Add 10 mL of chloroform and cool at 21.5 to 22.5°C for $\frac{1}{2}$ h in an insulated, thermostatically controlled water bath.

NOTE 6—In the case of grossly adulterated samples, a smaller quantity (0.15 or 0.1 g) should be used instead of 0.2 g of the material in order that the excess of iodine monochloride may not be too greatly reduced, since the excess of halogen is one of the factors in determining the amount of absorption. In case less than 25 mL of the $\text{Na}_2\text{S}_2\text{O}_3$ solution is required, another test should be made using a smaller quantity of the lac resin.

NOTE 7—In weighing lac resin some difficulty is at times experienced on account of its electrical resistance properties. In very dry weather it may be found that the necessary handling to prepare it for weighing has

electrostatically charged it, and that it may be necessary to leave it in the balance pan at rest for a few minutes before determining the weight.

NOTE 8—If a number of samples are being run, allow at least 5 min between the additions of the Wijs solution.

10.2.2 Add 20 mL of the Wijs solution to the bottle, using a pipet. Replace the stopper and half immerse the bottle in the water bath held from 21.5 to 22.5°C for exactly 1 h (Note 8). Gently swirl the contents of the bottle occasionally during the hour. Remove the bottle from the bath and add 10 mL of the KI solution washing into the bottle any Wijs solution on the stopper with the same.

10.2.3 Immediately titrate with the standard $\text{Na}_2\text{S}_2\text{O}_3$ solution, allowing the solution to run rapidly and swirling the contents of the bottle vigorously and continuously until the solution becomes a straw color. Add 5 mL of the starch indicator solution and continue the titration dropwise until the blue color just disappears. The end point is sharp and any blue color returning after 30 s should be disregarded.

10.2.4 *Blank*—Run a blank determination on the reagents at the same time and through the entire procedure. The blank is necessary because of the effect of temperature changes on volume and possible loss in strength of the Wijs solution.

10.2.5 *Reference Standard*—Run a determination on a sample of pure shellac of known iodine value (10.1.2) with each set of test specimens.

10.3 Calculation—Calculate the iodine value as follows:

$$\text{Iodine value} = [(B - V)N \times 12.69]/S$$

where:

V = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the specimen, mL,

B = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the blank, mL,

N = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, and

S = sample used, g.

PURITY

11. Qualitative Test for Rosin

11.1 Reagents:

11.1.1 *Acetic Acid* (Glacial).

11.1.2 *Ethyl Alcohol* (Absolute).

11.1.3 *Halphen-Hicks Reagent*¹³—Prepare the following two solutions:

11.1.3.1 *Solution A*—One part by volume of phenol dissolved in 2 parts by volume of carbon tetrachloride.

11.1.3.2 *Solution B*—One part by volume of bromine dissolved in 4 parts by volume of carbon tetrachloride.

11.2 Procedure:

11.2.1 Weigh 2 ± 0.1 g of the mixed sample (Section 7) into a 2-L Florence flask and dissolve in 20 mL of either absolute ethyl alcohol or glacial acetic acid, warming on a steam bath if necessary. Cool, add 100 mL of petroleum ether, and mix thoroughly.

11.2.2 Add sufficient water to bring the petroleum ether layer into the neck of the flask when separated. Add the water portionwise, shaking the flask between additions to

¹² Standard samples of pure orange shellac, rosin-free of accurately known iodine number may be purchased from the Secretary of the U. S. Shellac Importers Assn., Inc., 425 Park Ave., New York, NY 10022.

¹³ For more detailed information, reference may be made to Hicks, E. F., "New Color Reactions for Some of the Resins with Halphen's Reagent for Colophony," *Industrial and Engineering Chemistry*, Vol 3, 1911, p. 86.

prevent coagulation of the precipitated lac resin. Allow to stand until the petroleum ether separates into a distinct layer.

11.2.3 Siphon off at least 50 mL of the petroleum ether solution, filter if cloudy, and heat on an electric hot plate or steam bath until the petroleum ether is completely removed.

11.2.4 Dissolve the residue in several millilitres of Solution A, and transfer 1 to 2 mL of the mixture to one of the cavities of a white porcelain spot plate. Immediately fill an adjacent cavity with Solution B. Cover the plate with an inverted watch glass and note the color developed in the Solution A mixture by the bromine vapors from Solution B. The development of a fugitive violet color, best observed on the flat portion of the spot plate, indicates the presence of rosin. A control sample containing rosin should be run simultaneously, as a guide in judging the color developed with the test specimen.

12. Qualitative Test for Copal

12.1 Reagents:

12.1.1 Ethyl Alcohol, Denatured—(See 8.2).

12.1.2 Methyl Alcohol (99 %).

12.2 Procedure:

12.2.1 Weigh to 0.1 g about 15 g of the mixed sample (Section 7) into an Erlenmeyer flask. Add twice its weight of the denatured alcohol, stopper the flask and let stand with periodic shaking until the sample is in the solution. Filter the solution through a folded filter paper, discarding the first 5 mL of filtrate.

12.2.2 Transfer 10 mL of the filtrate to a large test tube (6 by 3/4 in.) (150 by 20 mm) and nearly fill the tube with methyl alcohol. Stopper the tube and mix its contents thoroughly. Immediate formation of a cloudiness or precipitate indicates the presence of copal. Lac resin free of copal should remain clear.

13. Estimation of Adulteration

13.1 Since the variation between the highest and lowest iodine values of a pure lac resin is not great, it is recommended that the following assumptions (Note 9) be made:

Type of Lac Resin	Assumed Iodine Value
Rosin-free and copal-free shellac, button lac, and garnet lac	15
Rosin-free and copal-free bleached lac ^a	10
Rosin	228
Copal	130

13.2 Calculate the percent of adulteration as follows:

Percent of rosin in orange shellac, button lac, and garnet lac

$$= [(x - 15)/(228 - 15)] \times 100$$

Percent of rosin in bleached lac = $[(x - 10)/(228 - 10)] \times 100$

Percent of copal in orange shellac, button lac, and garnet lac

$$= [(x - 15)/(130 - 15)] \times 100$$

Percent of copal in bleached lac = $[(x - 10)/(130 - 10)] \times 100$

where:

x = iodine value of the sample under test, determined in accordance with Section 10.2.

NOTE 9—The results obtained by assuming the values of 15 and 10 as the iodine value of orange and bleached shellac, respectively, and 228 as the iodine value of rosin may give a slightly lower percent rosin, under some circumstances, than that which is actually present.

VOLATILE MATTER (MOISTURE)

14. Test Method A—For Orange Shellac, Button Lac, Garnet Lac, and Dry Bleached Lac

14.1 Procedure:

14.1.1 Weigh to 0.1 mg approximately 2 g of the mixed sample from the air-tight container (7.1.1 or 7.1.2) into a weighed, clean, dry, flat-bottom glass dish about 4 in. (100 mm) in diameter and provided with a ground-glass cover. Place the dish, with the cover removed, in a well-ventilated oven maintained at $40 \pm 1^\circ\text{C}$ for 6 h.

14.1.2 Transfer the dish and cover to a vacuum desiccator containing concentrated sulfuric acid (H_2SO_4 , sp gr 1.84). Immediately evacuate the desiccator and keep the specimen uncovered in the vacuum for 18 h. Release the vacuum, replace the cover on the dish, and weigh immediately.

14.2 Calculation—Calculate the percent volatile matter (moisture) in the sample as follows:

$$\text{Volatile matter (moisture), \%} = [1 - (S_2/S_1)] \times 100$$

where:

S_1 = sample used, g, and

S_2 = dried specimen, g.

15. Test Method B—For Bleached Lac in Form of Hanks, Bars, and Crushed Fresh-Ground

15.1 Procedure:

15.1.1 Thoroughly mix the "original observation" sample in its original airtight container by rolling and tumbling. Transfer 25 to 50 g to a mortar and crush as rapidly as possible into fine granules. Keep the mortar covered as well as possible to avoid any loss of moisture. Immediately transfer approximately 10 g of the crushed lac to a weighed, clean, dry, flat-bottom glass dish about 4 in. (100 mm) in diameter, provided with a ground-glass cover, and weigh to 0.1 mg. Record the weight of the lac taken for use as S_1 .

15.1.2 Place the dish and contents in a vacuum desiccator containing H_2SO_4 (sp gr 1.84). Remove the cover from the dish and immediately evacuate the desiccator. Keep the dish in the vacuum at room temperature for 18 to 24 h. Replace the cover on the dish, remove from the desiccator, and weigh. Record the weight of the partially dried lac for use as S_2 .

15.1.3 Grind the partially dried lac resin until it entirely passes a No. 40 (425- μm) sieve.⁵ Thoroughly mix the sieved lac on a mixing sheet (7.1). Transfer approximately a 2-g specimen of it from the mixing sheet to a weighed, covered dish of the type described in 15.1.1, and weigh to 0.1 mg. Record the weight of the specimen taken for use as S_3 .

15.1.4 Heat the dish with cover removed in a well-ventilated oven at $40 \pm 1^\circ\text{C}$ for 18 h. Replace the cover on the dish, cool in a desiccator, and weigh. Record the weight of dried lac obtained for use as S_4 .

15.2 Calculation—Calculate the percent of volatile matter (moisture) in the original lac sample as follows:

$$\text{Volatile matter (moisture), \%} = [1 - (S_4 \times S_2)/(S_3 \times S_1)] \times 100$$

where:

S_1 = crushed wet lac taken for drying in vacuum, g, (15.1.1)

S_2 = partially dried lac, g, (15.1.2),

S_3 = partially dried, sieved lac used as specimen for final oven drying, g, (15.1.3), and

S_4 = completely dried lac specimen obtained in 15.1.4, g.

16. Wax

16.1 Apparatus:

16.1.1 *Extraction Apparatus*—An assembly suitable for continuous extraction with hot solvent, such as that used for the determination of insoluble matter (Section 8).

16.1.2 *Filtering Medium*—A Büchner funnel, having an inside diameter of 2¼ to 3½ in. (55 to 90 mm), and prepared just before use in the following manner. Fit a disk of filter paper snugly over the bottom of the funnel and then with the suction on pour over the filter paper in a layer of uniform thickness 1 g of filter aid (16.2.3) suspended in water.

16.1.3 *Extraction Thimble*—A thimble, such as prescribed in 8.1.6, that has been previously extracted with chloroform by the procedure given in 8.3.

16.2 Reagents:

16.2.1 *Chloroform*, redistilled, free of nonvolatile residue.

16.2.2 *Ethyl Alcohol, Denatured*, as prescribed in 8.2.

16.2.3 *Filter Aid*¹⁴—A suitable filter aid, extracted with chloroform and dried before using.

16.2.4 *Sodium Carbonate* (Na_2CO_3).

16.3 Procedure:

16.3.1 Weigh to 1 mg approximately 10 g of the mixed sample (Section 7) into a 200-mL tall-form beaker. Dissolve 2.5 g of Na_2CO_3 in 150 mL of hot water and add to the beaker. Immerse the beaker in a boiling water bath and stir until the lac resin is in solution. Cover the beaker with a watch glass and allow to remain in the bath for 2 to 3 h, without agitation.

16.3.2 Remove the beaker from the bath and place it in cold water. The wax will come to the top of the solution where it will solidify as a layer or float as small, hard particles. Add 0.5 g of the filter aid to the lac solution and filter through the Büchner funnel with the aid of suction. Transfer all of the wax from the beaker to the filter with a stream of water from a wash bottle, using a policeman if necessary. Finally pour a few millilitres of the alcohol over the filter to facilitate drying. Place the funnel in an oven and dry its contents at $60 \pm 2^\circ\text{C}$ for several hours.

16.3.3 Insert a thin spatula under the edge of the filter paper and transfer the contents of the funnel to a sheet of filter paper. Securely wrap the transferred material in the filter paper, and bind it firmly with fine copper wire. Place the packet in the prepared extraction thimble. Wash out both the beaker in which the lac resin was originally dissolved and the Büchner funnel with portions of hot chloroform, filtering the chloroform solutions through the extraction thimble into the flask to be used in the extraction. Place the thimble in the extraction apparatus, and extract with hot chloroform for 2 h.

16.3.4 Transfer the chloroform extract to a weighed beaker and heat on a steam-bath until most of the chloroform has been evaporated. Finally heat the residue in the beaker in an oven at $105 \pm 2^\circ\text{C}$ to constant weight (within 10 mg). Cool in a desiccator and weigh.

16.4 *Calculation*—Calculate the percent of wax in the lac resin as follows:

$$\text{Wax, \%} = [R/S(1 - M)] \times 100$$

where:

R = wax obtained, g,

S = sample used, g, and

M = volatile matter (moisture) content of the sample, expressed as a decimal fraction.

17. Matter Soluble in Water

17.1 Procedure:

17.1.1 Weigh to 0.1 g 10 g of the mixed sample (Section 7) into a 400-mL beaker. Add 200 mL of water and stir thoroughly. Cover the beaker with a watch glass and let stand with occasional stirring for 4 h at $25 \pm 2^\circ\text{C}$.

17.1.2 Filter the water solution through a filter paper into a 400-mL beaker. Transfer all the lac resin from the beaker to the filter paper with a stream of water from a wash bottle using a policeman if necessary. Finally wash the lac resin and filter paper with approximately 100 mL of water.

17.1.3 Evaporate the filtrate almost to dryness in a weighed evaporating dish on a steam bath. Dry the extracted residue for 1-h periods in an oven maintained at $105 \pm 2^\circ\text{C}$ until the difference in successive weighings does not exceed 1 mg. Cool in a desiccator and weigh.

17.2 *Calculation*—Calculate the percent of matter soluble in water as follows:

$$\text{Matter soluble in water, \%} = [R/S(1 - M)] \times 100$$

where:

R = residue obtained, g,

S = sample used, g, and

M = volatile matter (moisture) content of the sample, expressed as a decimal fraction.

18. Ash

18.1 Procedure:

18.1.1 Weigh to 1 mg between 3 and 5 g of the mixed sample (Section 7) into a weighed porcelain crucible. Place the crucible in a hood and heat with a low flame until the contents of the crucible are a dry, charred mass. Transfer to a muffle furnace and ignite the residue at a dull red heat (not exceeding 600°C) until the ash is free of carbon. Cool in a desiccator and weigh. Repeat the ignition, cooling and weighing until the difference between successive weighings does not exceed 1 mg.

18.1.2 If a carbon-free ash cannot be obtained in this manner, add water to the crucible, bring the water to a slow boil and digest the ash for 5 to 10 min. Filter through an ashless filter paper, washing out the crucible with a stream of hot water from a wash bottle using a policeman if necessary. Retain the filtrate. Transfer the filter paper with the insoluble residue to the crucible and ignite at a dull red heat in a muffle furnace until all the carbon is consumed. Then add the filtrate to the crucible and evaporate to dryness over a low flame or hot plate. Finally ignite the crucible and its contents at dull red heat to constant weight (within 1 mg) as prescribed in 18.1.1.

18.2 *Calculation*—Calculate the percent ash of the lac resin as follows:

$$\text{Ash, \%} = [R/S(1 - M)] \times 100$$

¹⁴ A diatomaceous silica such as Filter-Cel, tradename of Mariville Corp., Filtration and Minerals Div., 137 W. Central Ave., Lompoc, CA 93436, has been found satisfactory for this purpose.

where:

R = ash obtained, g,

S = sample used, g, and

M = volatile of matter (moisture) content of the sample, expressed as a decimal fraction.

19. Color

19.1 Apparatus:

19.1.1 *Glass Tubes*—Clear glass tubes with closed, flat, even bottom, an external length of approximately 114 mm, and a uniform internal diameter of 10.65 ± 0.025 mm throughout the length of the tube. Viscosity tubes described in Test Method D 1545 are satisfactory.

19.1.2 *Glass Plate*—White Carrara or Vitrolite glass plate approximately 6 by 8 in., (150 by 200 mm), one side of which has been polished to a smooth, high gloss surface.

19.1.3 *Film Applicator*—Film applicator to case a film 2 in. (50 mm) wide and 2 mils (50 μ m) thick. A clearance of 4 mils should provide the required thickness.

19.2 *Reagent*—Ethyl Alcohol, Denatured, as prescribed in 8.2.

19.3 *Color Comparison Material*—The lac resin mutually agreed upon by the purchaser and the seller for the color comparison.

19.4 Procedure:

19.4.1 Weigh 10 ± 0.1 g each of the mixed sample (Section 7) and the comparison lac resin, which shall have been similarly prepared, into separate Erlenmeyer flasks. Add to each flask an amount of alcohol equal to twice the weight of the resin and stopper the flasks. Keep the flasks at 21 to 32°C and shake at frequent intervals until the resins are completely dissolved (cut). Allow the flasks to stand undisturbed for $\frac{1}{2}$ h. Compare the color of the lac solutions by one or both of the following procedures as agreed upon by the purchaser and the seller.

19.4.2 *Comparison of Solutions*—Fill separate glass tubes to their bottom lines with each of the solutions, being careful not to disturb any settlement that may have occurred in either flask. Place the tubes side by side and make the color comparison by viewing the liquids while they are held against a background substantially equal in illumination to that of a fairly light overcast northern sky.

19.4.3 *Comparison of Films*—Without disturbing any settlement that may have occurred in the flask, decant a suitable portion of the solution of the sample and draw down on the white glass plate with a film, 2-in. (50-mm) wide and 2 mils (50 μ m) thick, using the apparatus described in 19.1.3. In a similar manner, draw down a film of the comparison solution on the same plate keeping the films as close together as possible. Air-dry the films at 21 to 32°C for 24 h. Compare the color of the dried films by viewing them by reflected light at an illumination substantially equal to that of a fairly overcast northern sky.

20. Color of Orange Shellac

20.1 Apparatus:

20.1.1 *Funnel*—Porcelain No. 1 Büchner funnel 55 mm in inside diameter.

20.1.2 *Colorimetric Tubes*—Nessler color comparison tubes, approximately 20 mm in diameter and 300 mm in length.

20.1.3 *Standard Light Source*—A light source consisting of a 100-W frosted daylight bulb mounted at the center of the back wall of a 10-in. (255-mm) cubic box so that the tip of the bulb faces the front. The front opening of the box shall be covered with a white opaque paper or a ground-glass plate. The lamp shall be inserted in a socket which just protrudes through the wall of the box so that the distance from the tip of the lamp to the paper or glass is approximately 4.5 in. (115 mm). The five inside walls of the box shall be painted white. Any other suitable apparatus or standard daylight lamp may be used, provided it produces light of the same characteristics as specified above.

20.2 Reagents:

20.2.1 *Ethyl Alcohol*—Specially denatured 95 % (190 proof) ethyl alcohol; either formula No. 1 or formula No. 30 of the U.S. Internal Revenue Bureau.

20.2.2 *Filter Aid*—Any high-grade analytical filter aid for rapid flow.

20.2.3 *Ferric Sulfate* ($\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$)—Any hydrated analytical reagent.

20.2.4 *Sodium Hydroxide, Standard Solution* (1 N)—Dissolve 40 g of pure NaOH in 500 mL of distilled water and dilute to 1 L in a volumetric flask. Standardize against pure oxalic acid dihydrate.

20.2.5 *Sodium Thiosulfate, Standard Solution* (0.2 N)—Dissolve 49.66 g sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in distilled water that has been previously boiled to free it from carbon dioxide and dilute to 1 L of the solution. It is best to let this solution stand about 2 weeks before standardizing with pure resublimed iodine or potassium bi-iodate. Preserve in a brown stock bottle with a guard tube filled with soda lime.

20.2.6 *Starch Solution*—Make a paste of 0.2 g of soluble starch (potato starch) in cold water and pour into 100 mL of boiling water, cool and bottle or use the solution prepared for the iodine number determination (10.1.5).

20.2.7 *Potassium Iodide (KI)*—Iodate-free crystal.

20.2.8 *Nickel Sulfate*—Reagent grade ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$).

20.3 Preparation of Color Standards

20.3.1 *Ferric Sulfate*—Dissolve 400 g of $\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$ in about 600 mL of water by heating to boiling while stirring constantly. After complete solution, cool to room temperature and dilute to 1 L in a volumetric flask. Standardize this stock solution by titrating with iodine in the following manner: Dissolve 20 g of KI in 30 mL of water and add 5 mL of the stock solution. Add to this approximately 2 weight % of H_2SO_4 (sp gr 1.84). Allow to stand for 5 min and titrate with 0.2 N sodium thiosulfate solution in the usual manner, not adding the starch indicator until near the end of the titration. Run at the same time a blank determination on the KI solution and correct for any nonuniformity of this reagent. Adjust the stock solution to approximately 0.725 M $\text{Fe}_2(\text{SO}_4)_3$ solution (Note 10); 5 mL of the stock solution should then be equivalent to 36.2 ± 0.8 mL of 0.2 N sodium thiosulfate solution.

NOTE 10—The amount of coordinated water in ferric sulfate may vary, and has to be taken into account in weighing out the required amount of ferric salt. It is usually about 6 mols per mol of ferric sulfate for the powdered analyzed reagent.

20.3.2 *Nickel Sulfate*—Dissolve 50 g of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ in about 300 mL of water. After complete solution, dilute to 500 mL in a volumetric flask.

20.3.3 *Reference Standard for All Grades*¹⁵—Transfer exactly 20 mL of the stock ferric sulfate solution (20.3.1) to a 100-mL volumetric flask. Add 11.5 mL of the 1.0 N NaOH solution from a buret, and shake until all the precipitate formed has dissolved. Then, add 10 mL of the nickel sulfate solution and dilute to 100 mL with distilled water. If any of the solutions show permanent precipitates, prepare fresh solution.

20.4 Procedure:

20.4.1 Transfer exactly 5 g of the sample to a clean 4-oz (120-mL) wide-mouth bottle and add exactly 50 mL of alcohol. Shake until solution is complete and then cool to 10°C.

20.4.2 Place a 55-mm tough, hard, close texture filter paper or its equivalent in the Büchner funnel which has been mounted in the neck of a 2-L suction flask with a rubber stopper. Pour evenly upon the filter paper a 1-g suspension of filter aid in 50 mL of alcohol, and suck completely dry with a partial vacuum, using a water suction pump. Remove the funnel and add about 400 mL of alcohol to the flask. (Note 11) Place an 8-in. (200-mm) test tube in the suction flask, supporting, if necessary, with filter paper, so that the tip of the Büchner funnel when inserted in the neck of the flask will come well within the open test tube. Reinsert the funnel in the flask.

20.4.3 Add 1 g of filter aid to the cold shellac solution and stir thoroughly. Transfer completely to the Büchner funnel and filter at the rate of 2 drops per second by means of a carefully regulated vacuum. This may be conveniently done by the use of a water pump to which has been attached a trap carrying a stopcock to admit air. Slowly increase the amount of vacuum toward the end of the filtration in order to maintain a constant filtration rate until the filtration is practically complete, and then suck dry. The final volume in the test tube should be 49 to 50 mL. If it is less than 48 mL, repeat the procedure.

NOTE 11—The method used in preparing the solution for comparison is very important. A slight loss in alcohol will materially affect the color when diluted for comparison. Alcohol is placed in the flask to avoid undue evaporation of the solution during filtration. The rate of filtration is an important factor.

20.4.4 Transfer 10 mL of filtered solution to one of the colorimetric tubes, and compare with 10 mL of the standard color solution by viewing the tubes transversely in front of the standard light source. Dilute the shellac solution with alcohol until it matches the reference standard color. Report the volume in millilitres of the diluted solution; this is taken as the color number of the sample. Determine the color of the filtered solution the same day upon which the samples are dissolved.

NOTE 12—Most filtered solutions made by dissolving shellac that has been ground for analysis will darken appreciably in color if allowed to stand for periods longer than 12 h.

¹⁵ This standard color solution was designed to match the No. 5 iodine color standard of the Angelo color method which has been used by various laboratories for obtaining numerical color values for lacs. It is closer in hue to most lacs than the iodine solution and hence easier to match.

21. Acid Value

21.1 Definition:

21.1.1 *acid value*—the number of milligrams of potassium hydroxide required to neutralize 1 g of moisture-free lac resin.

21.2 Reagents:

21.2.1 *Neutral Ethyl Alcohol*—Alcohol, as described in Section 8, that has been neutralized with standard KOH or NaOH solution, using the phenolphthalein indicator to a faint but persistent pink color just prior to use.

21.2.2 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (8.2), methanol, or isopropanol.

21.2.3 *Potassium Hydroxide or Sodium Hydroxide Solution, Standard Aqueous or Alcoholic (0.1 N)*:

21.2.3.1 *Preparation of Aqueous Solution*—Prepare a stock concentrated solution by dissolving potassium hydroxide (KOH) or sodium hydroxide (NaOH) in water in the proportion of 112 g of KOH, or 85 g of NaOH in 200 mL of water. Allow the solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the carbonate precipitate into another clean bottle. Add clear barium hydroxide ($\text{Ba}(\text{OH})_2$) solution until no further precipitate forms. Again allow to settle until clear. Draw off 175 mL and dilute to 10 L with water. Preserve in a stock bottle provided with a guard tube filled with soda-lime.

21.2.3.2 *Preparation of Alcoholic Solution*—Place 5 to 10 g of KOH in a 2-L flask and add 1 to 1.5 L of alcohol prescribed in 8.2. Boil on a water bath under a reflux condenser for 30 to 60 min. Distill and collect the alcohol. Dissolve 5.6 g of KOH or 4 g of NaOH in 1 L of the distilled alcohol, keeping the temperature below 15.5°C while the alkali is being dissolved. This solution should remain clear.

21.2.3.3 *Standardization of Solution*—Standardize either solution by titrating against pure potassium acid phthalate,¹⁶ using phenolphthalein indicator. The solution will be approximately 0.1 N. Determine its exact normality to ± 0.001 N.

21.2.4 *Thymol Blue Indicator Solution*—Dissolve 0.04 g of thymol blue (thymol sulfonphthalein) in 100 mL of the alcohol prescribed in 8.2.

21.3 Procedure:

21.3.1 Weigh to 1 mg approximately 2 g of the mixed sample (Section 7) into a 250-mL Erlenmeyer flask and add 100 mL of the neutral alcohol. The sample should completely dissolve at room temperature within a few hours with the aid of periodic gentle swirling. Titrate in accordance with 21.3.2 or 21.3.3.

21.3.2 In case of bleached lac, add 1 mL of the phenolphthalein indicator solution and titrate with 0.1 N KOH or NaOH solution, with constant swirling of the contents of the flask. Take as the end point when a faint pink color remains after continuous swirling for 30 s.

21.3.3 In case of orange shellac, place several drops of the thymol blue indicator solution on a white porcelain spot plate. Titrate the solution with 0.1 N KOH or NaOH

¹⁶ National Institute of Standards and Technology standard reference material No. 84d is recommended for this purpose and should be handled as directed in the certificate of analysis accompanying the sample.

solution, with constant swirling, and determine the end point by transferring 1 or 2 drops of the solution on a glass rod to the indicator. The end point is reached when the first blue color is developed in the indicator.

21.4 *Calculation*—Calculate the acid value as follows:

$$\text{Acid value} = (VN \times 56.1)/S(1 - M)$$

where:

V = KOH or NaOH solution required for the titration, mL

N = normality of the KOH or NaOH solution,

S = sample used, g, and

M = volatile matter (moisture) content of the sample expressed as a decimal fraction.

22. Orpiment

22.1 Reagents and Materials:

22.1.1 In addition to conforming to the requirements of Section 6, all reagents shall be free of arsenic.

22.1.2 *Ammonium Hydroxide* (NH_4OH)—Concentrated ammonium hydroxide (sp gr 0.90).

22.1.3 *Carbon Disulfide* (CS_2).

22.1.4 *Carbon Tetrachloride* (CCl_4).

22.1.5 *Ethyl Alcohol, Denatured*, as prescribed in 8.2.

22.1.6 *Ferrous Sulfate or Ferrous Ammonium Sulfate* (FeSO_4 or $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4$).

22.1.7 *Hydrochloric Acid* (HCl)—Concentrated hydrochloric acid (sp gr 1.19).

22.1.8 *Hydrogen Peroxide* (H_2O_2)—Concentrated hydrogen peroxide 30 %.

22.1.9 *Hydrogen Sulfide* (H_2S)—A supply or source of gaseous hydrogen sulfide.

22.1.10 *Nitric Acid* (HNO_3)—Concentrated nitric acid (sp gr 1.42).

22.1.11 *Sulfuric Acid* (H_2SO_4)—Concentrated sulfuric acid (sp gr 1.84).

22.2 Apparatus:

22.2.1 *Filter Paper*—Ashless, medium-porosity filter paper.

22.2.2 *Funnel*—A jacketed-glass funnel or funnel about which has been wound a coil of copper or tin tubing through which steam may be passed to heat it.

22.2.3 *Gooch Crucible*—A Gooch crucible that has been prepared with an asbestos mat in the usual manner, ignited, cooled, and stored in a desiccator.

22.2.4 *Distillation Assembly*—A 300-mL Kjeldahl flask, fitted with a 2-hole rubber stopper, and attached to a bulb-type glass condenser by a U-tube having about $\frac{1}{4}$ -in. (6.4-mm) bore and a distance of 8 in. (205 mm) between arms. Through the other hole in the stopper a double-bulb safety tube or a small separatory funnel with a long, slim stem is inserted. The tip of the safety tube or funnel is bent to form a short U, the orifice of which points upward. The tube or funnel is fitted into the flask so that the U tip nearly touches the bottom of the flask. A 250-mL beaker, which serves as a receiver, is held on a movable support, immediately under the condenser tip.

22.3 Procedure:

22.3.1 Weigh to 0.1 g approximately 50 g of the mixed sample (Section 7) into a 500-mL Kjeldahl flask. Add 450 mL of the alcohol to the flask and heat its contents in a hot water bath at a gentle boil until the lac resin is completely dissolved.

22.3.2 Remove the flask from the bath and wait just long enough to permit settlement of the bulk of the insoluble matter from the solution. Decant the solution from the flask to the filter paper held in the jacketed funnel which must be kept hot during the entire filtration by passing steam through the coil or jacket. Keep a watch glass over the funnel when not pouring the solution into it (Note 13).

22.3.3 Wash the sediment layer in the flask with four successive 50-mL portions of boiling alcohol, and decant the washings through the filter. Finally wash the filter paper with 50 mL of the boiling alcohol. Continue heating the funnel to drive off most of the alcohol. Pour slowly over the entire surface of the filter paper approximately 200 mL of boiling carbon tetrachloride to dissolve any of the lac wax that may have remained on the filter paper (Note 14). Now leave the watch glass off and continue passing the steam through the funnel jacket to dry the solvent from the paper.

NOTE 13—If the funnel and its contents are not kept hot the wax will congeal and clog the filter paper, thus slowing up the filtration.

NOTE 14—All wax should be removed from the filter paper and inside of the flask, as otherwise the subsequent digestion procedure may be materially prolonged.

22.3.4 Carefully transfer the filter paper and its contents to the flask. Add 25 mL of HNO_3 (sp gr 1.42) to the flask, and apply gentle heat, digesting the contents of the flask for 20 min. Cool, add 25 mL of H_2SO_4 and again heat gently until most of the nitrous fumes have been driven off. Increase the heat and continue boiling until the evolution of sulfur trioxide (SO_3) fumes occurs.

22.3.5 If organic matter or charring is evident at this point, allow the flask to cool to room temperature. Rinse down the inside of the flask with a small amount of water and carefully add 3 to 4 mL of H_2O_2 . After any initial violent gas evolution has subsided again heat to SO_3 fumes. If discoloration reappears, repeat the hydrogen peroxide treatment and heating until a clear carbon-free solution is obtained.

22.3.6 Transfer the clear solution to the 300-mL Kjeldahl flask, rinsing the flask thoroughly with small portions of water until the volume of the solution and washing is between 125 and 150 mL. Heat the solution rapidly to boiling and evaporate just short of fuming to ensure complete removal of both HNO_3 and H_2O_2 .

22.3.7 Add 20 g of either ferrous sulfate or ferrous ammonium sulfate to the flask and connect the flask to the distillation assembly. Place 50 mL of water in the beaker, serving as receiver, and raise the beaker on its support until the end of the condenser is immersed in the water. Start H_2S bubbling into the water in the beaker (Note 15) and cold water flowing through the condenser. Place 50 mL of HCl in the safety tube or separatory funnel and adjust the stopcock so that the acid runs in a slow stream into the flask. Apply gentle heat and bring the solution to a steady boil. If orpiment is present a yellow precipitate will appear in the receiver when distillation starts. Discontinue the distillation when bumping begins.

22.3.8 Remove the flame, change the receiver, add another 50-mL portion of the HCl to the contents of the flask and repeat the distillation procedure (Note 16). When the second distillation is completed, lower the receiver, and wash down the inside of the condenser and the outside of the

condenser tip with water. If any orpiment adheres to the walls of the condenser, wash it off with a small amount of NH_4OH , allow it to drain into the receiver, and then rinse again with water. Check the contents of the beaker for acidity, adding more acid if necessary, and allow the H_2S to bubble through the solution for a few minutes longer.

NOTE 15—The H_2S supply should be of sufficient amount to afford a uniform and good rate of flow during the entire distillation.

NOTE 16—With a properly prepared specimen, practically all of the arsenic comes over in the first distillation, the second one serving mainly as a safeguard.

22.3.9 Filter the contents of both beakers through the prepared and weighed Gooch crucible. Wash all of the precipitate from each beaker into the crucible with a stream of hot water from a wash bottle, using a policeman if necessary. Next, wash out the beaker with two 50-mL portions of alcohol followed by two 50-mL portions of boiling CS_2 (Note 17). When filtering these washings, adjust the suction so that each portion will be retained in the crucible for approximately 5 min (Note 18). Dry the crucible and its contents at $105 \pm 2^\circ\text{C}$ for 2 h. Cool in a desiccator and weigh.

NOTE 17—Bring the CS_2 to a boil on a steam bath, making sure there is no gas flame or electric arc in the immediate area in which the CS_2 is being used.

NOTE 18—This treatment ensures complete removal of all traces of sulfur that may have precipitated with the orpiment and which, if left in, would materially affect the final result, especially with a sample quite low in arsenic.

22.3.10 If there is any reason for believing that the reagents used are not arsenic-free, run blank determinations concurrently, using the same reagents in the same amounts and make the proper correction for the amount of orpiment thus found.

22.4 Calculation—Calculate the percentage of orpiment (As_2S_3) as follows:

$$\text{Orpiment, \%} = [(R - B)/S(1 - M)] \times 100$$

where:

R = residue from specimen, g (22.3.9),

B = residue from blank, g (if any) (22.3.10),

S = sample used, g, and

M = volatile matter (moisture) content of the sample, expressed as a decimal fraction.

23. Saponification Value

23.1 Definition:

23.1.1 *saponification value*—the number of milligrams of potassium hydroxide that react with 1 g of the moisture-free

sample. It is a measure of the alkali reactive groups in lac resins.

23.2 Reagents:

23.2.1 *Hydrochloric Acid, Standard (0.5 N)*—Add 45 mL of concentrated hydrochloric acid (HCl , sp gr 1.19) to about 900 mL of water, cool, and dilute to 1 L. Standardize against freshly standardized NaOH solution or by any other accurate method.

23.2.2 *Neutral Alcohol*, as prescribed in Section 21.2.1.

23.2.3 *Phenolphthalein Indicator Solution*, as prescribed in 21.2.2.

23.2.4 *Potassium Hydroxide, Alcoholic Solution (0.5 N)*—Prepare the solution as prescribed in 21.2.3, Item 2, except use 33 g of KOH in 1 L of the alcohol.

23.2.5 *Thymol Blue Indicator Solution*, as prescribed in 21.2.4.

23.3 Procedure:

23.3.1 Weigh to 1 mg approximately 1 g of the mixed sample (Section 7) into an alkali-resistant, standard-taper, ground-glass joint, 250-mL Erlenmeyer flask. Using a constant volume pipet, add 25 mL of the alcoholic KOH solution to the flask containing the specimen and to a second flask for use as a blank. Add 25 mL of the neutral alcohol to each flask and attach a standard-taper, ground-glass joint condenser to each flask. Connect the condensers with soda-lime tubes and reflux on a steam bath for at least 1 h. Wash down the condensers and the sides of the flasks with 20 mL of the neutral alcohol and titrate in accordance with 23.3.2 or 23.3.3.

23.3.2 In the case of bleached lac, add several drops of the phenolphthalein solution to each flask and titrate with 0.5 N HCl , with constant swirling of the contents of the flask, until the pink color just disappears.

23.3.3 In the case of orange shellac place several drops of the thymol blue indicator solution on a white porcelain spon plate. Titrate the solutions with 0.5 N HCl , with constant swirling, and determine the end point by transferring one or two drops of the solution on a glass rod to the indicator. The end point is when the color of the indicator changes to red.

23.4 Calculation—Calculate the saponification value as follows:

$$\text{Saponification value} = [(B - V)N \times 56.1]/S(1 - M)$$

where:

V = HCl required for titration of the specimen, mL,

B = HCl required for titration of the blank, mL,

N = normality of the HCl ,

S = sample used, g, and

M = volatile matter (moisture) content of the sample expressed as a decimal fraction.

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Standard Guide for Chemical Analysis of White Pigments¹

This standard is issued under the fixed designation D 34; the number immediately following the designation indicates the 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 to replace Method 7091, 7253 of Federal Test Method Standard No. 141 A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This guide covers procedures for the chemical analysis of white pigments.

2. Referenced Documents

2.1 ASTM Standards:

C 471 Test Methods for Chemical Analysis of Gypsum and Gypsum Products²

D 715 Test Methods for Analysis of Barium Sulfate Pigment³

D 717 Test Methods for Analysis of Magnesium Silicate Pigment³

D 718 Test Methods for Analysis of Aluminum Silicate Pigment³

D 719 Test Methods for Analysis of Diatomaceous Silica Pigment³

D 1199 Specification for Calcium Carbonate Pigments³

D 1301 Test Methods for Chemical Analysis of White Lead Pigments³

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 Paint Materials.

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² Annual Book of ASTM Standards, Vol 04.01.

³ Annual Book of ASTM Standards, Vol 06.02.

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D 1394 Test Methods for Chemical Analysis of White Titanium Pigments³

D 3280 Test Methods for Analysis of White Zinc Pigments³

3. Significance and Use

3.1 This compilation of available ASTM methods for the analysis of white pigment serves as a guide to chemists.

4. Test Methods

4.1 Tests shall be conducted in accordance with the following ASTM methods. Test procedures not covered by ASTM methods shall be agreed upon by the purchaser and seller.

4.2 *Lead Pigments*—Test Method D 1301.

4.3 *Zinc Pigments*—Test Methods D 3280.

4.4 *Titanium Dioxide Pigments*—Test Methods D 1394.

4.5 *Calcium Carbonate Pigments: Whiting, Paris White, Spanish White, Chalk*—Specification D 1199.

4.6 *Calcium Sulfate Pigments: Gypsum, Terra Alba, Plaster of Paris*—Methods C 471.

4.7 *Barium Pigments: Barytes or Barite and Blanc Fixe*—Test Methods D 715.

4.8 *Silica Pigments*—Test Methods D 719.

4.9 *China Clay*—Test Method D 718.

4.10 *Magnesium Silicate*—Test Methods D 717.

5. Keyword

5.1 chemical analysis; white pigment



Standard Methods of Chemical Analysis of Red Lead¹

This standard is issued under the fixed designation D 49; the number immediately following the designation indicates the year of original adoption 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 were updated editorially in September 1990.

1. Scope

1.1 These methods cover procedures for the chemical analysis of red lead having the approximate formula Pb_3O_4 (probably $\text{PbO}_2 \cdot 2\text{PbO}$).

1.2 *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.*

2. Referenced Documents

2.1 ASTM Standards:

D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²

D 215 Methods of Chemical Analysis of White Linseed Oil Paints³

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²

D 1193 Specification for Reagent Water⁴

D 1208 Test Methods for Common Properties of Certain Pigments²

D 1301 Test Methods for Chemical Analysis of White Lead Pigments²

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids⁵

3. Treatment of Sample

3.1 If the pigment is lumpy or not finely ground, grind it to a fine powder and mix thoroughly. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. All samples shall be preserved in stoppered bottles or containers.

4. Purity of Reagents

4.1 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.

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

5. Moisture

5.1 Determine moisture content with a 2-g specimen in accordance with Method A of Method D 280. The specimen is dried for 2 h at 105°C. The loss in weight is considered as moisture.

6. Organic Color

6.1 Boil 2 g of the sample with 25 mL of 95 % ethyl alcohol, let settle, decant the supernatant liquid; boil the residue with 25 mL of distilled water and decant as before; boil the residue with 25 mL of diluted NH_4OH (1+4) and again decant. Boil another 2-g portion of the sample with 25 mL of chloroform, let settle, and decant the supernatant liquid. If any one of the above solutions is colored, organic coloring matter is indicated. If the solutions remain colorless, organic colors are probably absent.

NOTE 1—If it is desired to test for organic colors resistant to the above reagents, the test procedures described in the following books may be used, taking into account the nature of the pigment involved: Zerr and Mayer, *Tests for Coal Tar Colors in Aniline Lakes*; Schultz and Julius, *A Systematic Survey of the Organic Coloring Matters*; Mulliken, *Identification of Pure Organic Compounds*; Commercial Dyestuffs, Vol III.

7. Total Lead and Insoluble Matter

7.1 Treat 1 g of the sample with 15 mL of HNO_3 (1+1) and sufficient H_2O_2 to dissolve all PbO_2 on warming. If any insoluble matter is present, add 25 mL of water, boil, filter, and wash with hot water. The insoluble matter contains free SiO_2 and should be examined for BaSO_4 and silicates, if appreciable.

7.2 To the original solution or filtrate from the insoluble matter add 20 mL of H_2SO_4 (sp gr 1.84) and evaporate to SO_3 fumes. Cool, add 150 mL of water, and 150 mL of 95 %

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 D 01.21 on Chemical Analysis of Paint and Paint Materials.

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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 11.01.

⁵ Annual Book of ASTM Standards, Vol 06.03.

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."

ethyl alcohol, let stand *cold* for 2 h, filter, on a Gooch crucible, wash with 95 % alcohol, dry at 105 to 110°C, and weigh as PbSO_4 . Calculate to PbO .

7.3 Red lead is rarely adulterated, but should the specimen contain soluble barium compounds, the PbSO_4 obtained in 8.2 will contain BaSO_4 . In this case, precipitate the lead as sulfide from a slightly acid (HCl) solution, dissolve the PbS in hot diluted HNO_3 , and determine the lead as sulfate or chromate.

7.4 If the specimen contains significant amounts of calcium or magnesium, boil the $\text{HNO}_3 - \text{H}_2\text{O}_2$ solution (7.1) until all the lead is converted into nitrate and then determine the lead as PbCrO_4 .

7.5 If soluble barium, calcium, or magnesium are to be determined, precipitate the lead as sulfide from a slightly acid solution (HCl), dissolve the PbS in hot diluted HNO_3 , and determine the lead as sulfate. Boil the filtrate from the PbS to expel H_2S , add a little bromine water to oxidize iron (if present), boil to expel bromine, and precipitate the barium with a few millilitres of H_2SO_4 (1+3). Filter and weigh as BaSO_4 . Calculate to BaO or BaCO_3 . To the filtrate from the BaSO_4 add NH_4OH in slight excess, filter off any precipitate of $\text{Fe}(\text{OH})_3 + \text{Al}(\text{OH})_3$, wash with hot water. Manganese, if present, can be precipitated by adding bromine and NH_4OH and warming. Filter, wash with hot water, ignite, and weigh as Mn_2O_3 . Unite all the filtrates, make slightly acid with acetic acid, heat to boiling and pass H_2S into the hot solution until saturated (20 to 30 min); add 5 g of NH_4Cl and let stand 5 h, filter off any ZnS , wash with H_2S water, dissolve the ZnS in hot diluted HCl and determine the zinc by titration with $\text{K}_4\text{Fe}(\text{CN})_6$. Or, boil off the H_2S , filter out any separated sulfur and determine the zinc as $\text{Zn}_2\text{P}_2\text{O}_7$. Calcium may be determined in the filtrate from the ZnS by expelling H_2S and then adding NH_4OH and ammonium oxalate. Titrate the calcium oxide precipitate using the procedure described in 13.3 of Method D 50. In the filtrate from calcium determine magnesium by precipitating with sodium phosphate solution, finally weighing as $\text{Mg}_2\text{P}_2\text{O}_7$.

8. Lead Peroxide (PbO_2) and True Red Lead (Pb_3O_4).

NOTE 2—Method of Diehl⁷ modified by Topt⁸—not applicable when substances are present, other than oxides of lead, that liberate iodine under conditions given, or substances such as metallic lead which reduce PbO_2 to PbO without the liberation of iodine.

8.1 *Solutions Required:* (a) *Red Lead Solution*—Dissolve in 1-L beaker 600 g of crystallized sodium acetate and 48 g of KI in about 500 mL of acetic acid (1+3) (made by mixing 150 mL of glacial acetic acid with 450 mL of water). Warm the beaker and contents on a steam bath, stirring occasionally, until a clear solution is obtained. Cool this solution to room temperature, dilute to exactly 1000 mL with the acetic acid (1+3) and mix thoroughly. If preferred, the red lead solution may be prepared separately for each titration, as follows: Dissolve 30 g of the crystallized sodium acetate and 2.4 g of KI in 25 mL of the acetic acid (1+3), warming gently and stirring until a clear solution is obtained. Cool this solution to room temperature, dilute to 50 mL with the acetic acid solution (1+3), and mix thoroughly.

8.2 *Sodium Thiosulfate Solution (0.1 N)*—Dissolve 24.83 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$), freshly pulverized and dried between filter paper, and dilute with water to 1 litre at the temperature at which the titrations are to be made. The solution is best made with well-boiled water free from CO_2 , or let stand 8 to 14 days before standardizing, as described in Section 5 of Method D 1959.

8.3 *Starch Solution*—Stir 2 to 3 g of potato starch with 100 mL of salicylic acid solution (1 %), and boil the mixture until the starch is practically dissolved, then dilute to 1 L (Note 3), or prepare as described in 7.8.2 of Method D 1959.

NOTE 3: *Lead Peroxide*—If the pigment contains an appreciable amount of nitrite (nitrate has no effect on the method), leach out water-soluble matter as below, dry the residue and determine PbO_2 as above, calculating to basis of original specimen.

9. Procedure

9.1 Weigh 1 g of the finely ground sample, transfer to a 200-mL Erlenmeyer flask, add 20 mL of water; then add as quickly as possible 40 mL of the "red lead solution" at room temperature. If the sample is red lead that has been extracted from a paint or paste, in place of the water use 10 mL of a mixture of 7 parts by volume of chloroform and 3 parts by volume of glacial acetic acid, and then add without delay the red lead solution. Add 30 mL of water containing 5 or 6 g of sodium acetate and titrate at once with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution, adding the latter rather slowly and keeping the liquid constantly in motion by whirling the flask. When the solution has become light yellow, rub-up any undissolved particles with the rod until free iodine no longer forms, wash off rod, add the $\text{Na}_2\text{S}_2\text{O}_3$ solution until *pale yellow*, add starch solution, and titrate until colorless. Add the 0.1 N iodine solution until blue color is just restored and subtract the amount used for the volume of $\text{Na}_2\text{S}_2\text{O}_3$ that had been added.

10. Calculation

10.1 Calculate the lead peroxide and true red lead contents as follows:

$$\text{PbO}_2 = I \times 0.942$$

$$\text{Pb}_3\text{O}_4 = \text{PbO}_2 \times 2.866 = I \times 2.7$$

where: I = iodine value of the $\text{Na}_2\text{S}_2\text{O}_3$ solution,

$$0.942 = \frac{\text{PbO}_2}{I_2} = \frac{239.19}{253.81}, \text{ and}$$

$$2.86616 = \frac{\text{Pb}_3\text{O}_4}{\text{PbO}_2} = \frac{685.57}{239.19}$$

11. Zinc

11.1 If the volume is appreciable, evaporate off the alcohol from the filtrate from total lead, make alkaline with NH_4OH , then acid with HCl (sp gr 1.19), add 3 mL more of HCl , dilute to about 250 mL with water, heat nearly to boiling and titrate with standard $\text{K}_4\text{Fe}(\text{CN})_6$ solution in accordance with 16.1.11 of Method D 215. Report as ZnO (includes cadmium). Iron, copper, or other interfering substances should first be removed as described in 25.2.3 of Method D 215.

12. Matter Soluble in Water

12.1 Determine the amount of water soluble material

⁷ Dinglers Polytechnisches Journal, DFJOA, Vol 246, p. 196.

⁸ Zeitschrift für Analytische Chemie, ZANCA, Vol 26, p. 296.

present in accordance with the procedure in Section 5 of Methods D 1208 or determine in accordance with Test Method D 1301.

13. Total Silica

13.1 Digest 5 g of the sample in a covered casserole with 5 mL of HCl and 15 mL of HNO_3 (1+1). Evaporate to dryness to dehydrate. Cool, treat with hot water and HNO_3 , boil, filter, wash with hot acid ammonium acetate solution; then dilute HCl and finally hot water. Ignite and weigh as SiO_2 . The residue may be treated with H_2SO_4 and HF in cases of doubt as to purity.

14. Carbon Dioxide

14.1 Determine carbon dioxide by the evolution method, using diluted HCl and stannous chloride.

15. Soluble Sulfates

15.1 *Sulfates Other Than Barium Sulfate*—Treat 0.5 g of the sample with 5 mL of water, 3 g of NH_4Cl , and 5 mL of HCl saturated with bromine. Digest (covered) on steam bath about 15 min. Add 25 mL of water, neutralize with dry Na_2CO_3 , and add about 2 g more. Boil 10 to 15 min, let settle, dilute with hot water, filter, and wash with hot water. Redissolve in HCl, reprecipitate as above, and wash thoroughly with hot water. Acidify the united filtrates with HCl, adding a slight excess; boil and add a slight excess of BaCl_2 solution (10 %). Let stand on a steam bath for 1 h. Filter and wash with hot water. Ignite and weigh as BaSO_4 . Calculate to SO_3 (includes SO_3 formed from SO_2).

15.2 Or, dissolve 0.5 g of the sample in 25 mL of water, 10 mL of NH_4OH (sp gr 0.90) and HCl in slight excess; dilute to

about 150 mL with water and add a piece of aluminum foil which should about cover the bottom of the beaker (being held on the bottom by means of a stirring rod). Heat gently till all lead is precipitated, decant through a filter, pressing the lead sponge with a flattened rod, and washing with hot water. Add to the filtrate a little bromine water, boil until bromine is expelled, add 15 mL of BaCl_2 solution (10 %), let stand on a steam bath for 1 h, filter, wash with hot water, ignite, and weigh as BaSO_4 (any SrSO_4 present is not decomposed in this method).⁹

16. Iron Oxide

16.1 Determine iron oxide in accordance with Section 12 of Method D 50, or in a large beaker, treat 20 g of the sample with 20 mL of water, 20 mL of HNO_3 (sp gr 1.42), and 3 mL of formaldehyde solution. Warm until all PbO_2 is dissolved, dilute with water, warm, filter off insoluble matter, and wash with hot water. Ignite filter and insoluble matter, and evaporate with H_2SO_4 and HF. To filtrate from insoluble matter add 14 mL of H_2SO_4 (1+1), filter off PbSO_4 , and wash. Dissolve the residue from HF and H_2SO_4 in H_2SO_4 and add to the filtrate from PbSO_4 . Dilute to 500 mL and determine iron colorimetrically in an aliquot, using the same amounts of HNO_3 , H_2SO_4 , and formaldehyde in the comparison solution.¹⁰ Calculate to Fe_2O_3 .

⁹ The solubility of BaSO_4 is increased by the presence of aluminum chloride. See Skoog, D. A. and West, D. M., *Fundamentals of Analytical Chemistry*, Holt, Rinehart and Winston, Inc., New York, 1969, p. 192.

¹⁰ Lunge-Berl, "Chemische-technisch Untersuchungs-Methoden," Vol 2, p. 95, 6th Ed.

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Standard Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese¹

This standard is issued under the fixed designation D 50; the number immediately following the designation indicates the year of original adoption 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 7141, 7151 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 These test methods cover procedures for the chemical analysis of yellow, orange, red, and brown pigments containing iron and manganese. The test methods apply specifically to the following pigments: synthetic hydrated yellow iron oxide, yellow ochre, red and brown iron oxides, raw and burnt umber, raw and burnt sienna, and venetian red.

1.2 The analytical procedures appear in the following order:

Dry Pigments	Sections	ASTM Method Refer- ences
Moisture and Other Volatile Matter	6	D 280
Loss on Ignition	7	
Coarse Particles	8	D 185
Matter Soluble in Water	9	D 1208
Organic Coloring Matter	10	
Iron Oxide	11 and 12	
Calcium Compounds (Reported as CaO)	13 and 14	
Sulfates Soluble in Hydrochloric Acid	15 and 16	
Lead Chromate (in Ochres)	17	
Calcium Carbonate (in Venetian Red)	18	C 25
Manganese (in Siennas and Umbers)	19 and 20	
Pigment Pastes in Oil		
Pigment Content	21	D 1208
Moisture and Other Volatile Matter	22	D 1208
Nonvolatile Matter in Vehicle	23	
Moisture by Distillation	24	D 1208
Coarse Particles and Skins	25	D 185
Consistency (Stormer)	26	D 562

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 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime²

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.

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² Annual Book of ASTM Standards, Vol 04.01.

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints^{3,4}

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

D 562 Test Method for Consistency of Paints Using the Stormer Viscometer⁴

D 1193 Specification for Reagent Water⁵

D 1208 Test Methods for Common Properties of Certain Pigments³

E 11 Specification for Wire Cloth Sieves for Testing Purposes²

3. Significance and Use

3.1 These test methods compile in one place, recommended procedures for analyzing inorganic colored pigments. These pigments are used extensively in paints, and for this reason their compositions are important to the formulators and user.

4. Preparation of Sample

4.1 Mix the sample thoroughly and take a representative portion for analysis. Reduce any lumps or coarse particles to a fine powder by grinding. Grind extracted pigments to pass a No. 80 (180- μ m) sieve (see Specification E 11 for detailed requirements). Discard any skins that do not pass through the sieve. Mix the finely ground pigment thoroughly.

5. Purity of Reagents

5.1 *Purity of Reagents*—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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vols 11.01 and 06.03.

⁶ "Reagent Chemicals, American 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."

water conforming to Specification D 1193.

DRY PIGMENTS

Moisture and Other Volatile Matter

6. Procedure

6.1 Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

Loss on Ignition

7. Procedure

7.1 Weigh accurately approximately 1 g of pigment into a previously weighed and ignited porcelain crucible (Note 1). Cover and ignite to constant weight over a bunsen or Meker burner or in an electric furnace at a temperature of approximately 900°C. Cool in a desiccator, weigh, and calculate the percent loss on ignition. This figure may include combined water, carbon dioxide (CO₂), organic matter, and some sulfuric oxide (SO₃) if much calcium sulfate (CaSO₄) is present. The CO₂ may be determined on a separate portion if desired.

NOTE 1—It is inadvisable to use platinum unless it is known that attacking substances are absent.

Coarse Particles

8. Procedure

8.1 Determine coarse particles in accordance with Test Method D 185.

Matter Soluble in Water

9. Procedure

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

Organic Coloring Matter

10. Procedure

10.1 Boil 2 g of the sample with 25 mL of water, let settle, and decant the supernatant liquid. Boil the residue with 25 mL of ethyl alcohol (95 %) and decant as before. Boil the residue with 25 mL of 1 N alcoholic sodium hydroxide (NaOH) solution and again decant. Boil another 2-g portion of the sample with 25 mL of chloroform, let settle, and decant the supernatant liquid. If any one of the above solutions is colored, organic coloring matter is indicated (Note 2). If the solutions remain colorless, organic colors are probably absent, but may be tested for by reference to procedures given in standard reference works, taking into account the nature of the pigment involved.⁷

NOTE 2—With this class of pigments indication of presence of an organic color may often be noted by the characteristic odor given off on ignition.

Iron Oxide

11. Reagents

11.1 *Stannous Chloride Solution* (SnCl₂·2H₂O)—Dis-

solve 50 g of SnCl₂·2H₂O in 300 mL of hydrochloric acid (HCl) (sp gr 1.19) and dilute with water to 500 mL. Keep the clear solution in a tightly stoppered bottle containing some metallic tin.

11.2 *Mercuric Chloride Solution* (HgCl₂)—Prepare a saturated solution of HgCl₂ (60 to 100 g/L).

11.3 *Sulfuric-Phosphoric Acid Mixture*—Mix 150 mL of sulfuric acid (H₂SO₄) (sp gr 1.84) with 150 mL of phosphoric acid (H₃PO₄) (85 %) and dilute with water to 1 L.

11.4 *Diphenylamine Indicator*—Dissolve 1 g of diphenylamine in 100 mL of H₂SO₄ (sp gr 1.84).

11.5 *Standard Potassium Dichromate Solution* (K₂Cr₂O₇) (0.1 N)—Dissolve 4.904 g of K₂Cr₂O₇ in water and dilute to 1 L. Standardize against the National Bureau of Standards standard sample No. 27 of Sibley iron ore.

11.6 *Potassium Ferricyanide Solution* (K₃Fe(CN)₆)—A very dilute solution is most satisfactory. Dissolve approximately 0.01 g of K₃Fe(CN)₆ in 50 mL of water. This solution must be made fresh when wanted because it does not keep.

12. Procedure

12.1 According to the amount of iron in the pigment, weigh 0.3 to 1.0 g of the pigment and ignite in a porcelain crucible at a dull red heat to destroy organic matter. Transfer to a 400-mL beaker and add 25 mL of HCl (sp gr 1.19). Cover with a watchglass and digest just short of boiling (80 to 90°C) until no dark specks can be seen in the insoluble residue. The addition of a few drops of SnCl₂ solution after adding the acid greatly assists dissolving of the iron. When the residue is light in color, the solution of iron may be considered complete. This may take from 15 min to 1 h, or longer.

12.2 Add 25 to 50 mL of water and heat to gentle boiling (avoid vigorous prolonged boiling). Slowly add SnCl₂ solution dropwise until the last drop makes the solution colorless or free from any tinge of yellow, then add 1 or 2 drops in excess. It is best to keep the watchglass on the beaker while adding the SnCl₂ solution, with agitation of the hot iron solution after each addition. If too much SnCl₂ is added by mistake, add potassium permanganate (KMnO₄) to the solution until a yellow color appears, then again add SnCl₂ dropwise until the yellow color just disappears, again adding one or two drops in excess. Dilute with 200 mL of cold water; then add all at once with vigorous stirring 15 mL of HgCl₂ solution. Let stand 3 to 4 min. A slight white precipitate should form. If none, or a heavy grayish precipitate forms, the determination should be discarded and repeated.

12.3 Add 15 mL of H₂SO₄-H₃PO₄ mixture and 3 drops of diphenylamine indicator solution. Titrate with K₂Cr₂O₇ solution, taking the sudden change of the dark green color to a blue-black color as the end point.

12.4 If preferred, K₃Fe(CN)₆ may be used as an external indicator. In this case omit the addition of the H₂SO₄-H₃PO₄ mixture and the diphenylamine indicator. Titrate with the K₂Cr₂O₇ solution as in 12.3, except toward the end take out a very small drop of the solution being titrated and touch this to a drop of the K₃Fe(CN)₆ solution, best placed on a paraffined surface. Toward the end point the blue is replaced by a bluish-green coloration, perceptible at the junction of the two solutions. Take as the end point when no trace of the

⁷ Reference may be made to the following: Payne, H. F., "Organic Coatings Technology," Vol II, John Wiley & Sons, Inc., New York, N. Y., 1961.

bluish-green coloration can be detected.

12.5 *Calculation*—Calculate the percent of iron oxide (Fe_2O_3), A , as follows:

$$A = (FV/S) \times 100 \quad (1)$$

where:

F = Fe_2O_3 equivalent of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution, g/mL,

V = $\text{K}_2\text{Cr}_2\text{O}_7$ solution required, mL, and

S = sample used, g.

Calcium Compounds (Reported as CaO)

13. Reagents

13.1 *Ammonium Oxalate Saturated Solution*—Heat to boiling 5 g powdered ammonium oxalate $[(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}]$ and 100 mL of water. Allow to cool.

13.2 *Hydrogen Peroxide (H_2O_2)* (30 %).

13.3 *Potassium Permanganate, Standard Solution (KMnO_4)* (0.1 N)—Dissolve 3.16 g of KMnO_4 in water and dilute to 1 L. Let stand 8 to 14 days, siphon off the clear solution (or filter through a medium porosity fritted disk), and standardize against the National Institute of Standards and Technology (NIST) standard sample No. 40 of sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) as follows: In a 400-mL beaker dissolve 0.2500 to 0.3000 g of the NIST $\text{Na}_2\text{C}_2\text{O}_4$ in 250 mL of hot water (80 to 90°C) and add 15 mL of H_2SO_4 (1+1). Titrate at once with 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 solution shall not be below 60°C by the time the end point has been reached. (More 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 (nonmercury type) as a stirring rod is most convenient. Keep the KMnO_4 solution in a glass-stoppered bottle painted black to keep out light, or in a brown glass bottle stored in a dark place.

13.4 Calculate the calcium oxide (CaO) value of the solution as follows:

$$C = (W_1 \times V_1) \times 0.4185 \quad (2)$$

where:

C = CaO equivalent of the KMnO_4 solution, g/mL,

W_1 = sodium oxalate used, g, and

V_1 = KMnO_4 solution required for the titration, mL.

$$0.4185 = \text{CaO}/\text{Na}_2\text{C}_2\text{O}_4 = 56.08/134.01 \quad (3)$$

14. Procedure

14.1 Weigh 2.5 g of sample, transfer to a porcelain crucible, and ignite at a dull red heat to destroy organic matter. Cool, transfer to a 600-mL beaker, and add 100 mL of HCl (1+1). Digest just short of boiling until no dark specks can be seen in the insoluble residue. Add ammonium hydroxide (NH_4OH) (sp gr 0.90) in slight excess and about 2 mL of H_2O_2 (30 %). Cool, transfer to a 500-mL graduated flask, and dilute to 500 mL. Mix thoroughly and filter through a dry paper.

14.2 Take 100 mL of the filtrate (corresponding to 0.5 g of sample), add a few drops of NH_4OH , heat to boiling, and add an excess of saturated ammonium oxalate solution. Con-

tinue the boiling until the precipitate becomes granular. Let stand about 30 min, filter, and wash three times with 20-mL aliquots of hot water until free of ammonium oxalate.

14.3 Place the beaker in which 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) and dilute to about 250 mL. Heat to 90°C and titrate at once with KMnO_4 solution. (The temperature of the solution should not be below 60°C when the end point is reached.)

14.4 *Calculation*—Calculate the percent of calcium compounds, A , in terms of CaO, as follows:

$$A = (V_2 C / 0.5) \times 100 \quad (4)$$

where:

V_2 = KMnO_4 solution required by the specimen, mL, and

C = CaO equivalent of the KMnO_4 solution, g/mL.

Sulfates Soluble in Hydrochloric Acid

15. Reagent

15.1 *Barium Chloride Solution*—Dissolve 117 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

16. Procedure

16.1 Weigh 1 g of the sample and add 30 mL of HCl (sp gr 1.19). Boil 10 min, add about 50 mL of water, boil, filter, and wash with hot water. Heat the solution to boiling, add NH_4OH in excess, filter, and wash the precipitate several times with hot water. Dissolve the precipitate in hot HCl (1+1), reprecipitate with NH_4OH , and wash well with hot water.

16.2 Combine the united filtrates and make distinctly acid with HCl , boil, and add dropwise, while stirring, an excess of BaCl_2 solution. Boil about 10 min. The BaSO_4 precipitate should normally stand for 2 to 4 h before filtering in order to coagulate the precipitate. If the pad is thick enough on the Gooch crucible and filtrate is free of sulfate, then standing may not be necessary. Filter on a Gooch crucible, and wash with hot water. Ignite at 900°C, and weigh as barium sulfate (BaSO_4).

16.3 *Calculation*—Calculate the percent of SO_3 , A , as follows:

$$A = [(P \times 0.343)/S] \times 100 \quad (5)$$

where:

P = weight of BaSO_4 , g, and

S = weight of original sample, g.

$$0.343 = \text{SO}_3/\text{BaSO}_4 = 80.06/233.42$$

Lead Chromate (in Ochres)

17. Procedure

17.1 Test for lead by any standard method of qualitative analysis. The following method is suggested: Dissolve a small portion of the pigment in cold nitric acid (HNO_3) (1+5), stirring vigorously. Add a few millilitres of potassium iodide

(KI) solution (100 g/L). The appearance of yellow crystals indicates the presence of lead.

Calcium Carbonate (in Venetian Red)

18. Procedure

18.1 Determine carbon dioxide (CO_2) in accordance with Test Methods C 25.

18.2 *Calculation*—Calculate the percent of calcium carbonate (CaCO_3), A , as follows:

$$A = [(W_3 \times 2.274)/S] \times 100 \quad (6)$$

where:

W_3 = CO_2 , g, and

S = sample used, g. $2.274 = \text{CaCO}_3/\text{CO}_2 = 100.09/44.0$

Manganese (in Siennas and Umbers)

19. Reagents

19.1 *Standard Potassium Permanganate Solution* (KMnO_4) (1 mL = 0.00035 g Mn)—Dissolve 1 g of KMnO_4 in 1 L of water. Age and standardize the solution as in Section 13, using one third the quantities of reagents specified therein. Calculate the manganese value of the solution as follows:

$$M = (W_4/V_3) \times 0.165 \quad (7)$$

where:

M = manganese equivalent of the KMnO_4 solution, g/mL,

W_4 = sodium oxalate used, g, and

V_3 = KMnO_4 solution required for the titration, mL.

19.2 *Sodium Bismuthate* (NaBiO_3).

19.3 *Ferrous Ammonium Sulfate Solution*—To 12 g of ferrous ammonium sulfate ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$) add 25 mL of H_2SO_4 (sp gr 1.84) and 25 mL of H_3PO_4 (85 %) and dilute to 1 L with water. Each day the solution is used run a blank titration against the KMnO_4 as follows: Measure into a 200-mL Erlenmeyer flask 50 mL of HNO_3 (1+3), cool, and add about 0.2 g of NaBiO_3 . Dilute with 50 mL of HNO_3 (3+97), filter by suction through Gooch crucible with a medium porosity fritted disk into a 300-mL Erlenmeyer flask, and wash with 50 mL of HNO_3 (3 + 97). Run in exactly 25 mL of the ferrous solution and titrate to a faint pink color with the KMnO_4 solution.

20. Procedure

20.1 Ignite 1 g of the sample in a porcelain crucible at a dull red heat to destroy organic matter. Transfer to a platinum dish and add 10 mL of water, 4 mL of H_2SO_4 (sp gr 1.84), 20 mL of HF (48 %), and a few drops of H_2SO_3 . Evaporate until the H_2SO_4 fumes freely, cool, and add 25 mL of HNO_3 (1+3). If no appreciable residue remains, transfer to a 100-mL volumetric flask, using 25 mL of HNO_3 (1+3) to rinse the dish, dilute to the mark with water, and mix thoroughly. If there is an appreciable residue, filter through a small qualitative filter paper and wash with water. Ignite the residue in a platinum crucible, and fuse with a little sodium pyrosulfate ($\text{Na}_2\text{S}_2\text{O}_7$) or potassium pyrosulfate ($\text{K}_2\text{S}_2\text{O}_7$). Dissolve in water, with the addition of a little HNO_3 , add to the main filtrate, and evaporate nearly to

dryness. Take up in HNO_3 (1+3) and transfer to the flask as before.

20.2 Pipet a 10-mL aliquot into a 200-mL Erlenmeyer flask and add 30 mL of water and 10 mL of HNO_3 (sp gr 1.42). Add about 0.5 g of NaBiO_3 and heat for a few minutes, or until the pink color has disappeared with or without the precipitation of magnesium dioxide (MnO_2). Add a few small crystals of sodium nitrite (NaNO_2) or potassium nitrite (KNO_2) to dissolve the MnO_2 and boil the solution several minutes to expel nitrous oxide fumes (a little anhydrous sodium carbonate (Na_2CO_3) will aid this). Add water to bring the volume up to 50 mL and cool to about 15°C . Add about 0.5 g of bismuthate and shake the flask well. Add 50 mL of HNO_3 (3+97), filter by suction through an asbestos felt into a 300-mL Erlenmeyer flask, and wash with 50 to 100 mL of the HNO_3 (3+97). Run in exactly 25 mL of ferrous ammonium sulfate solution and titrate to a faint pink color with KMnO_4 solution.

NOTE 3—If 25 mL of ferrous ammonium sulfate solution is not sufficient to decolorize the solution, add a further 10 mL or more as required. Adjust the blank (19.3) accordingly.

20.3 *Calculation*—Calculate the percent of manganese, A , as follows:

$$A = [(B_2 - V_4)M/W_5] \times 100 \quad (8)$$

where:

B_2 = KMnO_4 solution required for titration of the blank (18.3), mL,

V_4 = KMnO_4 solution required for back-titration of the specimen (19.2), mL,

M = manganese equivalent of the KMnO_4 solution, g/mL, and

W_5 = sample in the aliquot, g.

PIGMENT PASTES IN OIL

21. Pigment Content

21.1 Determine pigment content in accordance with Test Methods D 1208.

22. Moisture and Other Volatile Matter

22.1 Determine moisture and other volatile matter in accordance with Test Methods D 1208.

23. Nonvolatile Matter in Vehicle

23.1 Subtract the percent pigment content and percent volatile matter from 100 and report as percent vehicle.

24. Moisture by Distillation

24.1 Determine moisture by distillation in accordance with Test Methods D 1208.

25. Coarse Particles and Skins

25.1 Determine coarse particles and skins in accordance with Test Methods D 185.

26. Consistency (Stormer)

26.1 Determine consistency in accordance with Test Method D 562.

27. Keywords**27.1 brown pigment (iron and manganese); calcium; col-****ored pigment; iron oxide; red pigment; soluble sulfate; yellow pigment**

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Standard Specification for Zinc Oxide Pigments¹

This standard is issued under the fixed designation D 79; the number immediately following the designation indicates the year of original adoption 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 pigments commercially known as "zinc white" or zinc oxide. The pigments may be purchased in the dry form or as a paste in oil.

NOTE—Zinc oxides are used in many industries. For additional information, see Specification D 4295 and Test Methods D 4315 for descriptions of zinc oxide use in rubber compounding.

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out³
- D 332 Test Method for Relative Tinting Strength of White Pigments by Visual Observation³
- D 1483 Test Method for Oil Absorption of Pigments by Gardner-Coleman Method³
- D 2745 Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements³
- D 3280 Test Methods for Analysis of White Zinc Pigments³
- D 4295 Classification for Rubber Compounding Materials—Zinc Oxide⁴
- D 4315 Test Methods for Rubber Compounding Material—Zinc Oxide⁴
- E 20 Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 μm by Optical Microscopy⁵

3. Significance and Use

3.1 Zinc oxide functions as both a chemical and a pigment. It is used in a variety of applications including rubber, paint, reprography, glass, chemicals, etc. In paint, it contributes to mildew protection, ultraviolet absorption, hiding power, and neutralization of acids formed upon oxidation of the paint film.

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 D01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 09.01.

⁵ Annual Book of ASTM Standards, Vol 14.02.

4. Composition and Properties

4.1 *Dry Pigment, French Process*—In the manufacture of French process zinc oxide, metallic zinc is vaporized, either in a boiler or a refining column, and the resulting vapor is burned in a controlled manner in an orifice. The fine particles of zinc oxide are cooled enough to agglomerate and are collected by a system of fabric bags. French process oxide shall conform to the properties listed in Table 1.

4.2 *Dry Pigment, American Process*—In the manufacture of American process zinc oxide, zinc ore is reduced in the presence of a carbonaceous fuel. The resulting vapor is burned in a combustion chamber, and the fine particles of zinc oxide are cooled enough to agglomerate and are collected by a system of fabric bags. American process oxide shall conform to the properties listed in Table 1.

4.3 *Paste in Oil*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall conform to the following requirements:

Pigment, %	80 to 86
Linseed oil, %	14 to 20
Moisture and other volatile matter, max, %	0.5
Coarse particles and skins (total residue retained on a No. 325 (45- μm) sieve), max, % of the dry pigment	1.5

4.4 In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller. The most frequently specified properties are oil absorption, tinting strength, and particle shape. Appropriate test methods are listed in Section 6. In the event that either an acicular type or a nodular (spherical) type of zinc oxide is desired, the particle shape shall be determined by examining or photographing microscopic mounts (100 $\times$ or more) of the sample and the mutually agreed upon standard that are to be prepared as specified in Practice E 20.

5. Sampling

5.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing

TABLE 1 Composition

	French Process	American Process
Zinc oxide, min, %	99	98.5
Total sulfur, max, %	0.1	0.2
Moisture and other volatile matter, max, %	0.5	0.5
Total impurities, including moisture and other volatile matter, max, %	1.0	1.5
Coarse particles (total residue retained on a No. 325 (45- μm) sieve), max, %	0.10	0.25

between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4540 kg), except that for shipments of less than 10 000 lb, two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

6. Test Methods

6.1 Tests shall be conducted in accordance with the appropriate ASTM test methods, where applicable. Test procedures not covered by ASTM test methods shall be

mutually agreed upon by the purchaser and the seller.

6.1.1 *Coarse Particles*—Test Methods D 185.

6.1.2 *Moisture in Pigments*—Test Methods D 280.

6.1.3 *Chemical Analysis of Dry Pigments*—Methods D 3280.

6.1.4 *Oil Absorption of Pigments by Gardner-Coleman Method*—Test Method D 1483.

6.1.5 *Oil Absorption of Pigments by Spatula Rub-Out*—Test Method D 281.

6.1.6 *Instrumental Tinting Strength of White Pigments*—Test Method D 2745.

6.1.7 *Tinting Strength of White Pigments*—Test Method D 332.

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Standard Specification for Basic Carbonate White Lead Pigment¹

This standard is issued under the fixed designation D 81; the number immediately following the designation indicates the year of original adoption 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 material commercially known as basic carbonate white lead, used as a pigment and in putty. The pigment may be purchased in the dry form or as a paste in oil.

2. Referenced Documents

2.1 ASTM Standards:

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

D 1208 Test Methods for Common Properties of Certain Pigments³

D 1301 Test Methods for Chemical Analysis of White Lead Pigments³

3. Composition and Properties

3.1 *Dry Pigment*—The pigment shall be free of adulterants and shall contain not more than traces of impurities incident to well-controlled manufacture of high-grade basic carbonate white lead. The pigment shall conform to the following requirements:

Lead carbonate, %	62 to 75
Moisture and other volatile matter, max, %	0.7
Total other impurities, max, %	1.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	1.0

3.2 *Paste in Oil*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil. The paste shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall conform to the following requirements:

Pigment, min, %	89
Linseed oil, max, %	11
Moisture and other volatile matter, max, %	0.7
Coarse particles and skins (total residue retained on a No. 325 (45- μ m) sieve), max, % of the dry pigment	1.5

3.3 *Semipaste Containing Volatile Thinner*—The semi-

paste shall be made by thoroughly grinding the specified pigment with a mixture of linseed oil and a small amount of volatile thinner. The semipaste shall not be caked in the container and shall be readily stirred to a uniform mixture which shall mix readily with oil, turpentine, or volatile petroleum spirits to form a smooth paint of brushing consistency. The odor of the semipaste, as taken from the container, while drying or after drying, shall be not abnormally pungent or disagreeable. The semipaste shall conform to the following requirements:

Pigment, min, %	87.5
Linseed oil, max, %	10.5
Moisture and other volatile matter, % ⁴	1.5 to 3.0
Moisture, max, %	0.7
Coarse particles and skins (total residue retained on a No. 325 (45- μ m) sieve), max, % of the dry pigment	1.5

⁴ The volatile matter shall be turpentine, volatile petroleum spirits, or any mixture thereof.

3.4 The color and color strength, when specified, shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.1.1 *Coarse Particles*—Test Methods D 185.

5.1.2 *Moisture in Pigment*—Test Methods D 280.

5.1.3 *Lead Carbonate and Total Other Impurities*—Test Methods D 1301.

5.1.4 *Pigment, Linseed Oil, and Moisture and Other Volatile Matter in Paste in Oil*—Test Methods D 1208.

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 D01.31 on Pigment Specifications.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 81 - 21 T. Last previous edition D 81 - 80 (1986)¹¹.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

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Standard Specification for Red Lead Pigment¹

This standard is issued under the fixed designation D 83; the number immediately following the designation indicates the year of original adoption 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 four grades of red pigment commercially known as red lead. The pigment may be purchased in the dry form or as a paste in oil.

2. Referenced Documents

2.1 ASTM Standards:

- D 49 Test Methods for Chemical Analysis of Red Lead²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 *Dry Pigment*—The pigment shall be made by roasting litharge or metallic lead, or compounds of lead that yield litharge by heating, and shall consist entirely of oxides of lead, free of adulterants. The four grades of pigment shall conform to the following requirements:

True red lead (Pb_3O_4), min, %:	
85 % grade	85
95 % grade	95
97 % grade	97
98 % grade	98
Total impurities including moisture, water soluble matter, and matter insoluble in a mixture of nitric acid and hydrogen peroxide, max, %	1.0
Lead monoxide, PbO	remainder
Coarse particles (total residue retained on a 45- μm (No. 325) sieve), max, %	1.0

When mixed as indicated in the following table, the resulting paint, brushed on a smooth vertical iron surface, shall dry hard and elastic without running, streaking, or sagging:

Dry red lead	20 lb (9.1 kg)
Raw linseed oil	5 pt (2.4 L)
Turpentine	2 gills (0.24 L)
Liquid drier	2 gills (0.24 L)

3.2 *Paste in Oil*—The paste shall be made by thoroughly grinding the specified pigment with linseed oil (Note). The

paste as shipped by the seller, and for three months thereafter, shall not be caked in the container, and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall conform to the following requirements:

Pigment, %	92 to 94
Linseed oil, %	6.0 to 8.0
Moisture and other volatile matter, max, %	0.5
Coarse particles and skins (total residue retained on a No. 325 (45- μm) sieve), max, % of the dry pigment	1.5

When mixed as indicated in the following table, the resulting paint, brushed on a smooth, vertical iron surface, shall dry hard and elastic without running, streaking, or sagging:

Red lead paste	20 lb (9.1 kg)
Raw linseed oil	3 pt (1.4 L)
Turpentine	2 gills (0.24 L)
Liquid drier	2 gills (0.24 L)

NOTE—The storage of paste red lead in places of high temperature should be avoided, as heat accelerates the tendency of this material to cake or harden. Purchasers are cautioned that 85 % grade red lead should not be bought in paste form. The 95 % grade, if made into paste, should be used within a short period of time after grinding. When pure red lead paste is to be stored for a considerable period of time, the 97 % or 98 % grade of red lead should be specified. Therefore the manufacturer shall identify the grade of red lead used in the paste and the date of manufacture.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Chemical Analysis*—Methods D 49.

5.1.2 *Coarse Particles*—Test Methods D 185.

5.1.3 *Pigment, Linseed Oil, and Moisture and Other Volatile Matter in Paste in Oil*—Test Methods D 1208.

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 D01.31 on Pigment Specifications.

Current edition approved Aug. 31, 1984. Published December 1984. Originally published as D 83 - 21 T. Last previous edition D 83 - 73 (1984)¹.

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

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

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Standard Specification for Ochre Pigment¹

This standard is issued under the fixed designation D 85; the number immediately following the designation indicates the year of original adoption 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.

1. Scope

1.1 This specification covers ferrous earthy pigment included under the general term "ochre." The pigment may be purchased in the dry form.

2. Referenced Documents

2.1 ASTM Standards:

- D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile under the Test Conditions) in Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 The pigment shall be a hydrated oxide of iron permeating a siliceous base and shall be free from added impurities and added coloring matter. The pigment shall conform to the following requirements:

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 D 01.31 on Pigment Specifications.

Current edition approved June 26, 1987. Published August 1987. Originally published as D 85 - 21. Last previous edition D 85 - 81 (1986)¹.

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

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

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Ferric oxide, min, %	17
Calcium oxide, max, %	5
Lead chromate	none
Organic coloring matter	none
Moisture and other volatile matter, max, %	1.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	1.0

3.2 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Chemical Analysis of Dry Pigment*—Methods D 50.

5.3 *Coarse Particles*—Test Methods D 185.

5.4 *Mass Color and Tinting Strength*—Test Method D 387.

5.5 *Volatile Matter*—Test Methods D 1208.

5.6 *Moisture*—Test Methods D 280.

6. Keywords

6.1 ferrous; hydrated iron oxide; ochre; pigment



Standard Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green¹

This standard is issued under the fixed designation D 126; the number immediately following the designation indicates the year of original adoption 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 Method 7121 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.

⁶¹ NOTE—Keywords were added editorially in June 1991.

1. Scope

1.1 These test methods cover procedures for the chemical analysis of yellow, orange, and green pigments containing lead chromate and chromium oxide green.

1.2 The analytical procedures appear in the following order:

	Sections
CHROME YELLOW, CHROME ORANGE, AND MOLYBDATE ORANGE	
Organic Colors and Lakes	7
Moisture and Other Volatile Matter	8
Matter Soluble in Water	9
Lead Chromate	10 and 11
Total Lead	12
Sulfate	13 and 14
Carbon Dioxide	15
Molybdenum	16 and 17
Extenders	18 to 22
Calculation of Substances Other than Insoluble Lead Compounds	23 and 24
PURE CHROME GREEN AND REDUCED CHROME GREEN	
Organic Colors and Lakes	25
Moisture and Other Volatile Matter	26
Matter Soluble in Water	27
Iron Blue	28
Lead Chromate	29 and 30
Barium Sulfate and Insoluble Siliceous Material	31
Total Lead	32
Sulfate	33
Calcium Oxide Soluble in Acid	34 and 35
Extenders	36
Calculation of Insoluble Lead Compounds	37
CHROMIUM OXIDE GREEN	
Organic Colors and Lakes	38
Moisture and Other Volatile Matter	39
Matter Soluble in Water	40
Total Chromium as Chromium Oxide	41

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*

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 May 29, 1987. Published July 1987. Originally published as D 126 - 22. Last previous edition D 126 - 65 (1981)⁶¹.

statements are given in Note 3.

2. Referenced Documents

2.1 ASTM Standards:

- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 521 Test Methods for Chemical Analysis of Zinc Dust (Metallic Zinc Powder)²
- D 1013 Test Method for Determining Total Nitrogen in Resins and Plastics²
- D 1193 Specification for Reagent Water³
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

3. Summary of Test Methods

3.1 *Chrome Yellow, Chrome Orange, and Molybdate Orange:*

3.1.1 Organic colors and lakes are determined qualitatively by boiling the sample in water, then ethyl alcohol, and finally chloroform.

3.1.2 Moisture and other volatile matter are determined in accordance with Test Method A of Test Methods D 280.

3.1.3 Matter soluble in water is determined by boiling in water and filtering.

3.1.4 Lead chromate is determined by dissolving the sample in dilute HCl, filtering and titrating potentiometrically with FeSO₄ solution after addition of HClO₄.

3.1.5 Total lead is determined by precipitation as lead sulfide solution with H₂SO₄ and final precipitation as lead sulfate.

3.1.6 Sulfate is determined by dissolving the sample in acetic acid, neutralizing with sodium carbonate, plus addition of HCl to an aliquot followed by addition of BaCl₂ to precipitate as barium sulfate.

3.1.7 Carbon dioxide is determined by evolution.

3.1.8 Molybdenum is determined by precipitation as the sulfide, solution in HNO₃ and H₂SO₄, addition of NH₄OH

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 14.02.

and H_2SO_4 . The solution is reduced in a Jones reductor, collected under $\text{Fe}_2(\text{SO}_4)_3$ solution and titrated with KMnO_4 solution.

3.1.9 Extenders are either:

3.1.9.1 Calcium carbonate, calcium sulfate, magnesium carbonate or;

(a) The compounds in 3.1.9.1 are determined qualitatively by precipitation with ammonium solution.

(b) If chromium is present, it is reduced and the lead salts dissolved in dissolving solution. Hydroxides and hydrous oxides are precipitated by addition of HCl and NH_4OH and filtered. CaC_2O_4 is precipitated with calcium oxalate solution and filtered, ashed and weighed as CaO . Alternatively, the precipitate is dissolved in H_2SO_4 and titrated with KMnO_4 . Magnesium is determined on the filtrate from calcium determination by precipitation as the phosphate with ammonium phosphate solution.

3.2 Chromium Oxide Green:

3.2.1 Organic colors and lakes are determined qualitatively by boiling the sample in water, then ethyl alcohol, and finally chloroform.

3.2.2 Moisture and other volatile matter are determined in accordance with Test Method A of Test Methods D 280.

3.2.3 Matter soluble in water is determined by boiling in water and filtering.

3.2.4 Total chromium as chromium oxide is determined by dissolving the sample in dilute HCl , filtering and titrating potentiometrically with FeSO_4 solution after addition of HClO_4 .

4. Significance and Use

4.1 These test methods are for analysis designed as an aid in quality of yellow, orange, and green pigments containing lead chromate and chromium oxide green. Some sections may be applicable to analysis of these pigments when extracted from whole paints.

5. Purity of Reagents and Water

5.1 *Reagents*—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 *Water*—Unless otherwise indicated, references to water for use in the preparation of reagents and in analytical procedures shall be understood to mean reagent water conforming to Type II of Specification D 1193.

6. Preparation of Sample

6.1 Mix the sample thoroughly and take a representative portion for analysis. Reduce any lumps or coarse particles to a fine powder by grinding. Grind extracted pigments to pass a No. 80 (180- μm) sieve (Note 1). Discard any skins that do not pass through the sieve. Thoroughly mix the finely ground pigment and preserve in stoppered and suitably identified bottles or containers.

NOTE 1—Detailed requirements for this sieve are given in Specification E 11.

6.2 Moisten the weighed portions of extracted pigments with a small amount of suitable wetting agent (Note 1) before adding reagents for analysis.

NOTE 2—A 0.1 % solution of sodium dioctylsuccinosulfonate has been found satisfactory. (This material is sold under the trade name of Aerosol OT.) Wetting agents containing mineral salts, sulfates, or sulfonates which may be hydrolyzed to sulfates, should be avoided; the use of alcohol is also undesirable because of its tendency to reduce chromates.

NOTE 3: *Caution*—As 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 the sample. The wearing of a respirator and rubber or synthetic gloves are recommended. If hexavalent chromium materials come in contact with the skin, wash thoroughly with soap and water.

⁵ "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."

CHROME YELLOW, CHROME ORANGE, AND MOLYBDATE ORANGE

(Primrose, Lemon, and Medium Yellows; Chrome Oranges; Lead Molybdate or Basic Lead Chromate; Molybdate Orange)

ORGANIC COLORS AND LAKES

7. Procedure

7.1 Boil 2 g of the sample 2 min with 25 mL of water, let settle, and decant the supernatant liquid. Similarly, boil the residue with 25 mL of ethyl alcohol (absolute or 95 %) and decant as before. Likewise boil with 25 mL of chloroform and again decant. If any one of the above solutions is colored, organic colors are present. If all solutions remain colorless, organic colors are presumably absent. The presence of organic colors resistant to the above reagents is unlikely,

but may be tested for by reference to procedures given in standard reference works.⁶

MOISTURE AND OTHER VOLATILE MATTER

8. Procedure

8.1 Determine moisture and other volatile matter in accordance with Test Method A of Test Method D 280.

⁶ Reference may be made to the following: Payne, H. F., "Organic Coatings Technology," Vol II, John Wiley & Sons, Inc., New York, NY, 1961.

MATTER SOLUBLE IN WATER

9. Procedure

9.1 Place 2.5 g of the sample in a graduated 250-mL flask. Add 100 mL of water and boil for 5 min. Cool, dilute to exactly 250 mL, mix, and allow to settle. Filter the supernatant liquid through a dry paper and discard the first 20 mL. Evaporate 100 mL of the clear filtrate to dryness in a weighed dish, heat for 1 h at 105 to 110°C, cool, and weigh.

9.2 *Calculation*—Calculate the % of matter soluble in water as follows:

$$\text{Matter soluble in water, \%} = (R \times 2.5 \times 100)/S$$

where:

R = weight of residue, and

S = specimen weight, g.

LEAD CHROMATE⁷

10. Reagents

10.1 *Dissolving Solution*—Saturate 1 L of water with NaCl. Filter. Add to the filtered solution 150 mL of water and 100 mL of HCl (sp gr 1.19).

10.2 *Ferrous Sulfate, Standard Solution* (0.3 N)—Dissolve 86 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 500 mL of water to which 30 mL of H_2SO_4 (sp gr 1.84) has been added with constant stirring. Dilute to 1 L and standardize not more than 6 h before use by potentiometric titration against 0.7-g portions of $\text{K}_2\text{Cr}_2\text{O}_7$.

11. Procedure

11.1 Dissolve 1 g of the sample in 150 mL of the dissolving solution. Agitate for 10 to 15 min, keeping the solution cold until dissolution is complete (Note 4). If dissolution is not complete, filter through fine grade filter paper and wash with three 10-mL portions of cold dissolving solution. Add 10 mL of HClO_4 (70 %), dilute to 250 mL, and titrate potentiometrically with FeSO_4 solution.

NOTE 4—Incomplete solution of the pigment is evidence of the possible presence of barium sulfate, silica, silicates, or other acid-insoluble extenders (see Section 18). Some chrome yellows may contain organic addition agents and will give a turbid solution at this point.

Newer chemically resistant-type lead chromate type pigments (silica encapsulated) cannot be decomposed by the procedures described in this method. Pigments of this type may require treatment with strong alkali hydroxide or hydrofluoric acid.

Also, if trivalent antimony has been used in manufacturing the product, pentavalent antimony may be present which would interfere in the determination of lead chromate.

11.2 Alternatively, the solution may be reduced by a known excess of FeSO_4 solution and back-titrated with KMnO_4 solution in the presence of MnSO_4 , or excess KI may be added and the liberated iodine titrated with $\text{Na}_2\text{S}_2\text{O}_3$ solution, using starch indicator. The iodine liberation method is not applicable in the presence of molybdenum.

⁷ Sections 23 and 24 under "Calculation of Substances Other than Insoluble Lead Compounds" should be read carefully before proceeding with the analyses described in Sections 10 to 22.

TOTAL LEAD⁷

12. Procedure

12.1 Dissolve 0.5 g of sample as described in Section 11. Add 5 mL of ethyl alcohol (95 % or absolute) and boil until the chromium is reduced, as indicated by a green color. Filter if any insoluble residue is present, retaining the filtrate and washings for the determination. Add NH_4OH (sp gr 0.90) to this solution until a faint precipitate begins to form; then add 5 mL of HCl (sp gr 1.19) slowly, dilute to 500 mL, and pass a rapid current of H_2S into the solution until precipitation is complete. Settle, filter, and wash with water containing H_2S .

12.2 Rinse the precipitate from the filter (Note 5) into a beaker containing 25 mL of HNO_3 (1+3) and boil until all PbS has dissolved. Add 10 mL of H_2SO_4 (1+1) and evaporate to strong fumes of SO_3 . Cool and add 50 mL of water and 50 mL of ethyl alcohol (95 %) (Note 6). Let stand 1 h; then filter on a tarred Gooch crucible. Wash with ethyl alcohol (95 %), dry, ignite at 500 to 600°C, and weigh as PbSO_4 .

NOTE 5—If a trace of sulfide remains on the paper, the stained portion of the paper may be separately treated with bromine water, the paper filtered off, and the filtrate added to the body of the solution.

NOTE 6—Any sulfur remaining from decomposition of the sulfides may be mechanically removed as a globule of solidified sulfur at this point.

SULFATE⁷

13. Reagents

13.1 *Barium Chloride Solution*—Dissolve 117 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in water and dilute to 1 L.

13.2 *Dissolving Solution*—See Section 10.1.

13.3 *Sodium Carbonate Solution (saturated)*—Prepare a solution containing excess Na_2CO_3 at laboratory temperature, and free of SO_4 . Decant the clear solution for use as required.

14. Procedure

14.1 Digest 1.25 g of the sample with 100 mL of dissolving solution at 100°C for 5 min. Add 25 mL of glacial acetic acid and 15 mL of ethyl alcohol and heat gently for 10 min to reduce chromium, as indicated by the green color of the solution. Cool. Neutralize with saturated Na_2CO_3 solution and add a slight excess. Transfer to a 250-mL volumetric flask, dilute to the mark with distilled water, and mix. Filter without washing through a dry filter paper, discarding the first 10 to 15 mL.

14.2 Take a 200-mL aliquot of the filtrate, neutralize with HCl (1+1), and add 10 mL excess. Heat to boiling and boil for 5 min. To the gently boiling solution, add 15 mL of BaCl_2 solution dropwise with constant stirring. Digest on a steam bath for 2 h. Filter through an ignited tarred Gooch crucible, wash with HCl (1+99), and finally with hot water. Dry at 105 to 110°C, ignite at 900°C, and weigh.

CARBON DIOXIDE⁷

15. Procedure

15.1 Determine CO_2 by the evolution method on 2.5 g of

the sample, using dilute HNO_3 free of NO or NO_2 and absorbing the CO_2 in soda lime or in KOH solution.

MOLYBDENUM⁷

16. Reagents

16.1 *Ferric Sulfate Solution*—Dissolve 20 g of $\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ in 200 mL of water to which has been added 50 mL of H_2SO_4 (sp gr 1.84) and 20 mL of H_3PO_4 (85 %), and dilute to 1 L.

16.2 *Jones Reductor*—The reductor shall contain at least a 35-cm column of amalgamated zinc, prepared by shaking 20 to 30-mesh zinc free of iron or carbon with HgCl_2 solution (20 g/L) in sufficient quantity to produce an amalgam containing 1 to 5 % of mercury, and supported by a suitable inert pad of asbestos, glass wool, or other inert material.

16.3 *Potassium Permanganate, Standard Solution* (0.1 N)—Dissolve 3.16 g of KMnO_4 in water and dilute to 1 L. Let stand 8 to 14 days, siphon off the clear solution (or filter through a medium porosity fritted disk), and standardize against the National Bureau of Standards standard sample No. 40 of sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) as follows: In a 400-mL beaker dissolve 0.2500 to 0.3000 g of the Bureau of Standards sodium oxalate in 250 mL of hot water (80 to 90°C) and add 15 mL of H_2SO_4 (1+1). Titrate at once with 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 solution shall not be below 60°C by the time the end point has been reached. (More 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 (non-mercury type) as a stirring rod is most convenient.) Keep the KMnO_4 solution in a glass-stoppered bottle painted black to keep out light, or in a brown glass bottle stored in a dark place.

17. Procedure

17.1 Dissolve 1 g of the sample as described in Section 11. Add 5 mL of ethyl alcohol (95 % or absolute) and boil until chromium is reduced. Filter if any insoluble residue is present, retaining the filtrate and washings. Add NH_4OH (sp gr 0.90) cautiously until a faint precipitate begins to form, then add 15 mL of H_2SO_4 (sp gr 1.84) and dilute to 300 mL. Heat to boiling, pass in a rapid stream of H_2S for 15 min, and dilute with 300 mL of hot water. Pass in H_2S for 10 min, boil for 3 min, and cool. Pass in H_2S for 10 min, and let stand at room temperature for 1 h. Filter and wash with H_2SO_4 (1+99) saturated with H_2S .

17.2 Rinse the sulfide precipitate into the original beaker and add 20 mL of HNO_3 (sp gr 1.42) and 5 mL of H_2SO_4 (sp gr 1.84) (see Note 5). Cover and heat to fumes. Cool, add 10 mL of HNO_3 (sp gr 1.42), and again fume. Repeat this operation if necessary until a light-colored solution is obtained. Wash the cover and inside of the beaker and fume again to remove all HNO_3 . Dilute to 200 mL and add NH_4OH (1+4) until neutral; then add 10 mL of H_2SO_4 (sp gr 1.84).

17.3 Cool the solution and reduce by passing through a Jones reductor at a rate not exceeding 100 mL/min, collecting the effluent under 200 mL of $\text{Fe}_2(\text{SO}_4)_3$ solution. Titrate with KMnO_4 solution. A blank determination should also be made.

EXTENDERS⁷

18. General Considerations

18.1 Extenders fall into two groups, depending on their solubility or insolubility in the dissolving solution described in Section 10, as follows:

A. *Extenders Soluble in Dissolving Solution*—Calcium sulfate (gypsum), calcium carbonate (whiting), and magnesium carbonate.

B. *Extenders Insoluble in Dissolving Solution*—Silica, magnesium silicate, and clay (Note 7).

18.2 Extenders of group A may be present if the analysis shows sulfates and carbonates to be in the pigment, and are absent if sulfate and carbonate are absent. Since the latter situation rarely exists, it is advisable to test for the presence of calcium and magnesium to determine if extenders are present. Extenders of group B are recognized as an insoluble residue following acid solution of the pigment, and may be determined quantitatively if desired, by the method described in Section 31. Extenders of group A, if present, may affect the calculation of insoluble lead compounds as given in Section 23. Their qualitative or quantitative estimation may be necessary.

NOTE 7—Some lead chromates may contain zirconium or titanium compounds, some of which are insoluble in the dissolving solution, but are not to be considered as extenders, since they have been added to improve the properties of the pigment.

Qualitative Detection of Extenders of Group A

19. Reagents

19.1 *Ammonium Phosphate Solution*—Dissolve 100 g of $(\text{NH}_4)_2\text{HPO}_4$ in water and dilute to 1 L.

20. Procedure

20.1 Dissolve 1 g of the sample as described in Section 11. Add 5 mL of ethyl alcohol (95 % or absolute) and boil until the chromium is reduced. An insoluble residue at this point denotes the presence of extenders of Group B. Filter if necessary and wash well.

20.2 To the filtrate, add NH_4OH (1+4) until just ammoniacal, boil 5 min, and allow to digest in a warm place until the precipitate has coagulated. Filter, washing well with hot water and reserving the filtrate. Dissolve the precipitate on the filter with HCl (1+1), washing back into the original beaker. Reprecipitate, filter, and wash as before.

20.3 Combine the washings, make just acid with HCl (1+1), and evaporate to a volume of about 250 mL. Add 50 mL of $(\text{NH}_4)_2\text{HPO}_4$ solution, cool, and add 50 mL of NH_4OH (sp gr 0.90). Allow to stand overnight. A precipitate indicates the presence of extenders of Group A.

Quantitative Determination of Extenders of Group A

21. Reagents

21.1 *Ammonium Oxalate Solution*—Dissolve 30 g of

ammonium oxalate in water and dilute to 1 L.

21.2 *Ammonium Phosphate Solution*—See Section 19.

22. Procedure

22.1 If the sample is a chrome yellow or orange use the procedure given in Section 14 for dissolving and reducing chromium. For other chromium pigments heat gently 2 g of sample in a porcelain dish without ignition until iron blue, if present, is just decomposed. Transfer to a beaker and dissolve the lead salts in 150 mL of dissolving solution as described in Section 11.

22.2 Add 20 mL of HCl (sp gr 1.19) and digest 1 h at 100°C. Dilute to 300 mL, filter and wash thoroughly. Add NH_4OH (1+4) to the filtrate and washings until just ammoniacal, boil 5 min and allow to digest in a warm place until the hydroxides and hydrous oxides are coagulated. Filter, washing well with diluted water and reserve the filtrate. Redissolve the precipitate with HCl (1+1) washing back into the original beaker. Reprecipitate, filter and wash as before. Combine the washings with the original filtrate and add 50 mL of ammonium oxalate solution. Filter off the CaC_2O_4 precipitate on quantitative paper. Transfer to a tarred crucible, ash, ignite at 1300°C, cool in a desiccator, and weigh as CaO . Alternatively, the washed precipitate may be dissolved in H_2SO_4 (1+1) and the resulting solution titrated hot with 0.1 N KMnO_4 solution, as described in the Procedure section under Calcium in Test Methods D 521.

22.3 Make the filtrate from the calcium determination slightly acid with HCl (1+1) and evaporate to 250 mL volume. Add 50 mL of $(\text{NH}_4)_2\text{HPO}_4$ solution, cool, and add 50 mL of NH_4OH (sp gr 0.90). Allow to stand overnight. Filter on suitable quantitative paper, wash with NH_4OH (1+19), ignite in a platinum crucible at 1050°C for 1 h and weigh. Care must be taken to char the paper slowly before igniting.

22.4 *Calculation*—Calculate the percent of extenders (as oxides) as follows:

$$\text{Calcium oxide, \%} = \text{weight of CaO} \times 50$$

$$\text{Magnesium oxide, \%} = \text{weight of Mg}_2\text{P}_2\text{O}_7 \times 18.11$$

The calculation of the amount of extenders as carbonates or sulfates is described in Section 24.

CALCULATION OF SUBSTANCES OTHER THAN INSOLUBLE LEAD COMPOUNDS

23. Calculations Where Extenders Are Absent

23.1 "Insoluble lead compounds" in chrome yellow and chrome orange may consist of PbCrO_4 , PbSO_4 , $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$, PbO , or PbMoO_4 . The first two are characteristic of the chrome yellows, the first, third, and fourth of the basic chrome oranges, and the first, second, and last of molybdate oranges. For purpose of determining conformance with specification requirements, where "insoluble lead compounds" are defined as above, make the following calculations if extenders are absent:

23.2 If molybdenum is present, calculate the percent of PbMoO_4 from the titration of Section 17 as follows:

$$A = \text{PbMoO}_4, \% = \text{mL titration} \times \text{normality of KMnO}_4 \times 12.24$$

23.3 Calculate the percent of PbCrO_4 from the titration of Section 11 as follows:

$$B = \text{PbCrO}_4, \% = \text{mL titration} \times \text{normality of FeSO}_4 \times 10.77$$

23.4 Calculate the percent of total lead as oxide and the percent of excess PbO from the analysis of Section 12 as follows:

$$C = \text{PbO, \%} = \text{grams of PbSO}_4 \times 147.2$$

$$D = \text{excess PbO, \%} = C - (0.6906 B + 0.6078 A)$$

23.5 Calculate the sulfate as SO_3 from the analysis of Section 14 as follows:

$$E = \text{SO}_3, \% = \text{grams of BaSO}_4 \times 34.3$$

23.5.1 If E is equal to or greater than 0.3587 D , the % of PbSO_4 equals 1.3587 D , and the % total insoluble lead compounds equals $A + B + 1.3587 D$.

23.5.2 If E is less than 0.3587 D , the percentage of PbSO_4 equals 3.788 E , and a new excess of PbO is calculated as follows:

$$F = \text{Excess PbO, \%} = D - 2.788 E$$

23.6 Calculate the percent of CO_2 from the analysis of Section 15 as follows:

$$G = \text{CO}_2, \% = \text{grams of CO}_2 \times 40$$

23.6.1 If G is equal to or greater than 0.1314 F , the percent of basic lead carbonate ($2 \text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$) is 1.1584 F and the percent of total insoluble lead compounds is $A + B + 3.788 E + 1.1584 F$.

23.6.2 If G is appreciably in excess, extenders are probably present. If G is less than 0.1314 F , the percent of $2 \text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$ is 8.813 G and the excess PbO is:

$$H = \text{Excess PbO, \%} = F - 7.608 G$$

23.6.2.1 The percent of total insoluble lead compounds is $A + B + 3.788 E + 8.813 G + H$.

24. Calculations Where Extenders Are Present

24.1 If extenders are present, calculate the CO_2 equivalent as follows:

$$I = \text{CO}_2 \text{ equivalent of extenders} \\ = \text{CaO, \%} \times 0.786 + \text{MgO, \%} \times 1.100$$

24.2 If I is less than G , use $G - I$ as the net CO_2 for purposes calculating the $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$ content of the pigment.

$$\text{Total extenders} = \text{CaO, \%} + \text{MgO, \%} + I$$

24.3 If I is greater than G , some of the extender is present as CaSO_4 (Note 8). Calculate the total percent of extenders as follows:

$$J = \text{CO}_2 \text{ equivalent of MgO} = \text{MgO \%} \times 1.100$$

$$K = \text{CO}_2 \text{ present as CaCO}_3 = G - J$$

$$\text{CaCO}_3, \% = 2.73 K$$

$$L = \text{CaO present as sulfate, \%} = \text{CaO, \%} \pm 1.27 K$$

$$\text{CaSO}_4, \% = 2.43 L$$

$$\text{SO}_3 \text{ combined as CaSO}_4, \% = 1.43 L$$

24.3.1 $E - 1.43 L$ equals the net SO_3 to be used for calculating the PbSO_4 content of the pigment. If I is greater

than G , no calculation of $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$ is necessary, and the excess PbO is given by F .

Total extenders, % = $2.100 \times \text{MgO, \%} + 2.73 K + 2.43 L$

PURE CHROME GREEN AND REDUCED CHROME GREEN

ORGANIC COLORS AND LAKES

25. Procedure

25.1 Determine organic colors and lakes in accordance with Section 7.

MOISTURE AND OTHER VOLATILE MATTER

26. Procedure

26.1 Determine moisture and other volatile matter in accordance with Section 8.

MATTER SOLUBLE IN WATER

27. Procedure

27.1 Determine matter soluble in water in accordance with Section 9.

IRON BLUE

28. Procedure

28.1 Determine the total nitrogen (by the Kjeldahl-Gunning method) (Note 9) on 1 g of the sample, adding 2 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ before digestion for at least $2\frac{1}{2}$ h.

NOTE 9—For further details refer to Test Method D 1013.

28.2 *Calculation*—Calculate the percent of iron blue as follows:

Iron blue, % = nitrogen, % $\times 3.4$

NOTE 10—Qualitative tests may be made for other nitrogen-containing blue pigments.

LEAD CHROMATE

29. Reagents

29.1 *Ferrous Sulfate, Standard Solution* (0.3 N)—See Section 10.2.

29.2 *Potassium Permanganate Standard Solution* (0.1 N)—Prepare as described in 16.3. Standardization is unnecessary except for the alternative procedure given in Section 30.

30. Procedure

30.1 Mix thoroughly 1 g of sample with at least 10 g of Na_2O_2 in a 30-mL pure iron crucible. Heat gently until the fusion is complete; then heat strongly, rotating the crucible with iron tongs (not Nichrome or chromium plate) for a few minutes to ensure complete fusion. Allow to cool; then transfer the crucible and cover to a beaker containing 250 mL of water. When the action has ceased, rinse and remove the crucible and cover, and boil for at least 10 min to destroy excess peroxide. Make just acid with H_2SO_4 (1+1) and add about 35 mL in excess. Add 2.5 mL of 0.1 N KMnO_4 solution and heat to boiling. Add 10 mL of HCl (1+4) and boil to reduce manganese and lead as indicated by clearing of the solution. If the solution is not clear after 5 min boiling,

NOTE 8—It is impractical to detect by chemical means whether a pigment contains CaCO_3 and PbSO_4 , or CaSO_4 and $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$. This calculation assumes the former as more probable.

add 5 mL of additional HCl (1+4), and repeat until a clear solution is obtained. Cool and titrate with FeSO_4 solution on a potentiometric apparatus; or alternatively, add a measured excess of FeSO_4 and back-titrate with KMnO_4 solution in the presence of MnSO_4 .

BARIUM SULFATE AND INSOLUBLE SILICEOUS MATERIAL⁸

31. Procedure

31.1 Heat gently 1 g of sample in a porcelain dish without ignition until the iron blue is just decomposed. Transfer to a beaker and dissolve the lead salts as described in Section 11, warming if necessary. Filter through a tared Gooch crucible, wash thoroughly, ignite at 600 to 800°C, and weigh.

31.2 *Calculation*—Calculate the percent of BaSO_4 and insoluble siliceous material as follows:

BaSO_4 and insoluble siliceous material, %
= weight of precipitate $\times 100$

TOTAL LEAD

32. Procedure

32.1 Determine total lead on the filtrate from the determination of BaSO_4 and insoluble matter (Section 31) in accordance with the directions of Section 12.

SULFATE

33. Procedure

33.1 After decomposition of the iron blue in 1.25 g of sample as described in Section 31, digest the residue with 20 mL of HCl (sp gr 1.19) at 100°C until solution is complete. Add 300 mL of water and heat to boiling. Filter and wash thoroughly. Determine sulfate on the filtrate and washings as described in Section 14.

CALCIUM OXIDE SOLUBLE IN ACID

34. Reagents

34.1 *Ammonium Oxalate Solution*—See 21.1.

35. Procedure

35.1 Decompose 2 g of sample as described in Section 31. Add 20 mL of HCl (sp gr 1.19) and proceed in accordance with 21.1 to the end of the calcium determination.

35.2 *Calculation*—Calculate the percent of CaO soluble in acid as follows:

⁸ See Note 7 of Section 18.

CaO soluble in acid, % = grams CaO $\times$ 50

EXTENDERS

36. Procedure

36.1 The method described in Sections 31, 34, and 35 suffices for the detection and estimation of all common extenders except MgCO_3 . This may be tested for the filtrate

from the CaO determination by the method described in Section 22.

CALCULATION OF INSOLUBLE LEAD COMPOUNDS

37. Procedure

37.1 The insoluble lead compounds may be calculated by the methods of Sections 23 and 24.

CHROMIUM OXIDE GREEN

ORGANIC COLORS AND LAKES

38. Procedure

38.1 Determine organic colors and lakes in accordance with Section 7.

MOISTURE AND OTHER VOLATILE MATTER

39. Procedure

39.1 Determine in accordance with Section 8.

MATTER SOLUBLE IN WATER

40. Procedure

40.1 Determine in accordance with Section 9.

TOTAL CHROMIUM AS CHROMIUM OXIDE

41. Procedure

41.1 Using 0.4 g of the sample proceed as in Section 30.

41.2 *Calculation*—Calculate the percent of chromium as Cr_2O_3 as follows:

$\text{Cr}_2\text{O}_3, \% = \text{mL titration} \times \text{normality of } \text{FeSO}_4 \times 63.34$

42. Precision and Bias

42.1 Data are not available to determine the precision and bias of these test methods. There are no plans at present to obtain such data.

43. Keywords

43.1 chromium oxide green pigment; green pigment containing lead chromate and chromium oxide; lead chromate pigment; lead containing pigment; molybdate pigment; orange pigment containing lead chromate and chromium oxide; yellow pigment containing lead chromate and chromium oxide

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Standard Test Methods for Specific Gravity of Pigments¹

This standard is issued under the fixed designation D 153; the number immediately following the designation indicates the year of original adoption 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 4181 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.

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

1. Scope

1.1 These test methods cover three procedures for determining the specific gravity of pigments, as follows:

Test Method A—For Routine Testing of Several Samples Simultaneously.

Test Method B—For Tests Requiring Greater Accuracy than Test Method A.

Test Method C—For Rapid and Accurate Testing of Single Samples.

1.2 The specific gravity value obtained by these procedures may be used with the weight of a dry pigment to determine the volume occupied by the pigment in a coating formulation.

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.* For specific hazard statements, see Sections 5, 11, and 15.

2. Referenced Document

2.1 ASTM Standard:

D 1193 Specification for Reagent Water²

3. Purity of Reagents

3.1 *Purity of Water*—Reference to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

TEST METHOD A—FOR ROUTINE TESTING OF SEVERAL SAMPLES SIMULTANEOUSLY

4. Apparatus and Materials

4.1 *Pycnometer*—A pycnometer (Note 1) having a 50-mL capacity.

NOTE 1—The Weld type with the cap seal on the outside of the neck of the bottle is preferred because there is less danger of trapping air just

under the capillary tube than with types having the ground glass seal on the inside of the neck.

4.2 *Water Bath*, maintained at $25 \pm 0.5^\circ\text{C}$ and equipped with a stirring device.

4.3 *Manometer*, open- or closed-tube (see Part f of the apparatus for Test Method C), made of glass tubing 6 mm in diameter, fitted with rubber pressure tubing attached to a T-joint leading to the desiccator and the pump. For the open-tube type 860 mm of mercury shall be used. The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, shall be considered the absolute pressure of the system in millimetres of mercury.

4.4 *Desiccator*, glass, constructed with heavy walls to withstand a vacuum of one atmosphere, and with an opening at the side.

4.5 *Vacuum Pumps*—A laboratory water vacuum-type pump (Note 2), to remove the greater portion of air in the desiccator, and an oil vacuum-type pump, motor-driven, and capable of reducing the absolute pressure of the system to 3 mm.

NOTE 2—The water vacuum pump may be omitted if the rate of evacuation with the oil pump can be controlled so as to avoid a rapid ebullition of entrapped air and possible loss of specimen.

4.6 *Thermometer*, having a range from 0 to 60°C , and graduated in 0.1°C divisions.

4.7 *Weighing Bottle*, wide-mouth cylindrical glass (about 30 mm in height and 70 mm in diameter), provided with a ground-glass stopper.

4.8 *Immersion Liquid*—Kerosine has been found to be a good wetting vehicle for most pigments, and shall be used generally as the immersion liquid. Refined, white kerosine of narrow evaporation and boiling range shall be used. With some pigments that are not wetted well with kerosine, other immersion liquids such as glycerin, ethylene glycol, tetrahydronaphthalene, etc., may be substituted. The liquid must have a low evaporation rate and narrow boiling range, and the same procedure shall be followed as with kerosine. Water is not a preferred liquid because of the possibility of frothing.

5. Hazards

5.1 Before a desiccator is used for the first time, wrap it in a towel and test under an absolute pressure of under 3 mm. Exercise care in handling the desiccator when under vacuum,

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.

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² *Annual Book of ASTM Standards*, Vols 11.01 and 06.03.

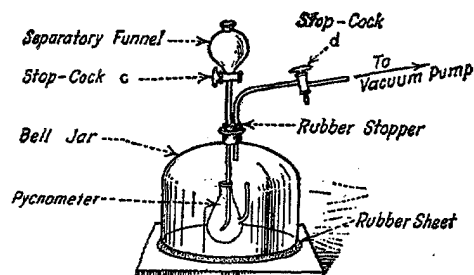


FIG. 1 Apparatus for Test Method B

since a sudden jar may cause it to collapse.

6. Standardization of Pycnometer

6.1 Fill the pycnometer with freshly boiled water at 23 to 24°C, gradually bring to $25 \pm 0.5^\circ\text{C}$, and then dry and weigh as specified in 7.6. Empty the pycnometer, and clean, dry, and reweigh it. Next fill the pycnometer with kerosine at 23 to 24°C, bring to $25 \pm 0.5^\circ\text{C}$, dry, and weigh as before. Calculate the specific gravity, S , of the kerosine at 25/25°C as follows:

$$S = A/B$$

where:

A = weight of kerosine, g, and

B = weight of water, g

7. Procedure

7.1 *Drying*—Dry the pigment, preferably in an electric oven, at $105 \pm 2^\circ\text{C}$ for 2 h.

7.2 *Weighing*—Transfer to a clean, dry, weighed pycnometer, sufficient sample to form a layer approximately $\frac{3}{4}$ in. (20 mm) deep. For black, blue, and lake pigments of low specific gravity, use about 1 g of sample; for inert

crystalline pigments, about 4 g; for opaque white pigments, 7 to 10 g; and for red lead, from 15 to 20 g. Weigh pigments of a hygroscopic nature from the weighing bottle.

7.3 *Number of Specimens*—Run all samples at least in duplicate.

7.4 *Addition of Kerosine*—Add enough kerosine to the pycnometer to form a clear layer approximately $\frac{1}{4}$ in. (6 mm) above the pigment. When necessary, stir the specimen with a polished round-bottom glass rod until completely covered by kerosine, adding more kerosine if necessary. Wash the rod with kerosine, adding the washings to the pycnometer.

7.5 *Removal of Occluded Air*—Place the pycnometer in the desiccator. Close the desiccator and attach to the water pump until the greater part of the air is removed from the system. Complete this procedure within a period of 5 to 10 min. Close the system with a pinchcock and attach the desiccator to the oil pump for the removal of the small amounts of air given off at the low pressures obtainable with the oil pump. Use the manometer to indicate whether the oil pump is giving the proper vacuum. When the manometer indicates that the absolute pressure is 3 mm and constant, cut off the oil pump for short periods, taking care that the vacuum does not change materially due to leakage. At first bubbles of air rise from the pigments very rapidly, then this action gradually decreases and finally stops. The time required for complete removal of air may vary from 30 min to 24 h, depending upon the nature of the pigment. When no more bubbles can be seen, it may be assumed that the occluded air has been removed and that the pigment is thoroughly wet with kerosine. Then slowly admit air to the desiccator by means of the pinchcock.

7.6 *Filling and Bringing to Temperature*—Remove the pycnometer from the desiccator, fill with kerosine at 24 to 25°C taking care to add a sufficient quantity to prevent air bubbles where the pycnometer is closed, and permit to come

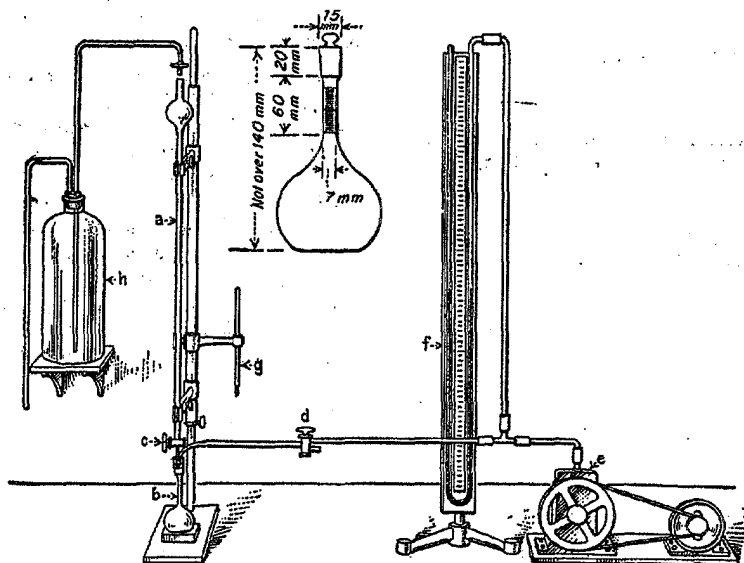
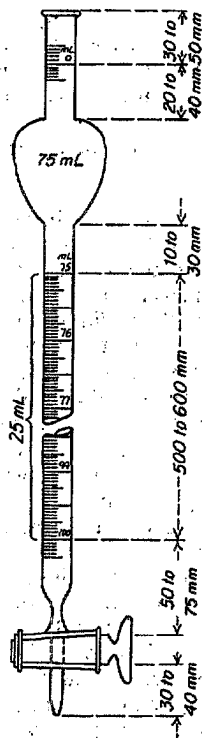


FIG. 2 Apparatus for Test Method C



Buret: Geissler, straight; glass stopcock, ground accurately.

Total capacity	100 mL
Capacity of bulb	0 to 75 mL
Graduated	75 to 100 mL
Subdivisions	0.05 mL
Rate of outflow	about 2 min
Permissible variations:	
Capacity, total	± 0.10 mL
Capacity graduated portion	± 0.03 mL

Markings on graduations shall be in conformity with the National Bureau of Standards Circular No. 9.³

FIG. 3 Buret in Apparatus

to constant temperature at $25 \pm 0.5^\circ\text{C}$ in the water bath. Carefully stopper the pycnometer and remove excess kerosine with lens paper. Take the pycnometer out of the bath, allow to come to room temperature, and weigh.

8. Calculation

8.1 Calculate the specific gravity, S , of the pigment as follows:

$$S = \frac{P_1}{W - \frac{K_1}{D}}$$

where:

P_1 = weight of pigment used, g,

W = weight of water to fill the pycnometer, g,

K_1 = weight of kerosine added to the pigment, g, and

D = specific gravity of the kerosine.

³ Available from National Institute of Standards and Technology, Gaithersburg, MD 20899.

9. Precision

9.1 Duplicate determinations by this test method should not differ by more than 0.02.

TEST METHOD B—FOR TESTS REQUIRING GREATER ACCURACY THAN TEST METHOD A

10. Apparatus (see Figs. 1 and 2)

10.1 *Pycnometer, Water Bath, Manometer, Vacuum Pump, Thermometer, Weighing Bottle, and Immersion Liquid*—See Section 4; also Fig. 2 (e) and (f).

10.2 *Bell Jar*, glass, with a two-hole rubber stopper. Into one hole of the stopper shall be fitted a separatory funnel with a well-ground stopcock (Fig. 1 (c)), extending into the pycnometer. Into the other hole of the stopper shall be fitted a glass tube with a well-ground three-way stopcock (Fig. 2 (d)) and connected with the vacuum pump (Fig. 2 (e)). The bell jar shall rest on a sheet of rubber, cemented or vulcanized to a glass or iron plate. With stopcock c closed and stopcock d open to the pump, the system shall maintain an absolute pressure of at most 3 mm. A desiccator may be used instead of a bell jar.

10.3 *Bottle*, storage, (Fig. 2 (h)) for kerosine or other wetting liquid.

11. Hazards

11.1 Before a bell jar (or desiccator) is used for the first time, test under a vacuum as described in Section 5.

11.2 Use a buret stopcock (Fig. 2 (c)) that is well ground and lubricated with silicone lubricants or use a PTFE-coated stopcock.

12. Procedure

12.1 Place the pycnometer containing the weighed, dried pigment under the bell jar. Close stopcocks c and d , start the vacuum pump, and then gradually open stopcock d to the pump. When an absolute pressure of 3 mm has been attained and can be maintained, fill the separatory funnel with kerosine, close stopcock d , and gradually open stopcock c , adding sufficient kerosine to cover the pigment. Then stop the pump and release the suction at stopcock d . Finally, fill the pycnometer with kerosine, and complete the test as described in 7.6 and Section 8, under Test Method A.

13. Precision

13.1 Duplicate determinations by this test method should not differ by more than 0.01.

TEST METHOD C—FOR RAPID AND ACCURATE TESTING OF A SINGLE SPECIMEN

14. Apparatus (see Figs. 2 and 3)

14.1 *Buret*, 100-mL, with a 75-mL bulb in the upper part, and with the lower part (25 mL) graduated in 0.05-mL divisions (see Fig. 3).

14.2 *Flask*—A special 100-mL graduated flask (Fig. 2 (b)) with ground-glass stopper. The flask shall be thick enough to withstand an absolute pressure of 1 mm, and shall weigh between 50 and 60 g. The neck of the flask shall be graduated in 0.05-mL divisions between the 99 and 100-mL marks. The dimensions of the flask shall be as shown in Fig. 2.

14.3 *Stopcocks*—A tightly ground stopcock (Fig. 2 (c)) as

part of buret, *a*, and a three-way stopcock (Fig. 2 (*d*)) connecting with the vacuum pump, *e*. To prevent leakage of kerosine use a buret stopcock (Fig. 2 (*c*)) that is well ground and lubricated with silicone lubricant or use a PTFE-coated stopcock.

14.4 *Vacuum Pump*—See 4.5; also Fig. 2 (*e*). In this procedure the oil vacuum pump shall be capable of reducing the absolute pressure of the system to 1 mm.

14.5 *Manometer, Thermometer, Weighing Bottle, and Immersion Liquid*—See Section 4; also Fig. 2 (*f*).

14.6 *Bottle*—See 10.3.

15. Hazards

15.1 The variations that occur under normal conditions in a room do not materially affect the specific gravity of a pigment. However, take care that the temperature of the liquid after transferring to the flask is approximately the same as it was when in the buret.

15.2 Since in determining both K_2 and V the tip of the buret and bore of the stopcock plug are empty, no correction is to be made; but stopcock *c* must be so well ground that under an absolute pressure of 1 mm for 30 min no leakage of kerosine shall take place. The usual sources of error are failure to remove all the air from the pigment, and leaks in the system. Use a minimum amount of rubber tubing in the system and, wherever it is used, coat the joints between rubber and glass with a melted mixture of beeswax and rosin.

15.3 In cleaning the flask of kerosine only, a rinsing two or three times with ether, followed by dry air (dried over sulfuric acid and calcium chloride), is considered sufficient. When pigment is also present, remove both pigment and kerosine and follow with ether rinses until no more pigment remains. Add some filter pulp (macerated filter paper) and water (with or without glass beads), and shake vigorously. Repeat if necessary. Rinse the flask with reagent water, and either dry in an oven, or rinse with alcohol and ether followed by dry air.

16. Standardization of Apparatus

16.1 Connect the flask to the buret and the pump by means of a two-holed rubber stopper. Evacuate the system with the buret stopcock (Fig. 2 (*c*)) closed until the pump maintains an absolute pressure of 1 mm in the flask. Close the three-way stopcock, *d*, for 30 s, and again open to the pump. There shall be no appreciable change in the mercury levels in the manometer, indicating that the system beyond stopcock *d* is tight. With the vacuum still maintained, fill the buret from the top with kerosine, adjusting the level to the

zero mark with a piece of capillary tubing. Now close stopcock *d*, and carefully open stopcock *c*, admitting about 75 mL of kerosine into the flask. Open stopcock *d* to the air, thus releasing the vacuum in the flask, and fill the flask with kerosine to a definite mark on the neck. Read the buret, calling this reading K_2 (the volume of the flask).

17. Procedure

17.1 Clean the flask dry, and weigh. Transfer a quantity of the dry pigment to be tested to the flask by means of a clean, dry, glass funnel with the stem reaching to the bottom of the bulb. A piece of stiff nickel wire is convenient to push the powder down the stem. Nearly fill the bulb of the flask with the pigment, which, however, shall occupy a volume of less than 25 mL after all air is expelled. Greater accuracy may be obtained with a large specimen than with a small one. Wipe the inside stem as well as the entire outside of the flask with a clean piece of dry, lintless cloth. Weigh the flask and pigment, and calculate the weight of pigment by deducting the weight of the empty flask. With the buret clean and dry, attach the flask to the evacuating system as shown in Fig. 2. After closing stopcocks *c* and *d*, start the pump and carefully open stopcock *d* to the pump. Continue evacuation until the pump maintains an absolute pressure of 1 mm in the flask, or until all the air is removed from the system. Then fill the buret from the top as described in Section 16, close stopcock *d*, gradually open stopcock *c*, and add kerosine until the pigment is covered. Tap the flask gently to dislodge any air bubbles. Stop the pump; open stopcock *d* to the air, and fill the flask up to the same mark as was obtained in determining its volume. Designate the volume of kerosine required as V . Read the height of the liquid in the buret to the nearest estimated 0.01 mL.

18. Calculation

18.1 Calculate the specific gravity, SG , of the pigment as follows:

$$SG = P_2 / (K_2 - V)$$

where:

P_2 = weight of pigment used, g,

K_2 = volume of kerosine required to fill the flask when empty, mL, and

V = volume of kerosine required to fill the flask when the pigment is present, mL.

19. Precision

19.1 Duplicate determinations by this test method should not differ by more than 0.01.

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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 (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 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.

^{ε1} 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.

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 Specification for Dry Bleached Lac¹

This standard is issued under the fixed designation D 207; the number immediately following the designation indicates the year of original adoption 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 two grades of dry bleached lac, namely, regular and refined.

NOTE—Dry bleached lac is also known as “bone-dry,” “bac-dry,” or “kiln-dry” bleached lac. The refined grade is bleached lac from which practically all the wax and insoluble matter has been removed during the process of manufacture.

2. Referenced Document

2.1 *ASTM Standard:*
D 29 Test Methods for Sampling and Testing Lac Resins²

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 D01.33 on Varnish and Resins, Including Shellac.

Current edition approved Sept. 12, 1955. Published November 1955. Originally published as D 207 - 25. Last previous edition D 207 - 49.

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

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3. Properties

3.1 Dry bleached lac shall conform to the following requirements:

	Regular	Refined
Matter insoluble in specified hot solvents, max, %	1.0	0.2
Moisture, max, %	6.0	6.0
Wax, max, %	5.5	0.2
Matter soluble in water, max, %	1.0	0.3
Ash, max, %	1.0	0.5
Rosin	none	none
Copals	none	none

4. Test Methods

4.1 The material shall be sampled and the properties enumerated in this specification shall be determined in accordance with Test Methods D 29.



Standard Specification for Lampblack Pigment¹

This standard is issued under the fixed designation D 209; the number immediately following the designation indicates the year of original adoption 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 pigment commercially known as lamp-black. The pigment may be purchased in the dry form or as a paste in oil.

2. 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 Muller³

D 1208 Test Methods for Common Properties of Certain Pigments³

3. Composition and Properties

3.1 *Dry Pigment*—The pigment shall be made by burning oils or tars in such a manner as to form a deposit of carbon or soot. It shall be high grade in every respect and shall be free from oil, greasy matter, and admixture of any other substance. The pigment shall conform to the following requirements:

Ash, max, %	0.5
Acetone extract, max, %	1.0
Moisture and other volatile matter, max, %	3.0
Coarse particles, (total residue retained on a 45- μ m (No. 325) sieve), max, %	0.5
Tone when diluted with zinc oxide	clear-blue-gray

3.2 *Paste in Oil*—The paste in oil shall be made by

thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min, %	25
Linseed oil, max, %	75
Moisture and other volatile matter, max, %	0.7
Coarse particles and skins (total residue retained on a 45- μ m (No. 325) sieve), max, % of the dry pigment	1.0

3.3 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4540 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.2 *Coarse Particles*—Test Methods D 185.

5.3 *Pigment, Linseed Oil, and Moisture and Other Volatile Matter in Paste in Oil*—Test Methods D 1208.

5.4 *Mass Color and Tinting Strength*—Test Method D 387.

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 D01.31 on Pigment Specifications.

Current edition approved Aug. 31, 1981. Published October 1981. Originally published as D 209 - 24. Last previous edition D 209 - 47 (1974).

² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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Standard Specification for Bone Black Pigment¹

This standard is issued under the fixed designation D 210; the number immediately following the designation indicates the year of original adoption 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 specification covers the pigment commercially known as bone black, ivory black, or drop black. The pigment may be purchased in the dry form or as a paste in oil.

2. 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 Muller³
- D 1208 Test Methods for Common Properties of Certain Pigments³

3. Composition and Properties

3.1 *Dry Pigment*—The pigment shall be made by the calcination of bones, shall be unmixed with any other substance, and shall conform to the following requirements:

Ash (pigment dried at 105°C), %, max	88
Ash insoluble in acids, max, %	3.0
Acetone extract, max, %	2.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	2.0

3.2 *Paste in Oil*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. As received it shall not be caked in the container and shall

break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min, %	45
Linseed oil, max, %	55
Coarse particles and skins (total residue retained on a No. 325 (45- μ m) sieve), max, % of the dry pigment	2.5

3.3 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.2 *Coarse Particles*—Test Methods D 185.

5.3 *Pigment and Linseed Oil in Paste in Oil*—Test Methods D 1208.

5.4 *Mass Color and Tinting Strength*—Test Method D 387.

6. Keywords

6.1 Black pigment; drop black; ivory black; pigment.

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 D01.31 on Pigment Specifications.

Current edition approved Aug. 28, 1981. Published October 1981. Originally published as D 210 - 24. Last previous edition D 210 - 81.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

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Standard Specification for Chrome Yellow and Chrome Orange Pigments¹

This standard is issued under the fixed designation D 211; the number immediately following the designation indicates the year of original adoption 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—Editorial changes were made throughout in March 1989.

Scope

1.1 This specification covers six types of commercially pure lead chromate pigments as follows:

- Type I—Primrose Chrome Yellow,
- Type II—Lemon Chrome Yellow,
- Type III—Medium Chrome Yellow,
- Type IV—Light Chrome Orange,
- Type V—Dark Chrome Orange, and
- Type VI—Chrome Yellow for Green.

1.2 The following hazard caveat applies to the test method portion of this specification only. *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 126 Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)⁴
- D 387 Test Method for Color and Strength of Color Pigments With a Mechanical Muller²
- D 523 Test Method for Specular Gloss⁵
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer³
- D 600 Specification for Liquid Paint Driers⁴
- 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 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems⁵

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 D01.31 on Pigment Specifications.

Current edition approved Sept. 8, 1967. Originally published as D 211 - 25T. Last previous edition D 211 - 63 T.

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.

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens for Broad-Band Filter Reflectometry⁵

2.2 Federal Specification⁶

TT-R-266 Resin, Alkyd; Solutions

3. Composition and Properties

3.1 The pigments shall be chemical precipitates consisting of normal or basic lead chromates, or mixtures of these, with or without admixtures of other insoluble compounds of lead or other materials used in manufacture to control certain properties. The pigments shall conform to the requirements for composition as prescribed in Table 1.

3.2 The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

3.3 When mutually agreed upon between the purchaser and the seller as being essential to the end use of the pigment, resistance to loss of gloss, chalking, and color change shall be tested as specified in 5.4. The exposed panel shall show no chalking, a loss of not more than 30 % of the original gloss, and a color change no greater than the lightness difference, ΔL , shown for each pigment type in Table 2.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4450 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or samples from the same production unit may be blended in equal quantities to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following test methods. Test procedures not covered by ASTM methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Chemical Analysis of Dry Pigment*—Test Methods D 126. The lead chromate analysis should be conducted in accordance with 11.1 of Test Methods D 126; the procedure

⁶ Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

TABLE 1 Requirements for Composition

	Type I	Type II	Type III	Type IV	Type V	Type VI
Lead chromate (PbCrO ₄), min %	50	65	87	55	55	75
Total matter soluble in water, max %	1.0	1.0	1.0	1.0	1.0	1.0
Total of all substances other than insoluble compounds of lead, max %	8.0	10.0	10.0	10.0	3.0	10.0
Moisture and other volatile matter, max %	1.5	1.5	1.5	1.5	1.5	1.5
Coarse particles (total residue retained on a 45-μm (No. 325) sieve), max %	1.0	1.0	1.0	1.0	1.0	1.0
Organic colors and lakes	none	none	none	none	none	none

TABLE 2 Permissible Lightness Difference (ΔL)

Type	Pigment	ΔL
I	Primrose chrome yellow	35
II	Lemon chrome yellow	8
III	Medium chrome yellow	6
IV	Light chrome orange	3
V	Dark chrome orange	3
VI	Chrome yellow for green	6

outlined in 11.2 is not applicable.

5.1.2 *Coarse Particles*—Test Methods D 185.

5.1.3 *Mass Color and Tinting Strength*—Test Method D 387.

5.1.4 *Resistance to Loss of Gloss, Chalking, and Color Change*:

5.1.4.1 A test enamel shall be prepared consisting of ingredients conforming to the applicable specifications in the following proportions:

Ingredient	Weight Percent
Pigment under test	30 to 34
Alkyd resin vehicle solids, min ^A	28
Total solids, min	60
Mineral spirits	^{B, C}
Driers	^{D, E}

^A An alkyd resin solution conforming to Type III of Federal Specification TT-R-266 is suggested.

^B See Specification D 235.

^C As required to give a Stormer consistency of 100 to 150 g at 200 r/min as prescribed in Test Method D 562.

^D See Class B in Specification D 600.

^E As required to obtain set-to-touch in 2 h and dry-hard in 8 h.

5.1.4.2 The enamel shall have a fineness of grind, as prescribed in Test Method D 1210, of 1.5 mils or less. Apply the enamel to duplicate flat metal panels by spray or applicator to complete hiding and allow to dry 72 h. Measure the gloss at 60° in accordance with Test Method D 523. Measure the directional reflectance of the coating in accordance with Test Method E 97. Subject the coated panels for 168 h to accelerated weathering under the conditions prescribed in Practice D 822. Examine the exposed coating for chalking. Wash the exposed panel under running water with a thoroughly degreased lamb's wool pad to remove scum or dirt. Wipe off water with clean cheesecloth and dry the panel for 2 h. Calculate the loss of gloss from gloss measurements made before and after exposure. After exposure, determine the directional reflectance for each panel as described in Test Method E 97. Calculate the color change or lightness-difference estimate (ΔL) as follows:

$$\Delta L = K(Y_2^{1/2} - Y_1^{1/2})$$

where:

Y_1 = luminous directional reflectance of the panel measured before exposure,

Y_2 = reflectance measured after exposure, and

K = 100 when the reflectances are expressed as decimal fractions or 10 if reflectances are expressed in percent.

The color change for the coating shall be the average obtained for the two panels.⁷

⁷ The method of determining the lightness-difference estimate is described in detail in example 4, "Photoelectric Tristimulus Colorimetry with Three Filters," Circular C429, Nat. Bureau Standards.

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Standard Specification for Chrome Green Pigment¹

This standard is issued under the fixed designation D 212; the number immediately following the designation indicates the year of original adoption 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 specification has been approved for use by agencies of the Department of Defense to replace Fed. Spec. TT-P-345. 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 covers the pigment commercially known as chrome green. The pigment may be purchased in the dry form or as a paste in oil.

2. Referenced Documents

2.1 ASTM Standards:

- D 126 Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints^{2,3}
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 *Dry Pigment*—The pigment shall be a precipitated mixture of lead chromate and iron blue (potassium or ammonium ferri ferrocyanoide) with or without other insoluble compounds of lead. It shall be free from admixture of any substances other than those added during manufacture, in small amounts up to a total of 7 %, to improve the stability or working properties, or both, of the pigment. Extenders and diluents such as barium sulfate, silica, silicates, calcium carbonate, calcium sulfate, magnesium carbonate, etc., shall be absent. The pigment shall conform to the following requirements:

Of the total lead ⁴ in the pigment, percent present as chromate (PbCrO_4), min	70
Total matter soluble in water, max, %	1.0
Moisture and other volatile matter, max, %	4.0

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 D01.31 on Pigment Specifications.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 212 - 47 (1974). Last previous edition D 212 - 80.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

Coarse particles (total residue retained on a No. 325 (45- μm) sieve), max, %	1.0
Organic colors or lakes	none

⁴ Calculated as metallic lead.

3.2 *Paste in Oil*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil. The paste as received shall not be caked in the container and shall break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min, %	70
Linseed oil, max, %	30
Moisture and other volatile matter, max, %	3.0
Coarse particles and skins (total residue retained on a No. 325 (45- μm) sieve), max, percent of the dry pigment	1.5

3.3 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb—two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Chemical Analysis of Dry Pigment*—Test Methods D 126.

5.1.2 *Coarse Particles*—Test Methods D 185.

5.1.3 *Moisture*—Test Methods D 280.

5.1.4 *Other Volatile Matter in Paste in Oil*—Test Methods D 1208.

5.1.5 *Mass Color and Tinting Strength*—Test Method D 387.

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Standard Specification for Orange Shellac and Other Lacs¹

This standard is issued under the fixed designation D 237; the number immediately following the designation indicates the year of original adoption 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 January 1991.

1. Scope

1.1 This specification covers three types and four grades of orange shellac and other lac, as follows:

1.1.1 *Type I*, orange shallac, grades A, B, C, and D,

1.1.2 *Type II*, button lac, and

1.1.3 *Type III*, garnet lac.

1.2 Stick-lac and seed-lac are not covered by this specification.

2. Referenced Document

2.1 ASTM Standard:

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 D01.33 on Polymers and Resins.

Current edition approved Sept. 30, 1957. Published November 1957. Originally published as D 237 - 26. Last previous edition D 237 - 55.

D 29 Test Methods for Sampling and Testing Lac Resins²

3. Physical Properties

3.1 The material shall conform to the requirements prescribed in Table 1.

NOTE—Type II customarily occurs in the form of circular disks about 3 in. (76 mm) in diameter and 1/8 in. (3.2 mm) in thickness and is known to the trade as pure button lac. Type III is dark garnet in color and is known to the trade as pure garnet lac. Garnet lac also is manufactured in admixture with rosin and as a dewaxed lac, but these are not covered by this specification.

4. Test Methods

4.1 The material shall be sampled and the properties enumerated in this specification shall be determined in accordance with Test Methods D 29.

5. Keywords

5.1 lac; orange shellac; shellac

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

TABLE 1 Requirements for Orange Shellac and Other Lacs

	Type I				Type II	Type III
	Grade A	Grade B	Grade C	Grade D	...	...
Matter insoluble in specified hot solvents, max, %	1.00	1.25	1.50	3.00	1.00	0.50
Iodine number, max	15.0	15.0	15.0	15.0	15.0	18.0
Moisture, max, %	2.0	2.0	2.0	2.0	2.0	2.0
Wax, max, %	5.5	5.5	5.5	5.5	5.5	3.5
Orpiment, max, %	0.03	0.03	0.03	0.2	0.03	0.03
Matter soluble in water, max, %	0.5	0.5	0.5	0.5	0.5	0.5
Ash, max, %	1.0	1.0	1.0	1.0	0.5	0.5
Rosin	none	none	none	none	none	none
Color	when specified, the color shall be no darker than that of a sample agreed upon between the seller and the purchaser ⁴					

⁴ Attention is called to the fact that the seller and the purchaser must agree upon one of the two methods for determining color appearing in Test Methods D 29.

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Standard Specification for Iron Blue Pigment¹

This standard is issued under the fixed designation D 261; the number immediately following the designation indicates the year of original adoption 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 4.1 was editorially changed in May 1987.

1. Scope

1.1 This specification covers the dry pigment commercially known as Prussian blue, Chinese blue, Milori blue, or iron blue.

2. Referenced Documents

2.1 ASTM Standards:

- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1135 Test Methods for Chemical Analysis of Blue Pigments²

3. Composition and Properties

3.1 The pigment shall be the blue product formed by the reaction of a solution of iron salts with a ferrocyanide or ferricyanide solution. It shall be free from admixture of any substances other than those added during manufacture, in small amounts up to a total of 5 %, for the purpose of improving the quality or working properties, or both, of the pigment. Extenders, and diluents such as barium sulfate, silica, silicates, calcium carbonate, calcium sulfate, magnesium carbonate, etc., shall be absent. The pigment shall conform to the following requirements:

Total matter soluble in water, max, %	1.0
Moisture, max, %	8.0
Organic colors or lakes	none
Acidity or alkalinity—the acidity or alkalinity of the aqueous extract, with methyl orange indicator, shall not exceed the chemical equivalent of 0.1 % of sulfuric acid, calculated on the dry pigment.	

3.2 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

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 D 01.31 on Pigment Specifications.

Current edition approved Oct. 31, 1975. Published December 1975. Originally published as D 261 - 27 T. Last previous edition D 261 - 47 (1970).

² Annual Book of ASTM Standards, Vol 06.02.

NOTE—The physical properties and tests of iron blue, particularly the tinting strength, are considered a better measure of value than the percentages of chemical constituents.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit or production in a shipment. When no markings distinguishing between units or production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SWI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Identification*—Test Methods D 1135.

5.3 *Extenders and Diluents*—Test Methods D 1135.

5.4 *Total Matter Soluble in Water*—Matter soluble in water shall be determined by either of the following methods, as mutually agreed upon between the purchaser and the seller:

5.4.1 *Water-Soluble Matter by Extraction*—Test Methods D 1135.

5.4.2 *Water-Soluble Salts by Electrical Conductivity*—Test Methods D 1135.

5.5 *Moisture in Dry Pigment*—Moisture shall be determined by either of the following methods, as mutually agreed upon between the purchaser and the seller:

5.5.1 *Brabender Moisture Tester*—Test Methods D 1135.

5.5.2 *Toluene Distillation*—Test Methods D 1135.

5.6 *Organic Colors or Lakes*—Test Methods D 1135.

5.7 *Mass Color and Tinting Strength*—Test Method D 387.

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Standard Specification for Ultramarine Blue Pigment¹

This standard is issued under the fixed designation D 262; the number immediately following the designation indicates the year of original adoption 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—Paragraph 4.1 was editorially changed in May 1987.

1. Scope

1.1 This specification covers the dry pigment commercially known as ultramarine blue, suitable for use in paints, enamels, lacquers, and similar products.

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller³
- D 1135 Test Methods for Chemical Analysis of Blue Pigments³
- D 1208 Test Methods for Common Properties of Certain Pigments³

3. Composition and Properties

3.1 The pigment shall be a manufactured product obtained by the calcining of mixtures of clays and silicas with sodium salts, sulfur, and carbonaceous material. It shall be a soft, dry, finely ground powder of good blue color, free of admixtures of color substances, and shall conform to the following requirements:

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 D 01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

Matter soluble in water, max, %	1.5
Moisture and other volatile matter, max, %	4.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	1.0
Organic colors or lakes	none

3.2 The mass, color and character of the tint and the tinting strength formed by mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following methods. Test procedures not covered by ASTM methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Identification*—Test Methods D 1135.

5.3 *Matter Soluble in Water*—Test Methods D 1135.

5.4 *Moisture and Other Volatile Matter in Dry Pigment*—Method A of Test Methods D 280 or Test Method D 1208.

5.5 *Coarse Particles*—Test Methods D 185.

5.6 *Organic Colors or Lakes*—Section 18 of Test Methods D 1135.

5.7 *Mass Color and Tinting Strength*—Test Method D 387.

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Standard Specification for Chrome Oxide Green Pigment¹

This standard is issued under the fixed designation D 263; the number immediately following the designation indicates the year of original adoption 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 dry pigment commercially known as chrome oxide green.

2. Referenced Documents

2.1 ASTM Standards:

- D 126 Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²

3. Composition and Properties

3.1 The pigment shall consist of practically pure chromium sesqui-oxide (Cr_2O_3) without any admixture, and shall conform to the following requirements:

Total chromium (calculated as Cr_2O_3), min, %	97
Total matter soluble in water, max, %	0.5

Moisture and other volatile matter, max, %	0.5
Coarse particles (total residue retained on a No. 325 (45- μm) sieve), max, %	2.0
Organic colors or lakes	none

3.2 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4540 kg) except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following methods where applicable. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Chromium Content*—Test Methods D 126.

5.3 *Total Matter Soluble in Water*—Test Methods D 126.

5.4 *Moisture Content*—Test Method D 280.

5.5 *Coarse Particles*—Test Methods D 185.

5.6 *Organic Colors and Lakes*—Test Methods D 126.

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 D01.31 on Pigment Specifications.

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

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

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Standard Specification for Gold Bronze Powder¹

This standard is issued under the fixed designation D 267; the number immediately following the designation indicates the year of original adoption 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 June 1987.

Scope

1.1 This specification covers the materials commercially known as gold bronze, pale gold bronze, and rich gold bronze powders.

2. Referenced Documents

2.1 ASTM Standards:

D 13 Specification for Spirits of Turpentine²

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³

D 480 Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes⁴

2.2 U.S. Federal Specification:

TT-V-121h Water-Resisting Spar Varnish⁵

3. Composition and Properties

3.1 The bronze powder shall be made from new ingot metals. It shall consist of fine polished flakes containing not less than 3 % of fatty or oily matter (polishing lubricant) to give good "leafing" properties.

3.2 The residue retained on a No. 100 (150- μ m) sieve, using alcohol as the wash liquid, shall not exceed 0.2 % (in accordance with Test Methods D 185).

3.3 The powder shall have good "leafing" properties. (By "leafing" is understood the property of forming an appar-

ently continuous brilliant film over the entire free surface of a mixture of the powder in a suitable liquid (Note), within 1 min after cessation of stirring the mixture.) In testing for leafing properties the powder shall be mixed in the proportion of 3 to 4 lb (370 to 475 g/L) to a gallon (3.8 L) of the liquid. As thus mixed it shall also give a free flowing, smooth, continuous coating in accordance with Test Method D 480.

NOTE—A suitable liquid is made by mixing spar varnish conforming to the U. S. Federal Specification for Water-Resisting Spar Varnish (No. TT-V-121 h) with turpentine conforming to Specification D 13 in such proportions that the mixture will have a viscosity of 0.65 to 0.85 poises (B to C on the Gardner-Holdt scale).

3.4 The gold bronze powder shall be suitable for making gold bronze paint. It shall match in shade and fineness a reference sample mutually agreed upon by the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Test shall be conducted in accordance with the appropriate methods of the American Society for Testing and Materials, where applicable. Test procedures not covered by ASTM methods shall be mutually agreed upon between the purchaser and the seller.

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 D01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vol 06.03.

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

⁴ Annual Book of ASTM Standards, Vol 06.02.

⁵ Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

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Standard Test Methods for Bleeding of Pigments¹

This standard is issued under the fixed designation D 279; the number immediately following the designation indicates the year of original adoption 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 4571.1 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.

^{ε1} NOTE—Keywords were added editorially in June 1991.

1. Scope

1.1 These test methods cover procedures for determining the bleeding characteristics of dry pigments by direct solvent extraction of the pigment and by overstripping a film with a white coating and observing for the color migration from the base coat containing the pigment.

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. Summary of Test Methods

2.1 *Test Method A*—The pigment is shaken with toluene, filtered, and the filtrate observed for color.

2.2 *Test Method B*—A coating using the pigment under test is prepared and applied to one half of a panel. A white finish is applied over the dried colored coating and extended to the uncoated portion of the panel. The dried overstripe coating is examined for color migration from the base coat.

3. Significance and Use

3.1 Test Method A determines the amount of color produced when the pigment is in direct contact with a selected solvent such as toluene. It is useful as a rapid, easily conducted test of the general bleeding characteristics of pigments.

3.2 Test Method B determines the amount of color migration into a white film applied over a base coat containing the pigment. It may give a more practical evaluation of whether a pigment will meet specific requirements for bleed resistance.

3.3 Both Test Method A and Test Method B measure the extent of bleed.

TEST METHOD A, DIRECT SOLVENT CONTACT WITH PIGMENT

4. Procedure

4.1 Place 0.50 g of pigment (Note 1) in a 25-mL test tube

and add 20-mL of reagent grade toluene (Note 2). Close with a stopper, shake well for 10 s, and let stand 15 min. Repeat the 10-s shake and let stand 45 min.

NOTE 1—Additional amounts of pigment may be used by prior agreement for denser inorganic pigments. A maximum of 1.5 g is recommended for even the dense lead chromate pigments.

NOTE 2—Other solvents may be used, by mutual agreement between the parties involved, in place of toluene in a specific pigment bleed evaluation.

4.2 Filter through a glass funnel using double filter paper that has first been wet with toluene and collect approximately 10 mL of filtrate. If the filtrate is cloudy, refilter to get a clear filtrate.

NOTE 3—In the case of colloidal particles, it may be necessary to centrifuge the filtrate.

4.3 Hold the test tube containing the filtrate above a white background and look down through the filtrate for coloration caused by pigment bleed. Describe the degree or severity of bleed according to the following terminology:

4.3.1 *None*—No perceptible color (that is, no bleed).

4.3.2 *Slight*—A faint but distinct coloration.

4.3.3 *Moderate*—A pronounced but not severe coloration.

4.3.4 *Severe*—An intense coloration.

TEST METHOD B, WHITE OVERSTRIPE OF A PIGMENTED FILM

5. Procedure

5.1 Prepare a coating of agreed upon composition, using the pigment under test, and prepare a film of this coating by any method that will give a smooth and uniform film of normal coating thickness (that is, in the range from 2 to 3 mils) (50 to 75 μm) leaving a portion of the substrate uncoated. Cure the film under conditions appropriate to the end use. For air dry films, dry at room temperature, approximately 25°C (77°F) for a minimum of 24 h.

5.2 Pretreatments of the colored coating, such as sanding or solvent wipe, or both, may be used before the overstripe, as agreed upon by the purchaser and seller.

5.3 Overstripe with a white finish of agreed upon composition. Ensure that the overstripe film is of uniform thickness and applied at complete hiding; otherwise, color show-through may be erroneously interpreted as bleed.

5.4 Extend the overstripe film to the uncoated portion of the panel. Dry the overstripe under conditions mutually

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.

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upon by the involved parties.
Examine for color migration from the base coat into white topcoat. Within 2 h after drying rate the extent of bleed in accordance with the scale in Test Method A (4.3).

Precision and Bias

The precision and bias are considered to be undeter-

minable as they will vary with the solvent or solvent blends used in Test Method A and with the type of base coat and type of overstripping used in Test Method B.

7. Keywords

7.1 bleeding pigments; pigment bleeding test

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Standard Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments¹

This standard is issued under the fixed designation D 280; the number immediately following the designation indicates the year of original adoption 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 Method 4071 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 These methods cover procedures for determining hygroscopic moisture (and other matter volatile under the test conditions) in pigments.

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.*

METHOD A—FOR PIGMENTS THAT DO NOT DECOMPOSE AT 110°C

2. Apparatus

2.1 *Weighing Bottle*, wide-mouth, cylindrical, glass (flat form, about 30 mm in height and about 70 mm in diameter), provided with a ground-in glass stopper. Or, an aluminum moisture dish (about 90 mm in diameter and about 50 mm in depth) with a tightly fitting cover.

2.2 *Oven* in which a temperature of from $110 \pm 2^\circ\text{C}$ is maintained.

2.3 *Analytical Balance*.

3. Procedure

3.1 Weigh accurately the glass weighing bottle and stopper or the aluminum moisture dish with cover. Place a specimen of from 3 to 5 g of the pigment in the clean, dry weighing bottle or in the clean, dry aluminum moisture dish. Insert the stopper (or cover) and weigh to 0.1 mg. Subtract the weight of the vessel from the total weight to obtain the weight of sample used in the test. Remove the stopper (or cover) and place it and the bottle (or dish) containing the specimen in an oven that has been previously heated to $110 \pm 2^\circ\text{C}$, heat for 2 h at a temperature of 105 to 110°C . Replace the stopper (or cover), cool in a desiccator, and weigh. Calculate the total loss in weight as percent of moisture and other volatile matter.

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 D01.21 on Chemical Analysis of Paints and Paint Material.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 280 - 28. Last previous edition D 280 - 75.

METHOD B—FOR PIGMENTS THAT DECOMPOSE AT 110°C

4. Apparatus

4.1 *Weighing Bottle*, glass, as described in 2.1.

4.2 *Open-Tube Manometer* made of glass tubing 6 mm in diameter, filled with mercury to approximately 860 mm, fitted with rubber pressure tubing attached to a T-joint leading to the desiccator and the pump. A suitable low-pressure gage may be used in place of the manometer.

NOTE 1—The difference in levels of the mercury in the manometer when the system is in operation, subtracted from the barometer reading taken at the same time, gives the pressure of the system in millimetres of mercury.

4.3 *Desiccator*, glass, having a hole at the side or in the cover, constructed with heavy walls to withstand a vacuum of one atmosphere. The hole at the side shall be closed with a one-hole rubber stopper carrying a glass tube with a rubber tube connection and a pinchcock or with a glass stopcock ground to fit the tubulature.

4.4 *Oil Vacuum Pump*,² able to achieve and hold a vacuum of 3 mm.

4.5 *Analytical Balance*.

5. Procedure

5.1 Weigh accurately the glass weighing bottle and stopper. Place a specimen of from 1 to 3 g of the pigment in the clean, dry weighing bottle, insert the stopper and weigh to 0.1 mg. Subtract the weight of the vessel from the total weight to obtain the weight of sample used in the test.

5.2 Remove the stopper and place it and the bottle containing the specimen in the desiccator containing *fresh*, anhydrous magnesium perchlorate.³ Close the desiccator, attach to the pump, and *gradually* evacuate until the pressure is constant at 3 mm or less (Note 2). Close the pinchcock or stopcock, stop the pump, and let stand at room temperature (70 to 90°F) for 24 h.

5.3 Slowly admit air that has been dried by passage through *fresh* magnesium perchlorate to the desiccator by means of the pinchcock or stopcock, remove the cover, quickly replace the stopper in the weighing bottle, and weigh. Repeat the evacuations and weighings until the loss in weight does not exceed 0.5 mg in 24 h. Calculate the total loss in

² The "Hyvac" oil pump is satisfactory.

³ Available from Chemical Suppliers under the name Anhydrous or Dehydrite.

weight as percent of moisture and other volatile matter (Note

NOTE 2—Caution should be used in evacuating glass desiccators. The vacuum should be applied gradually. Desiccators will maintain a vacuum for a greater length of time when a thin film of a suitable

stopcock lubricant is applied to the ground surfaces. Desiccators when evacuated should be wrapped in towels or covered by other means to prevent possible injury to the operator.

NOTE 3—This is an empirical method and the details should be strictly followed. The determination of the true hygroscopic moisture content of very finely divided pigments is very difficult, if not impossible in some cases.

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Standard Test Method for Oil Absorption of Pigments by Spatula Rub-out¹

This standard is issued under the fixed designation D 281; the number immediately following the designation indicates the year of original adoption 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 test method covers the determination of the oil absorption of pigments by the spatula rub-out technique.

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 234 Specification for Raw Linseed Oil²

D 1483 Test Method for Oil Absorption of Pigments by Gardner-Coleman Method³

3. Summary of Test Method

3.1 A stiff, putty-like paste is formed by the dropwise addition of linseed oil to pigment that is being thoroughly rubbed with a spatula. The amount of oil required to produce the end point is used to calculate an oil absorption value.

4. Significance and Use

4.1 The oil absorption value obtained by this test method gives information about the vehicle demand of the pigment when it is used in a pigment paste. Oil absorption values can be used to characterize pigments or batches of a given pigment.

4.2 This test method differs from Test Method D 1483 in that D 1483 involves only a gentle stirring and folding of the pigment, whereas this test method requires a thorough rubbing action. Because the end points are different, the values obtained from the two test methods generally differ.

5. Apparatus and Materials

5.1 *Balance*, capable of weighing to 0.01 g.

5.2 *Dropping Bottle*, fitted with ground-in pipet and rubber bulb or buret, graduated in 0.1-mL divisions.

5.3 *Smooth Glass Rub-up Plate or Marble Slab* (glass should have a surface similar to Hoover Muller Plates).

5.4 *Spatula*, sharp-edged, steel, having a blade of $\frac{1}{2}$ or $\frac{3}{4}$ by 4 in. (15 or 20 mm by 100 mm).

5.5 *Oil*, linseed, raw, conforming to Specification D 234 except that it shall have an acid number of 3 ± 1 . Linseed oil used in comparative tests must have the same acid value. Other liquids, such as refined oil, may be used by mutual agreement.

6. Procedure

6.1 *Procedure A (Weighing Bottle)*—Weigh exactly 1 g., or any multiple thereof (Note 1), of the thoroughly mixed and air dried pigment and place upon a glass plate or marble slab. Weigh to 0.1 g a dropping bottle containing raw linseed oil along with the pipet and rubber bulb. Add the linseed oil gradually, drop by drop (by means of the pipet), to the pigment. After the addition of each drop, thoroughly incorporate the oil by rubbing up with the spatula. The test is complete when exactly enough oil has been incorporated with the pigment to produce a very stiff, putty-like paste, that does not break or separate. Weigh the bottle and oil to 0.1 g and determine by difference the weight of oil used.

NOTE 1—The specimen weight depends upon the specific gravity, fineness, and other characteristics of the pigment. For example, 20 g is taken for white lead, but about 1 g is sufficient for carbon black. In any event, the specimen size should be large enough so that at least 1 g of oil is required.

6.2 Procedure B (Buret):

6.2.1 Follow Procedure A, except add the linseed oil from a buret rather than a dropping bottle pipet. Calculate the weight of oil in grams by multiplying the volume oil used by its density (0.93 g/mL).

6.3 It is suggested that when a new pigment is to be tested, a preliminary rub-out be made to determine an approximate end point. Once this is established, the actual determination should be made with a slower addition of oil and a more vigorous rub-out through the critical region, therefore permitting a more precise assessment of the correct oil absorption end point.

7. Calculation

7.1 From the weights of oil and pigment used in the test, calculate the number of pounds of oil required to exactly wet 100 pounds of pigment or grams of oil per 100 grams of pigment.

8. Report

8.1 Report the oil absorption of the pigment as the number of pounds (grams) of oil required to exactly wet 100 pounds (grams) of pigment.

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.

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² Annual Book of ASTM Standards, 06.03.

³ Annual Book of ASTM Standards, 06.02.

9. Precision

9.1 On the basis of an interlaboratory study of this test method in which one operator in each of five laboratories tested two grades of zinc oxide covering a small oil absorption range by spatula rub-out, the within-laboratory coefficient of variation was found to be 4.6 % at 10 df and the between-laboratories coefficient of variation 11.6 % at 8 df. Based on these coefficients, the following criteria should be used for judging the precision of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results obtained by a single operator should be suspect if they differ by more than 14.3 % relative.

9.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 38.0 % relative.

10. Keywords

10.1 oil absorption; pigments (general properties); spatula rub-out test

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Standard Test Methods for Chemical Analysis of Cuprous Oxide and Copper Pigments¹

This standard is issued under the fixed designation D 283; the number immediately following the designation indicates the year of original adoption 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 7232 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 25 was added and the title was changed editorially in May 1990.

1. Scope

1.1 These test methods cover procedures for the chemical analysis of cuprous oxide and copper pigments.

1.2 The analytical procedures appear in the following order:

	Sections
Total Copper	7
Total Reducing Power as Cuprous Oxide	8 and 9
Metallic Copper	10 and 11
Cuprous Oxide	12
Cupric Oxide	13
Metals Other than Copper	14 to 16
Chlorides and Sulfates	17 and 18
Acetone-Soluble Matter	19
Water	20
Stability	21
Coarse Particles	22
Coarse Particles Insoluble in Nitric Acid	23

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 hazard statements are given in Section 6.*

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 1193 Specification for Reagent Water³
- D 1208 Test Methods for Common Properties of Certain Pigments⁴

3. Significance and Use

3.1 This collection of test methods is used by pigment producers and paint manufacturers for process control, product acceptance, and research and development.

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. 26, 1984. Published January 1984. Originally published as D 283 - 28. Last previous edition D 283 - 52 (1978)^{e2}.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

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

⁴ Annual Book of ASTM Standards, Vol 06.02.

4. Treatment of Sample

4.1 Grind dry pigments, if lumpy or not finely ground, to a fine powder and thoroughly mix (Note 1). Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. In all cases thoroughly mix the sample before taking portions for analysis. Preserve all samples in dry, dark, airtight and completely filled bottles or containers to prevent oxidation. Some commercial copper oxides appear to segregate or oxidize rather easily. Therefore, the thorough mixing of the sample to ensure homogeneity and the rapid handling of the sample, when exposed to light and air, are extremely important factors in obtaining accurate results.

NOTE 1—It is very important that the sample be thoroughly mixed. Some samples of cuprous oxide are not homogeneous so are likely to give trouble when an attempt is made to obtain concordant results. By placing a few grams of a sample on a sheet of white paper and drawing it out with a spatula, it is frequently found that the sample contains coarse particles of black scale, along with small balls of bright red cuprous oxide. Thus, it may be necessary to pass the pigment through a No. 60 (250- μ m) sieve, break up any lumps of ground pigment by gentle pressure, and grind any coarse particles failing to pass through the sieve. Since oxidation of slight or even considerable magnitude may take place, these operations should be performed quickly, avoiding prolonged grinding and exposure to light and air.

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.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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

5.3 *Ammonium Sulfate* ((NH₄)₂SO₄).

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."

5.4 *Nitric Acid* (sp gr 1.42)—Concentration nitric acid (HNO_3).

5.5 *Perchloric Acid* (70 %)—Concentrated perchloric acid (HClO_4).

6. Hazards

6.1 **Warning**—Concentrated mineral acids cause burns of the skin and eyes. Concentrated bases are also hazardous. Avoid contact with skin or eyes. In case of contact immediately flush skin or eyes with plenty of water. See appropriate MCA Safety Data Sheets for further information before handling dangerous chemicals.

6.2 Chemicals that have been declared toxic should be disposed of as hazardous chemicals and not discharged into a sink.

TOTAL COPPER

7. Procedure

7.1 Weigh accurately 1.0 g of the sample and transfer to a 300-mL electrolytic beaker. Add 10 mL of concentrated nitric acid (HNO_3) and boil several minutes. Add 10 mL of HClO_4 (70 %) and fume for 5 min. Cool, dilute to a volume of approximately 150 mL, and add 1 mL of HNO_3 and 1 g of $(\text{NH}_4)_2\text{SO}_4$.

7.2 Carefully weigh the platinum electrode to 0.1 mg. Electroplate the copper on a rotating platinum electrode at a current of 2 A for 2 h. Dilute with water and continue the electrolysis for 15 min. When the deposition is complete, remove the electrode, wash with water and acetone, dry, and weigh to 0.1 mg. Save the electrolyte for the determination of metals other than copper (Section 15).

7.3 Calculate the percent of total copper, Cu.

TOTAL REDUCING POWER AS CUPROUS OXIDE

8. Reagents

8.1 *Ceric Ammonium Nitrate, Standard Solution* (0.1 N)—Mix 54.826 g of ceric ammonium nitrate ($\text{Ce}(\text{NH}_4)_2(\text{NO}_3)_6$) (either reagent grade or reference standard purity) with 56 mL of sulfuric acid (H_2SO_4) (1+1). Dissolve the salt and acid in water, cool to room temperature, and dilute to 1 L. Standardize this solution against analyzed bright copper foil that has been freed from all oxide coating.

8.2 *Ferric Chloride Solution*—Dissolve 75 g of ferric chloride ($\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$) in a mixture of 150 mL of hydrochloric acid (HCl) (sp gr 1.19) and 400 mL of distilled water. Add 5 mL of hydrogen peroxide (H_2O_2) (30 %) and boil to remove the excess.

8.3 *Ferrous Ammonium Sulfate, Standard Solution* (0.03 N)—Dissolve 12 g of ferrous ammonium sulfate ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6 \text{H}_2\text{O}$) in 200 to 300 mL of water and add 40 mL of H_2SO_4 (sp gr 1.84), while stirring constantly. Dilute to 1 L in a volumetric flask. A few pieces of mossy aluminum may be added to stabilize the solution. The solution should be restandardized frequently against 0.1 N ceric ammonium nitrate solution.

8.4 *Orthophenanthroline Indicator Solution* (0.5 % in water)—Orthophenanthroline ferrous complex (ferroin) shall be used as the indicator.

9. Procedure

9.1 Weigh accurately (Note 2) 0.15 g of the sample and place in a 250-mL, vented, glass-stoppered Erlenmeyer flask previously filled with carbon dioxide (CO_2) or other inert gas. Add a few small glass beads and 10 mL of FeCl_3 solution. Heat gently for 15 min, stirring occasionally and maintaining at all times an atmosphere of CO_2 or other inert gas.

NOTE 2—The use of a 0.4-mL micro beaker for weighing the specimen is advised. The beaker is weighed first and the sample introduced into the beaker, the correct weight obtained, and the entire beaker and its contents dropped into the flask. This eliminates errors in weighing caused by brushing the sample from glass balance pans.

9.2 After the specimen has been dissolved, cool, add 50 mL of distilled water, and titrate at once with 0.1 N ceric ammonium nitrate solution until near the end point. Add 2 drops of orthophenanthroline indicator solution and complete the titration. A sharp, distinct color change from orange to pale green occurs at the end point. Back-titrate with 0.03 N ferrous ammonium sulfate solution to the orange color.

9.3 *Calculation*—Calculate the percent of total reducing power, A, as cuprous oxide (Cu_2O) as follows:

$$A = \frac{V_1 N_1 - V_2 N_2 \times 0.07154}{S_1} \times 100 \quad (1)$$

where:

V_1 = $\text{Ce}(\text{NH}_4)_2(\text{NO}_3)_6$ solution required to titrate the specimen, mL,

N_1 = normality of the $\text{Ce}(\text{NH}_4)_2(\text{NO}_3)_6$ solution,

V_2 = $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ solution required for the back-titration, mL,

N_2 = normality of the $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ solution,

S_1 = specimen weight, g, and

0.07154 = equivalent weight of $\text{Cu}_2\text{O}/1000$.

METALLIC COPPER⁶

10. Reagents

10.1 *Ceric Ammonium Nitrate, Standard Solution* (0.1 N)—See 8.1.

10.2 *Denatured Alcohol* (Formula No. 2B)

10.3 *Extraction Solution*—Add 40 mL of HCl (sp gr 1.19) to 1 L of denatured alcohol. Mix thoroughly. Add 40 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) and stir until completely dissolved.

10.4 *Ferric Chloride Solution*—See 8.2.

10.5 *Orthophenanthroline Indicator*—See 5.4.

11. Procedure

11.1 Add approximately 20 mL of 4-mm diameter glass beads to a 250-mL Phillips beaker (assay flask). Weigh accurately 0.15 g of sample on a tared, glazed paper approximately $\frac{1}{2}$ in. (13 mm) square or a small watch glass that will fit into the Phillips beaker, and transfer the specimen and paper or watch glass to the beaker.

11.2 Add 25 mL of denatured alcohol and swirl vigorously for approximately 1 min to remove any surface coating

⁶ This test method is based on the procedure described by Irvin Baker and R. Stevens Gibbs, "Determination of Metallic Copper in Cuprous Oxide—Cupric Oxide Mixtures," *Industrial and Engineering Chemistry, Analytical Edition*, Vol 13, February 15, 1946, p. 124.

and break up lumps. While swirling the flask, slowly add 100 mL of the extraction solution.

11.3 After the addition of the extraction solution, swirl the flask vigorously for 5 min, adding lumps of dry ice (solid carbon dioxide) (Note 3) continuously during this time to lower the temperature of the solution to approximately -10°C . Break up any lumps in the solution with a glass rod or policeman.

11.4 Filter off the metallic copper-cupric oxide residue using a 125-mm close-texture paper, a filter cone to support the paper, and suction (Note 4). Continue the addition of dry ice to the flask and filter paper during the filtration to keep the solution cold. Wash the flask and filter paper with 150 to 200 mL of denatured alcohol, continuing the use of suction.

NOTE 3—During the 5-min swirling period approximately 25 to 30 g of dry ice is added in approximately 5-g portions. The initial lumps of dry ice volatilize rapidly due to the temperature of the solution. Approximately 15 g of dry ice is added in the first 2 min with a subsequent temperature drop to 0°C . The remaining 15 g volatilizes more slowly and gradually lowers the temperature to the vicinity of -10°C . These directions are not critical, but merely serve as a guide. The solution must be kept very cold in order to obtain correct and reproducible results. During the filtration period, additions of dry ice to the flask should be continued to keep the solution cold until all of the extraction solution has been filtered.

NOTE 4—In the great majority of cases, the metallic copper-cupric oxide residue is completely retained by the use of a suitable close-texture filter paper. The filtration is rapid and can be readily completed in 5 min with proper suction. The filtrate should be carefully examined for the presence of finely divided particles of copper. In the event that extremely finely divided particles of copper are present and pass through the filter paper, as evidenced by the presence in the filtrate of a reddish color, which may be transient, the following method of filtration should be used: Place a 25-mm diameter, beveled-edge, perforated porcelain filter disk in a 60°, 75-mm diameter glass funnel. Using suction, prepare an asbestos filter pad on the porcelain disk of sufficient thickness and retentiveness to hold the finely divided residue. Wash the asbestos pad several times with denatured alcohol to remove all water from the pad. Filter the metallic copper-cupric oxide residue on this asbestos pad with the suction on at the start of the filtration. Keep the solution cold by the addition of dry ice and proceed with washing the residue, transferring it to the original extraction flask, and the subsequent titration as directed in Section 11.

11.5 Transfer the entire residue, filter paper, and beads to the original extraction flask and dissolve the residue in 25 mL of FeCl_3 solution, keeping an atmosphere of CO_2 above the sample by addition of dry ice. Heat on a steam bath to dissolve the copper. Add 50 mL of distilled water and 3 drops of orthophenanthroline indicator solution. Titrate with 0.1 N ceric ammonium nitrate solution until the color changes from orange to pale green.

11.6 *Calculation*—Calculate the percent of metallic copper content, B , as follows:

$$B = \frac{V_3 C}{S_2} \times 100 \quad (2)$$

where:

V_3 = ceric ammonium nitrate solution required for titration of the sample, mL,
 C = copper equivalent of the ceric ammonium nitrate solution, g/mL, and
 S_2 = specimen weight, g.

CUPROUS OXIDE

12. Calculation

12.1 Calculate the percent of cuprous oxide (Cu_2O), D , as follows:

$$D = A - 2.252B \quad (3)$$

where:

A = total reducing power as cuprous oxide (Section 9), %, and
 B = metallic copper (Section 11), %.

CUPRIC OXIDE

13. Calculation

13.1 Calculate the percent of cupric oxide (CuO), E , as follows:

$$E = 1.252(F - B) - 1.112D \quad (4)$$

where:

F = total copper (Section 7), %,
 B = metallic copper (Section 11), %, and
 D = cuprous oxide (Section 12), %.

METALS OTHER THAN COPPER

14. Reagents

14.1 *Ceric Ammonium Nitrate, Standard Solution* (0.1 N)—See 8.1.

14.2 *Diphenylamine Indicator*—Dissolve 1 g of diphenylamine in 100 mL of H_2SO_4 (sp gr 1.84).

14.3 *Potassium Ferricyanide Solution* (50 g/L)—Dissolve 5 g of potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$) in 100 mL of distilled water. Keep tightly stoppered in a dark bottle.

14.4 *Potassium Ferrocyanide, Standard Solution* (1 mL = 0.001 g Zn)—Dissolve 5.0 g of potassium ferrocyanide ($\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$) in distilled water and dilute to 1 L. Standardize with analyzed reagent grade zinc prior to use.

15. Procedure

15.1 Filter the electrolyte from the determination of total copper (Section 7). Add 5 mL of H_2SO_4 (sp gr 1.84) and evaporate just to dryness to remove all HClO_4 and HNO_3 . Add approximately 150 mL of water and neutralize with NH_4OH (sp gr 0.90), adding 5 mL in excess. Add 10 g of NH_4Cl . Boil the solution gently for several minutes and allow the precipitate to settle. Filter through a close-texture paper, and wash the precipitate with 50 mL of hot NH_4Cl solution (20 g/L).

15.2 *Determination of Iron*—Dissolve the precipitate from the filter paper with hot HCl (1+1). Determine iron by the procedure of reduction with SnCl_2 solution, followed by addition of HgCl_2 and titration with 0.1 N ceric ammonium nitrate solution.

15.2.1 *Calculation*—Calculate the percent of iron, G , as follows:

$$G = \frac{V_4 N_1 \times 0.056}{S} \times 100 \quad (5)$$

where:

V_4 = ceric ammonium nitrate solution required to titrate the specimen, mL,
 N_1 = normality of the ceric ammonium nitrate solution, and

= specimen weight (Section 7), g.

15.3 *Determination of Cobalt and Nickel*—Evaporate the solution (see 15.1) to 15 to 20 mL. If the solution is colored, the presence of nickel or cobalt, or both, may be indicated. If present, they must be removed before determining zinc, determined by any standard procedure and reported as percent of cobalt or nickel, or both.

15.4 *Determination of Zinc*—Neutralize the solution (see 15.4) with H_2SO_4 (1+2) and add 15 mL in excess. Dilute to 100 mL with water. Add 10 drops of $\text{K}_3\text{Fe}(\text{CN})_6$ solution and 5 drops of diphenylamine indicator solution. Heat to 60°C and titrate with standard $\text{K}_4\text{Fe}(\text{CN})_6$ solution. A color change from blue to greenish yellow occurs at the end point.

15.4.1 *Calculation*—Calculate the percent of zinc, H , as follows:

$$H = \frac{V_3 Z}{S} \times 100 \quad (6)$$

where:

V_3 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the specimen, mL,

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, and

S = specimen weight (Section 7), g.

16. Report

16.1 Report as percent of metals other than copper the total percent of metals determined in accordance with Section 15.

CHLORIDES AND SULFATES

17. Reagents

17.1 *Barium Chloride Solution* (100 g/L)—Dissolve 117 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in water and dilute to 1 L.

17.2 *Silver Nitrate Solution*—Dissolve 5 g of silver nitrate (AgNO_3) in 100 mL of water.

17.3 *Sodium Carbonate Solution*—Prepare a saturated solution of sodium carbonate (Na_2CO_3) that is free of both chlorine (Cl) and sulfate (SO_4).

18. Procedure

18.1 Weigh accurately approximately 10 g of the sample, add an excess of concentrated HNO_3 , and heat gently until the sample is decomposed. Dilute with water to approximately 150 mL. Add an excess of Na_2CO_3 solution (16.3), and bring to a boil. Transfer to a 500-mL volumetric flask and dilute to the mark with water. Let settle.

18.2 Draw off two separate 50-mL aliquots of the clear solution. Make each slightly acid with HNO_3 .

18.3 *Determination of Chlorides*—To one of the 50-mL aliquots (18.2), add a few drops of AgNO_3 solution. If no precipitate forms, report no chlorides present. If a precipitate forms, add slowly an excess of AgNO_3 solution. Heat to boiling; after the precipitate has settled, filter through a Gooch crucible, and wash with cold water. Dry at 130°C , cool, and weigh.

18.3.1 *Calculation*—Calculate the percent of chlorides as Cl, J , as follows:

$$J = \frac{P_1 \times 0.247}{S_3 \times 0.1} \times 100 \quad (7)$$

where:

P_1 = AgCl , g, and

S_3 = specimen weight, g.

18.4 *Determination of Sulfates*—Dilute the second 50-mL aliquot (18.1) to approximately 200 mL. Add approximately 1 mL of HCl (1+1). Heat to boiling and add slowly an excess (10 mL) of BaCl_2 solution. Let settle, filter through a Gooch crucible, and wash. Dry, ignite at full red heat, cool in a desiccator, and weigh.

18.4.1 *Calculation*—Calculate the percent of sulfates as SO_4 , K , as follows:

$$K = \frac{P_2 \times 0.412}{S_3 \times 0.1} \times 100 \quad (8)$$

where:

P_2 = BaSO_4 , g, and

S_3 = specimen weight, g.

ACETONE-SOLUBLE MATTER

19. Procedure

19.1 Weigh accurately 5.0 g of the well-mixed sample and transfer to a 250-mL glass stoppered Erlenmeyer flask. Add 100 mL of acetone and shake vigorously for 15 min. Allow to settle and filter through a double, close-texture paper into a 250-mL Griffin beaker. Wash the flask and paper well with acetone to remove all acetone-soluble matter.

19.2 Evaporate the acetone on a water bath to a low volume and transfer to a 15 by 55-mm weighed aluminum moisture dish. Wash out the beaker with a small amount of acetone. Finally evaporate all acetone and heat the residue for 30 min in an oven at 105°C .

19.3 Report the weight of the dried residue as acetone-soluble matter.

WATER

20. Procedure

20.1 Determine the water content in accordance with Test Methods D 1208.

STABILITY

21. Procedure

21.1 Spread approximately 100 g of the sample on a watch glass and heat in an oven at a temperature of 100°F (40°C) and approximately 95 % relative humidity for a period of 72 h. Remove from the oven. Note any change in color of the pigment. Determine its total reducing power in accordance with Section 9.

21.2 Determine the percent of moisture on 50 g of the sample remaining from the test described in 18.1 using the distillation method specified in Section 20.

21.3 *Calculation*—Calculate the percent decrease in total reducing power, L , as follows:

$$L = \frac{N}{100 - M} \quad (9)$$

where:

L = total reducing power after stability test calculated on dry basis,

N = total reducing power after stability test as determined in 21.1, and

M = moisture (21.2), %.

$$L = \frac{A - E}{A} \times 100 \quad (10)$$

where:

L = decrease in total reducing power (from stability test), % and

A = total reducing power before stability test as calculated in 9.3.

COARSE PARTICLES

22. Procedure

22.1 Determine the percent of coarse particles on a 25-g specimen as described in Test Methods D 185 using both a No. 200 (75- μ m) and a No. 325 (45- μ m) sieve.

COARSE PARTICLES INSOLUBLE IN NITRIC ACID

23. Procedure

23.1 Transfer the residue retained on a No. 200 (75- μ m) sieve to a beaker, add 50 mL of HNO_3 (1+3), and boil for 5 min. Cool and filter through a tared Gooch crucible, previously washed with HNO_3 (1+3), or a fritted-glass filter

of appropriate porosity. Wash the insoluble residue on the filter with hot water. Dry the crucible or fritted-glass filter and contents at 105 to 110°C and weigh.

23.2 *Calculation*—Calculate the percent of coarse particles insoluble in nitric acid, T , as follows:

$$T = \frac{R}{S} \times 100 \quad (11)$$

where:

R = weight of residue, g, and

S = weight of original specimen, g.

24. Precision

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

25. Keywords

25.1 chemical analysis; copper pigments; cuprous oxide pigments

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Standard Test Methods for Chemical Analysis of Mercuric Oxide Pigment¹

This standard is issued under the fixed designation D 284; the number immediately following the designation indicates the year of original adoption 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 analysis of mercuric oxide pigment.

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. A specific hazard statement is given in the Note of 11.1.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1208 Test Methods for Common Properties of Certain Pigments³

3. Significance and Use

3.1 These test methods are intended as a quick and reliable procedure for measuring purity of mercuric oxide pigment to determine if it meets purity standards as agreed upon between the producer and the consumer.

4. Purity of Reagents and Materials

4.1 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.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.

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

5. Preparation of Sample

5.1 If the sample is large, mix it thoroughly before taking a representative portion. Grind the representative portion to

a fine powder and thoroughly mix before taking portions for analysis. Keep the sample in a stoppered glass bottle.

6. Precision

6.1 Repeatability and reproducibility are believed to be well within the limits usually obtained on similar chemical test methods, but no actual figures on precision are available.

ALKALINITY OR ACIDITY

7. Procedure

7.1 Determine alkalinity or acidity in accordance with Test Methods D 1208.

FREE MERCURY

8. Procedure

8.1 Examine a representative specimen of the dry mercuric oxide under a microscope for the presence of free mercury.

TOTAL MERCURY

9. Reagents and Materials

9.1 *Ammonium Thiocyanate, Standard Solution* (1 mL = 0.012 g Hg)—Dissolve 9 g of ammonium thiocyanate (NH_4CNS) in water and dilute to 1 L. Standardize the solution against mercury, as follows: Weigh to 0.1 mg about 4.6 g of mercury, and dissolve it in 40 mL of warm HNO_3 (1+1). Dilute to 200 mL with water and add KMnO_4 solution (50 g/L) dropwise until the pink color persists for 5 min in order to ensure the absence of nitrous acid (HNO_2) and monovalent mercury. Add FeSO_4 solution (50 g/L) dropwise to destroy excess permanganate. Add 4 mL of ferric ammonium sulfate indicator solution, and titrate with the NH_4CNS solution.

9.2 Calculate the mercury equivalent M of the NH_4CNS solution, in grams per millilitre, as follows:

$$M = W_1 / V_1$$

where:

W_1 = mercury used, g, and

V_1 = NH_4CNS solution required for titration, g.

9.3 *Ferric Ammonium Sulfate Indicator Solution*—Dissolve enough ferric ammonium sulfate ($\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$) in water to make a saturated solution at room temperature and add HNO_3 (sp gr 1.42) dropwise to bleach the brown color of the solution. About 30 g of ferric ammonium sulfate per 100 mL of water will be required for the saturated solution.

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 D 01.21 on Chemical Analysis of Paints and Paint Materials.

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² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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 Company, Inc., New York, NY, and the "United States Pharmacopeia."

9.4 *Ferrous Sulfate Solution* (50 g/L)—Dissolve 5 g of ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in water and dilute to 100 mL.

9.5 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

9.6 *Nitric Acid* (1+1)—Mix 1 volume of HNO_3 (sp gr 1.42) with 1 volume of water. This acid must be free of nitrous acid (HNO_2).

9.7 *Potassium Permanganate Solution* (50 g/L)—Dissolve 5 g of potassium permanganate (KMnO_4) in water and dilute to 100 mL.

10. Procedure

10.1 Weigh to 1 mg about 0.5 g of the sample, previously dried for 1 h at 150°C into a 750-mL Erlenmeyer flask. Add 40 mL of HNO_3 (1+1), and warm gently until the specimen is dissolved.

10.2 Dilute to 200 mL with water, and add 4 mL of ferric ammonium sulfate indicator solution. Titrate with 0.1 N NH_4CNS solution until a distinct pink color persists after vigorous shaking.

11. Calculation

11.1 Calculate the percent of mercury P as follows:

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$$P = [(V_2 \times M)/S] \times 100$$

where:

V_2 = NH_4CNS solution required for titration of the specimen, mL

M = mercury equivalent of the NH_4CNS solution, g/mL, and

S = dried specimen, g.

ASH

12. Procedure

12.1 Ignite 2.0 g of a dry specimen in a weighed porcelain crucible or dish under a well-ventilated hood. (Warning—See Note) Cool and weigh the residue. Calculate the percent of ash.

Note: Warning—The fumes are poisonous.

INDEX TERMS

13. Index Terms

13.1 These Test Methods are indexed under the following terms: mercuric oxide; pigments—mercuric oxide.



Standard Test Methods for Soluble Cellulose Nitrate¹

This standard is issued under the fixed designation D 301; the number immediately following the designation indicates the year of original adoption 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 material known as soluble cellulose nitrate (also known as soluble nitrocellulose), which is shipped wet in conformance with regulations of the Interstate Commerce Commission.

1.2 The test methods appear in the following sections:

	Sections
Drying Samples	5 to 7
Film Test	4
Nitrogen	20 and 21
Solubility and Appearance of Solution	8 to 10
Stability	19
Toluene Dilution	11 to 13
Viscosity	22 to 24
	14 to 18

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 Notes 2 through 5.

2. Referenced Documents

2.1 ASTM Standards:

- D 302 Specification for Ethyl Acetate (85 to 88 Percent Grade)²
- D 303 Specification for *n*-Butyl Acetate (90 to 92 % Grade)³
- D 362 Specification for Industrial Grade Toluene²
- D 1343 Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method⁴
- E 1 Specification for ASTM Thermometers⁵

3. Sampling

3.1 Samples shall be taken from not less than 10 % (at least two barrels) of each lot or batch in the shipment. In sampling the barrels, two samples of approximately 1 pt (0.5 dm³) each shall be taken from two well-separated points at least 1 ft (0.3 m) beneath the surface of the material in the barrel. These samples shall then be composited to represent each lot or batch in the shipment.

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 D01.36 on Cellulosics.

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² Annual Book of ASTM Standards, Vol 06.03.

³ Discontinued, see 1986 Annual Book of ASTM Standards, Vol 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.02.

⁵ Annual Book of ASTM Standards, Vols 05.03 and 14.03.

3.2 The samples shall meet the following requirements:

3.2.1 *Appearance*—The cellulose nitrate shall not be discolored and shall be free of lumps and foreign matter, such as charred particles.

3.2.2 *Ash*—Ash content shall not exceed 0.30 %, calculated on the basis of dry-weight soluble cellulose nitrate.

3.2.3 *Nitrogen*—The percent nitrogen, calculated on the basis of dry-weight soluble cellulose nitrate, shall be within the limits agreed upon by the purchaser and the manufacturer for the particular type of soluble cellulose nitrate.

3.2.4 *Stability*—The stability as determined by the 134.5 C test shall be not less than 25 min.

3.2.5 *Viscosity*—The viscosity shall be within the limits agreed upon by the purchaser and the manufacturer for the particular type of soluble cellulose nitrate.

3.2.6 *Solubility and Appearance of the Solution*—The solubility and appearance of the sample shall be equal to the reference standard for the particular type of soluble cellulose nitrate.

3.2.7 *Film Test*—The film test of the sample shall be equal to that of the reference standard for the particular type of soluble cellulose nitrate.

3.2.8 *Toluene Dilution Test*—The toluene dilution value of the sample shall be equivalent to that of the reference standard for the particular type of soluble cellulose nitrate.

DRYING SAMPLES

4. Procedure

4.1 Soluble cellulose nitrate is a flammable material, the degree of flammability varying with the extent and nature of the wetting medium. Cellulose nitrate is always wet with water or alcohol in commercial handling, shipping, and storage, in which condition it presents no unusual hazard. Dry cellulose nitrate, if ignited by fire, spark, or static electricity, burns very rapidly. Samples of dry cellulose nitrate *must not* be stored at any time. Dry only that portion required for immediate test. Wet the excess material and the samples left after testing with water and dispose of by burning on a safe burning ground.

4.2 Dry small quantities required for ash and nitrogen tests by spreading in a thin layer on a tray at room temperature for 12 to 16 h, followed by oven-drying in crucibles or weighing bottles 1 h at 100 to 105°C. The oven used for drying cellulose nitrate should have the latch removed. Wear a face mask (see 9.2) when the oven is opened after samples have been heated.

4.3 Dry larger quantities of *water-wet* material required for viscosity and toluene dilution tests, or a small quantity for stability tests, by blowing warm compressed air (at a temperature of 60 to 65°C, and a pressure of 40 to 60 psi

(275 to 415 kPa)) through the sample placed in a cylindrical holder with a screen over one end for $\frac{1}{2}$ to 1 h. Provide the compressed air line with a safety plug (Note 1) of Wood's metal, which melts at 70 to 75°C, so the air will be diverted from the sample if a temperature of 70°C is exceeded.

NOTE 1—Information on the availability of a suitable fusible plug assembly may be obtained from ASTM Headquarters.

4.4 If the material is *alcohol-wet*, it is necessary to modify the drying procedure. After placing the required amount of cellulose nitrate in the cylindrical holder, pour in sufficient distilled or iron-free water to fill it. Allow the bulk of the liquid to drain off. Then dry by blowing warm air through the holder as described in 4.3.

ASH

5. Significance and Use

5.1 Ash accounts for the nonsoluble, nonfilm forming portion of the polymer. It may affect solution clarity and film properties.

6. Procedure

6.1 Dry the cellulose nitrate as described in 4.2 and place a specimen of approximately 2.0 g in a tared and ignited crucible. After drying at 100 to 105°C for 1 h, cool in a

desiccator and weigh accurately. Moisten the specimen with 20 to 30 drops of ACS grade HNO_3 (sp gr 1.42) and heat on a steam bath until the specimen is decomposed to a gummy mass. If necessary to complete the decomposition, add several additional drops of HNO_3 at intervals. Then heat the crucible slowly over a bunsen burner or in an electric furnace until all volatile matter is driven off. Finally, ignite at red heat to constant weight, cool, and weigh.

7. Calculation

7.1 Calculate the percent ash as follows:

$$\text{Ash, \%} = (\text{wt of ash/wt of dry sample}) \times 100$$

NITROGEN

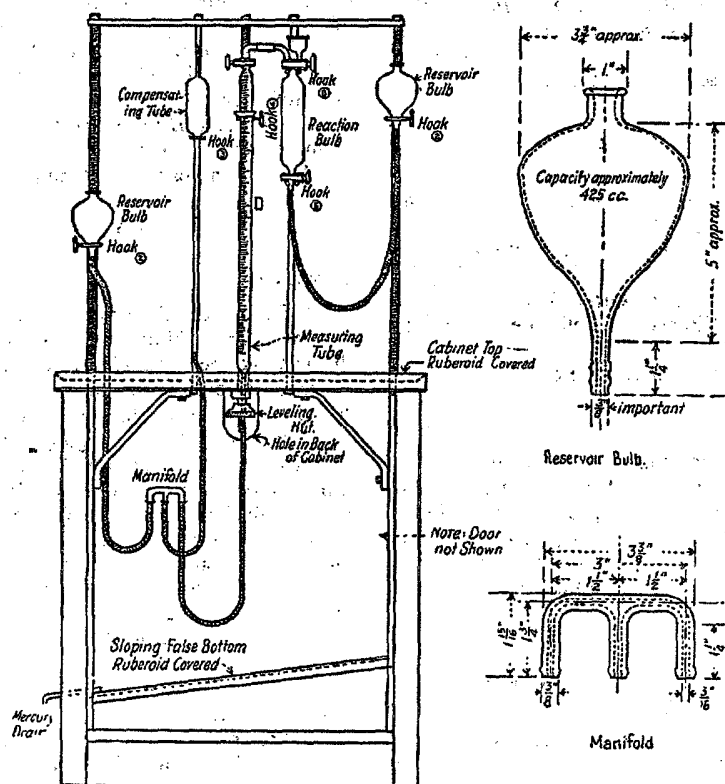
8. Significance and Use

8.1 The nature and strength of solvent systems required for cellulose nitrate are dependent upon the nitrogen content. Mismatches of solvent with nitrogen level can result in poor solution quality and colloid and gel formation.

9. Apparatus

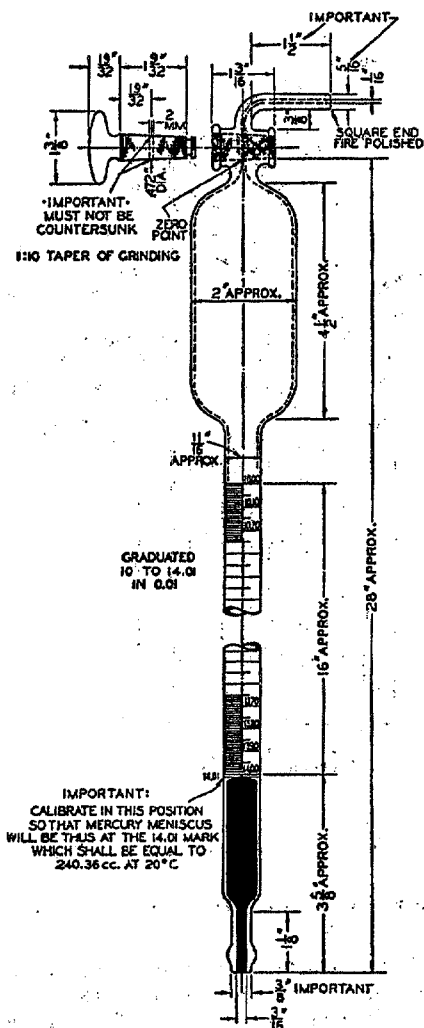
9.1 *Nitrometer*—Use the duPont Nitrometer, which is illustrated in Figs. 1 to 4.

9.2 *Face Mask*—A face mask, so constructed that a heavy



NOTE—1 in. = 25.4 mm.

FIG. 1 General Assembly of Apparatus for Nitrogen Determination



NOTE—1 in. = 25.4 mm.

FIG. 2 Measuring Tube for Nitrogen Determination

piece of cellulose acetate sheeting protects the face.

NOTE 2: Precaution—The cellulose acetate mask must be worn during the generation and measurement of the gas as a precaution in case of an explosion.

10. Procedure

10.1 Calibrate the measuring tube accurately in the usual manner, using mercury as the calibrating liquid.

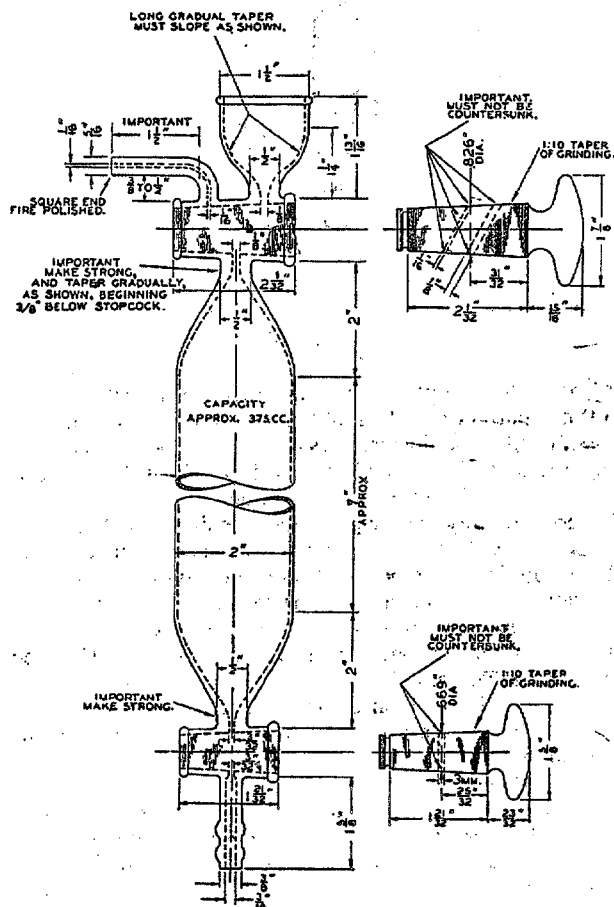
10.2 Standardize the apparatus as follows:

10.2.1 Fill the compensating, measuring, and reaction tubes and their rubber connections with mercury. Run 20 to 30 mL of H_2SO_4 (ACS grade, $94.5 \pm 0.5\%$) into the reaction bulb through the cup at the top and admit about 210 mL of air. Close the stopcocks, shake the bulb well, and allow to stand overnight. This desiccates the air which is then run into the compensating tube until the mercury is about on a level with the 12.50 % mark on the measuring tube, the two tubes

being held at the same height. Then seal the compensating tube using a small blowpipe flame.

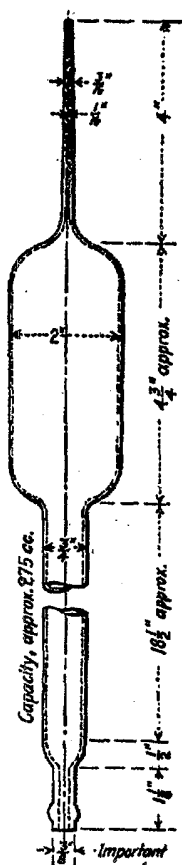
10.2.2 As a preferred alternative, nitrogen may be used in place of air.

10.2.3 Place in weighing bottles 0.95 ± 0.05 -g portions of ACS grade KNO_3 that has been recrystallized twice from distilled water and ground to pass a No. 100 (150- μm) sieve. Dry the specimens 2 to 3 h at 135 to 150°C. Stopper the bottles, cool in a desiccator, and weigh accurately. Transfer the KNO_3 to the cup of the reaction bulb and weigh the weighing bottle to obtain the weight of sample used. Add 1.0 mL of water and stir the mixture in the cup with a small glass stirring rod to liberate the entrained bubbles of air; work the undissolved crystals into the lower part of the cup, keeping them below the surface of the solution. It is not necessary that the KNO_3 dissolve before drawing it into the reaction bulb. *Make sure the lower stopcock is open*; then admit the mixture to the bulb by a series of quick openings of the upper stopcock, in the meantime keeping the crystals below the surface of the liquid. In this way, all but a small amount of the KNO_3 may be run into the bulb. Rinse the cup with a



NOTE—1 in. = 25.4 mm.

FIG. 3 Reaction Bulb



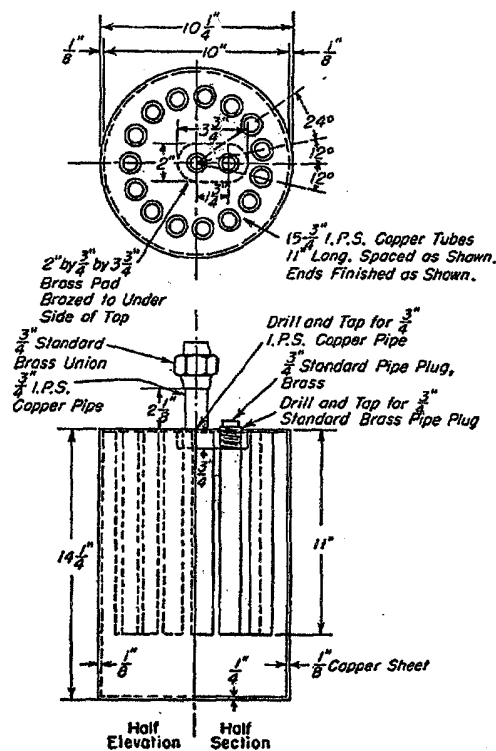
NOTE—1 in. = 25.4 mm.

FIG. 4 Compensating Tube

second 1.0-mL portion of water; then repeat with a third 1.0-mL portion (3 mL in all). This should be sufficient to dissolve all remaining particles of KNO_3 in the cup. Transfer 25 mL of the H_2SO_4 ($94.5 \pm 0.5\%$), divided in several portions, to the cup, and subsequently to the bulb by lowering the reservoir slightly and opening and closing the upper stopcock, care being taken that no air enters even the bore hole in the stopcock. There must always be a slight suction when introducing the specimen, the wash water, and the acid, but never enough to cause air to be sucked into the reaction bulb. The quantities of water and H_2SO_4 used should be constant. Then with the bottom stopcock still open, lower the reservoir bulb to give reduced pressure in the reaction bulb and gently shake the reaction bulb to start the decomposition.

10.3 After the evolution of NO has become slow (Precaution, see Note 3), lower the reservoir bulb until all but 25 mL of the mercury in the reaction bulb is withdrawn, close the bottom stopcock, and shake the reaction bulb vigorously for 5 min.

NOTE 3: Precaution—It is extremely important that the bottom stopcock be left open until the major part of the decomposition has



NOTE—1 in. = 25.4 mm.

FIG. 5 Copper Bath for Stability Test

occurred; otherwise, sudden evolution of gas will burst the bulb, scattering acid and glass.

10.4 When the reaction is completed, allow the gas to cool for 20 min; then transfer the gas to a measuring tube. By means of the leveling device make careful adjustment of the mercury levels so that the mercury in the measuring tube is at the 13.85% mark (the theoretical percent nitrogen in KNO_3) if an exactly 1.000-g specimen was used, or a proportional reading if less was used. Paste a strip of paper on the compensating tube at the level of the mercury, and the standardization is completed. It is advisable to make several check determinations, preferably on different days, to ensure accurate standardization. Determinations should check within $\pm 0.01\%$.

10.5 Dry the cellulose nitrate as described in 4.2 and place a specimen of 1.0 to 1.2 g in a weighing bottle. After drying at 100 to 105°C for 1 h, stopper, cool in a desiccator, and weigh accurately. Transfer the specimen to the cup of the decomposition bulb; then reweigh the empty bottle to get the weight of the specimen by difference. Add 5 to 10 mL of H_2SO_4 ($94.5 \pm 0.5\%$) to the cup and stir the mixture with a small stirring rod. Lower the mercury reservoir and then, with the lower stopcock open, draw the mixture in by opening the upper stopcock. Take care that no air is drawn in. Rinse the cup of the decomposing bulb several times with H_2SO_4 , using a total of 25 mL for dissolving and rinsing. Complete the determination in accordance with the proce-

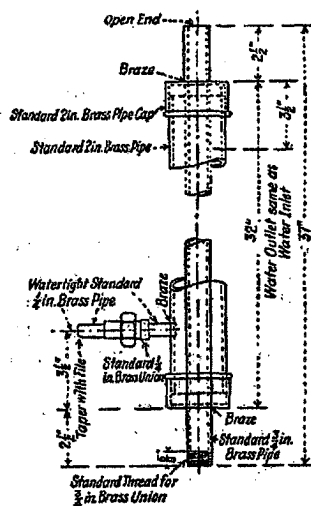


FIG. 6. Brass Condenser for Stability Test Apparatus

cedure described in 10.2 for standardization of the apparatus, and take a reading after adjusting the level of the mercury in the reading tube to the mark on the compensating tube. The reading divided by the weight of the specimen gives the percent nitrogen.

STABILITY

11. Significance and Use

11.1 Nitrocellulose stability is measured by detecting the evolution of nitrogen oxides under elevated temperature. The results are not necessarily a predictor of shelf life.

12. Apparatus

12.1 *Copper Bath*—Copper bath with copper or brass condenser, as shown in Figs. 5 and 6. These baths are usually made to hold 13 to 15 test tubes. To aid in heat transfer, add 15 to 25 mL of mineral oil to each copper well, in order to fill the space between the glass tube and the well. To maintain the bath at a temperature of $134.5 \pm 0.5^\circ\text{C}$, fill to within 3 in. (76 mm) of the top with a mixture consisting of ten parts of a commercial ethylene glycol solution (automobile radiator antifreeze containing a corrosion inhibitor) and one part of water. Adjust the temperature of the boiling liquid in the bath to $134.5 \pm 0.5^\circ\text{C}$ by adding more glycol or water, as necessary.

12.2 *Test Tubes*—Heat-resistant glass⁶ tubes, with an outside diameter of 18 mm, a wall thickness of 1.5 mm, and a length of 290 mm.

12.3 *Heater*—An electric hot plate for heating the bath.

12.4 *Face Mask*—See 9.2.

12.5 *Gloves*—A pair of heavy gloves.

12.6 *Pincers*—Long pincers for handling the test tubes.

12.7 *Thermometer*—An ASTM Stability Test Thermometer having a range from 130 to 140°C and conforming to the requirements for Thermometer 26C as prescribed in Specifi-

cation E 1. The thermometer should be fitted with a cork stopper and placed in an empty glass tube in the bath.

12.8 *Methyl Violet Test Paper*.⁷

13. Procedure

13.1 Conduct the test in a room that is free of acid fumes.

NOTE 4: Precaution—It is important that the operator wear the cellulose acetate mask and heavy gloves and the tubes be handled with long pincers.

13.2 Dry the sample as described in 4.2. Weigh duplicate specimens of 2.5 ± 0.1 g into test tubes and press the specimens down so that they occupy the lower 2 in. (51 mm) of the tubes; then swab out all cellulose nitrate particles adhering to the inside wall of the tubes. Crease a piece (20 by 70 mm) of normal methyl violet test paper for one half its length; then insert it in the tube with the uncreased portion downward, until the lower edge is 1 in. above the top of the specimen. The paper must remain in this portion throughout the test. Stopper the tube with a cork provided with hole or notch 4 mm in diameter. Place the tube, without jarring, in the heating bath maintained at $134.5 \pm 0.5^\circ\text{C}$. Beginning at the end of the first 20 min, inspect the tube at 5-min intervals by lifting the tube until the methyl violet paper, but not the cellulose nitrate, is visible above the surface of the bath. The end point is reached when the entire test paper changes in color to salmon pink. For example, if the color is not completely changed at 20 min but is completely changed at 25 min, record the stability of the specimen as 25 min.

NOTE 5: Precaution—As a safety measure, immerse the specimen in cold water immediately upon completion of the test.

VISCOSITY

14. Significance and Use

14.1 Coating and lacquer formulations are based on percent solids in a solvent system. Viscosity is a determining factor in limiting the percent solids in a given solvent system.

15. Solutions Required

15.1 Determine viscosity by dissolving the cellulose nitrate, dried as described in 4.3, according to a standard formula (Table 1) and noting the time for a steel ball to fall through a measured depth of the solution at 25°C . Use formula A for types designated as 5 s or over. Use formula B for types designated as $\frac{1}{2}$ and $\frac{3}{4}$ s. Use formula C for types designated as $\frac{1}{4}$ s and 30 to 35 cp. Cellulose nitrate will dissolve more quickly if it is first wet with alcohol and toluene and the mixture then allowed to stand a few minutes before the ethyl acetate is added. Completely dissolve the sample in the solvent mixture by agitating in a tightly closed bottle.

16. Viscosity Determination

16.1 Measure the viscosity, using the apparatus and following the procedure described in Test Method D 1343.

⁶ Borosilicate glass has been found satisfactory for this purpose.

⁷ The normal methyl violet test papers are available from the Naval Powder Factory, Indian Head, MD.

17. Report

17.1 Report the results in seconds (Note 6) for a $\frac{3}{32}$ -in. (2.4-mm) steel ball (density 7.7 ± 0.1) and a 2.00 ± 0.02 -in. (50.8 $\pm$ 0.5-mm) drop. The viscosity value shall be prefixed with the letter A, B, or C, corresponding to the formula of the solution employed.

NOTE 6—Results in seconds for a $\frac{3}{32}$ -in. steel ball may be converted to poises as follows:

$$n = 0.560 (a - b) t$$

where:

n = viscosity at the specified temperature, P,
 a = ball density, g/mL,
 b = solution density, g/mL, and
 t = time of fall, s.

18. Precision and Bias

18.1 *Precision*—The coefficient of variation between two results obtained by a single analyst is estimated to be 2.6 % at 10 df. Two results should be considered suspect, if they differ by more than 7 %.

18.2 *Bias*—No statement on bias can be made at this time as no material is available to serve as a standard.

SOLUBILITY AND APPEARANCE OF SOLUTION

19. Procedure

19.1 Compare either formula A, B, or C (Table 1) with a freshly-prepared standard solution of the same type of soluble cellulose nitrate of the same formula. Make the comparison in small vials and note the color, turbidity, "grain," and "flock."

TABLE 1 Solutions for Viscosity and Film Tests

	Formula A	Formula B	Formula C
Ingredients:			
Soluble cellulose nitrate (dried), weight percent	12.2	20.0	25.0
Completely denatured ethyl alcohol (188 to 190 proof), weight percent	22.0	20.0	18.75
Toluene, ^A weight percent	48.3	44.0	41.25
Ethyl acetate, ^B weight percent	17.5	16.0	15.0
Density of solution at 25°C (g/mL)	0.90	0.93	0.97

^A Toluene conforming to Specification D 362.

^B Ethyl acetate conforming to Specification D 302.

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FILM TEST

20. Significance and Use

20.1 The quality of a lacquer or other surface finish is dependent upon clarity and gloss of the dry film. This test method can reveal potential impurities and flaws in applied finishes.

21. Procedure

21.1 Dilute formula A, B, or C (Table 1) with an equal volume of normal butyl acetate and pour the solution beside a freshly prepared standard solution of the same type of soluble cellulose nitrate and of the same formula, on a clean glass plate. Allow to dry in a nearly vertical position in a dust-free atmosphere. Compare the specimen with the standard for undissolved particles, which indicate unnitrated cellulose or impurities, and for poor flow and poor gloss.

TOLUENE DILUTION

22. Significance and Use

22.1 This test method can ascertain whether cellulose nitrate of a given percent nitrogen is contaminated with material of a significantly different nitrogen content. Such cross contamination may adversely affect solution and film quality.

23. Procedure

23.1 Prepare a solution containing 12.2 weight percent of cellulose nitrate dried as described in 4.3, and 87.8 weight percent of butyl acetate conforming to Specification D 303. To 50 mL of this solution in a stoppered bottle add toluene, conforming to Specification D 362 in small quantities from a buret, shaking well after each addition.

24. Calculation and Report

24.1 Report as the dilution value the volume of toluene required to effect the first permanent separation of cellulose nitrate, calculated as a percent by volume of the original solution, as follows:

$$\text{Dilution value} = (\text{mL toluene} \times 100) / 50 \\ = \text{mL toluene} \times 2$$

NOTE 7—Large quantities of butyl acetate and toluene should be reserved for this test to avoid possible variation between different lots. The test should be run at 25°C.



Standard Test Method for Solvent-Extractable Material in Black Pigments¹

This standard is issued under the fixed designation D 305; the number immediately following the designation indicates the year of original adoption 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 9 was added editorially in May 1990.

Scope

1.1 This test method covers the determination of the solvent-extractable material in black pigments such as carbon black, lampblack, and bone black.

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 329 Specification for Acetone²

3. Significance and Use

3.1 This test method is used by black pigment producers and users for product acceptance.

4. Apparatus

4.1 *Extraction Apparatus*, consisting of a flask, siphon cup, and a condenser similar to the apparatus shown in either Fig. 1 or Fig. 2.

NOTE 1—A Siphlet apparatus may be used as an alternative.

4.2 *Extraction Thimbles*—The thimble must be made of greaseless paper and be of correct size to fill the selected apparatus.

NOTE 2—The recommended thimble size for each of the apparatus shown in Fig. 1 is listed below:

	Fig. 1	Fig. 2
Height	70 mm	62 mm
Inside diameter	28 mm	22 mm
Outside diameter	30 mm	24 mm

4.3 The thimbles are available in various heights and widths with two thickness levels, single thickness or double thickness. Single-thickness thimbles are recommended for this extraction procedure.

5. Reagents and Material

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended

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.

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² *Annual Book of ASTM Standards*, Vol 06.03.

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."

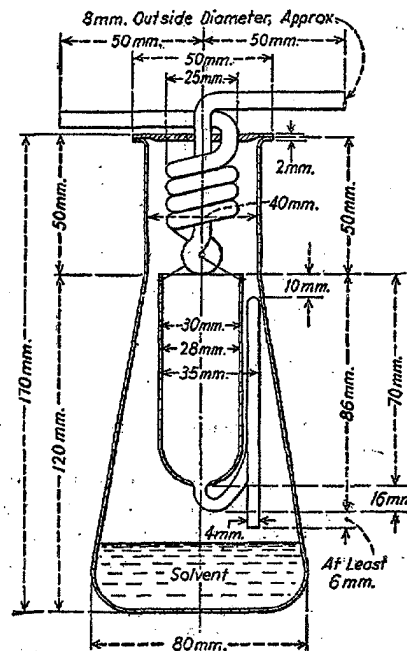


FIG. 1 Extraction Apparatus

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 *Solvent*—Acetone and benzene are the most commonly used solvents (see Specification D 329); however, other solvents may be used as agreed upon.

5.3 *Glass Wool*.

6. Procedure

6.1 Weight approximately 10 g of pigment (moisture-free)

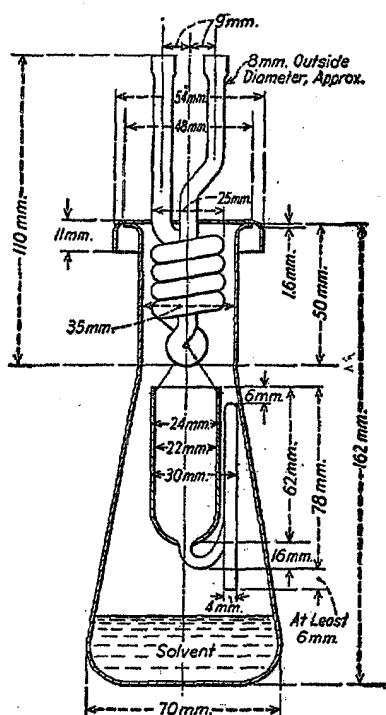


FIG. 2 Extraction Apparatus

into a weighed thimble and record the weight to 0.1 g. For pigments with high apparent density (bone black, iron oxides, etc.) a 40-g sample is recommended. Plug the open end of the thimble with glass wool and place in the siphon

cup. Add 200 to 250 mL of the solvent to the previously dried flask.

6.2 When using acetone as the extraction solvent, extract continuously for 4 h, heating at a rate such that the time required to fill and empty the siphon cup will not exceed 8 min. With other solvents extract for 16 h.

NOTE 3—If particles of carbon have escaped from the cup into the extraction liquid, filter the liquid, wash the paper with solvent, and return the total filtrate to the extraction flask, prior to continuing with 6.3.

6.3 Transfer quantitatively the extract solution to a 400-mL beaker that has been weighed to the nearest 0.1 g, and evaporate off the solvent on a steam bath or plate. Remove the beaker from the bath or hot plate just before the last trace of the solvent disappears. Dry the dish for 1 h at 105°C (221°F). Cool and weigh to the nearest 0.1 g.

7. Calculation

7.1 Calculate the percent extractable material, E , to the nearest 0.05 % as follows:

$$E = [(R - B)/(S - W)] \times 100$$

where:

R = weight of extractable material and weighed dish, g,

B = weight of weighed dish, g,

S = weight of thimble and specimen, g, and

W = weight of thimble, g.

8. Precision

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

9. Keywords

9.1 black pigment; solvent extractable

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22.1 This test method can ascertain whether cellulose nitrate of a given percent nitrogen is contaminated with material of a significantly different nitrogen content. Such cross contamination may adversely affect solution and film quality.

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23.1 Prepare a solution containing 12.2 weight percent of cellulose nitrate dried as described in 4.3, and 87.8 weight percent of butyl acetate conforming to Specification D 303. To 50 mL of this solution in a stoppered bottle add toluene, conforming to Specification D 362 in small quantities from a buret, shaking well after each addition.

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24.1 Report as the dilution value the volume of toluene required to effect the first permanent separation of cellulose nitrate, calculated as a percent by volume of the original solution, as follows:

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^{e1} NOTE—Section 9 was added editorially in May 1990.

Scope

1.1 This test method covers the determination of the solvent-extractable material in black pigments such as carbon black, lampblack, and bone black.

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.

Referenced Document

2.1 *ASTM Standard:*
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Significance and Use

3.1 This test method is used by black pigment producers and users for product acceptance.

Apparatus

4.1 *Extraction Apparatus*, consisting of a flask, siphon cup, and a condenser similar to the apparatus shown in either Fig. 1 or Fig. 2.

NOTE 1—A Siphlet apparatus may be used as an alternative.

4.2 *Extraction Thimbles*—The thimble must be made of greaseless paper and be of correct size to fill the selected apparatus.

NOTE 2—The recommended thimble size for each of the apparatus shown in Fig. 1 is listed below:

	Fig. 1	Fig. 2
Height	70 mm	62 mm
Inside diameter	28 mm	22 mm
Outside diameter	30 mm	24 mm

4.3 The thimbles are available in various heights and widths with two thickness levels, single thickness or double thickness. Single-thickness thimbles are recommended for this extraction procedure.

Reagents and Material

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended

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5.2 *Solvent*—Acetone and benzene are the most commonly used solvents (see Specification D 329); however, other solvents may be used as agreed upon.

5.3 *Glass Wool*.

Procedure

6.1 Weight approximately 10 g of pigment (moisture-free)

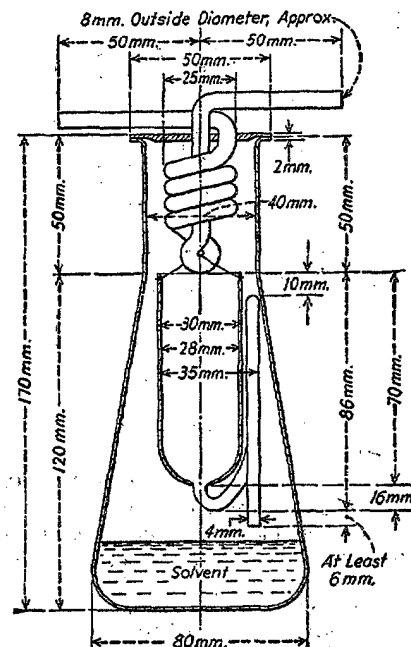


FIG. 1 Extraction Apparatus

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."

Standard Test Method for Relative Tinting Strength of White Pigments by Visual Observation¹

This standard is issued under the fixed designation D 332; the number immediately following the designation indicates the year of original adoption 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 4222 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.

⁶¹ NOTE—Keywords were added editorially in August 1991.

Scope

1.1 This test method describes the procedure for determining the relative tinting strength of white pigments by visual assessment of blue tints.

1.2 This test method is applicable only for comparing the test pigment with a reference standard of the same type and grade.

NOTE 1—ASTM Test Method D 2745 describes a procedure for instrumental evaluation of black tinted samples.

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 262 Specification for Ultramarine Blue Pigment²

D 2745 Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements²

3. Summary of Test Method

3.1 Specified amounts of white pigment and blue tinting pigment are dispersed together in oil using a glass hand muller or an automatic muller. Both the test and standard pigments are treated identically. The pastes are drawn-down together on a panel and visually assessed for tinting strength. To obtain a numerical rating of tinting strength, dispersions with the standard white pigment and more or less of the tinting pigment are made until the lightness of the test pigment paste is matched. The weight of the tinting pigment is used to calculate relative tinting strength.

4. Significance and Use

4.1 This test method is used as a referee method and for quality control. The vehicle (oil) for preparing the dispersions and the tinting pigment (ultramarine blue) are specified

but other vehicles and tinting pigments can be used. Any such changes in the test method must be agreed upon between the purchaser and the seller.

4.2 The results obtained with a muller do not necessarily agree with an industrial situation where different dispersing conditions exist. However, dispersing with a muller is a fast and relatively inexpensive way of testing tinting strength for routine quality control.

5. Apparatus

5.1 *Balance*, laboratory-type, sensitive to 0.1 mg, equipped with a counter-balanced watch glass.

5.2 *Buret*, 1-mL capacity, stopcock controlled, graduated in 0.1-mL divisions, or other suitable dispensing apparatus with a delivery accurate to 0.05 mL.

5.3 *Glass Hand Muller*—A weighted glass hand muller with beveled edge having a total weight of 15 lb (6.8 kg) and a grinding face of from 2¾ to 3 in. (70 to 75 mm) in diameter. The face shall be free of blowholes and other imperfections and kept roughened by lightly grinding with No. 303 optical emery, or its equivalent, and turpentine.

5.4 *Rubbing Surface*—A ground glass plate, at least 14 by 20 in. (355 by 510 mm), the surface of which is kept roughened by lightly grinding with No. 303 optical emery, or its equivalent, and turpentine.

5.5 *Automatic Muller*, automatic,³ equipped with a weight that exerts a permanent 50-lbf (220-N) force and an additional weight exerting a 50-lbf making a total of 100-lbf (445-N). The two glass plates shall be kept sharp by removing from the machine and grinding them face-to-face with No. 303 optical emery or equivalent, and water.

5.6 *Spatula*—A flexible spatula having a chromium-plated or plastic blade 3 to 6 in. (75 to 150 mm) long.

5.7 *Panels* of bright tin, glass, or white-lacquered cardboard.

5.8 *Scraper*—A French scraper or stiff scraping knife having a blade that is about 3 or 4 in. (75 to 100 mm) wide with a straight edge.

6. Materials

6.1 *Tinting Material*—Ultramarine blue conforming 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 D01.26 on Optical Properties.

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² Annual Book of ASTM Standards, Vol 06.02.

³ A muller supplied by Hoover Color Corp., P. O. Box 218, Hiwassee, VA 24347, has been found satisfactory for this purpose.

TABLE 1 Quantities of Materials for Tinting Strength Tests

Pigment Type	Weight of Pigment, g	Weight of Ultramarine Blue Tinting Pigment, g	Amount of Oil, mL
White Lead	2	0.2	0.5
Zinc oxide	2	0.2	0.7
Zinc oxide, leaded	2	0.2	0.5
Titanium dioxide (anatase)	2	0.4	1.0
(rutile)	2	0.4	0.9

Specification D 262 or a grade agreed upon by the purchaser and the seller.

6.2 Oil—Refined linseed oil with an acid number of approximately 4.

6.3 Reference Standard—A standard white pigment of the same type and grade as the pigment to be tested, as agreed upon between the purchaser and the seller.

7. Procedure A—Glass Hand Muller

7.1 Weigh 2 g of the standard white pigment and the amount of ultramarine blue listed in Table 1 to ± 2 mg. Transfer the weighed portions of white and blue pigments to the ground-glass plate. Add the amount of oil specified in Table 1 from the buret (Note). Be sure to allow the buret to drain to its true level because variations in the amount of oil decrease the precision of the test. Work the pigments and oil into a paste with the spatula; then rub up the paste with the glass hand muller, spreading it over an area 3 to 4 in. (75 to 100 mm) wide and from 12 to 15 in. (305 to 380 mm) long. In counting the rubs, one stroke up and one stroke back is considered one rub. Allow the muller to travel up one side and back the other side, twisting it slightly at the top and bottom of each stroke. After each 25 rubs with the muller, "pick up" the paste with the spatula by scraping the face of the muller and gathering the paste on the slab into a mound. Repeat until the paste has been given 100 rubs.

NOTE 2—Where the resulting paste is too fluid or too thick for mulling, adjust the quantity of oil to give a workable paste and prepare a new paste, mulling as before. Report the amount of oil used.

7.2 Work 2 g of the test pigment in exactly the same manner as prescribed in 7.1 for the standard, using the same amount of tinting material and oil.

7.3 Spread the paste of the standard and test pigments in parallel contiguous rectangular areas on the panel, each about 1 in. (25 mm) wide and 2 in. (50 mm) long. Use the scraper to smooth the surface of the pastes (here called "draw-downs") by drawing the scraper lightly over the pastes to give a straight and even line of contact between them. Keep the draw-downs sufficiently thick to obscure the panel.

7.4 Immediately examine the draw-downs of the two

pastes, on the top side only, for relative lightness of tint. If the sample is lighter than the reference standard it has greater tinting strength. If the sample differs appreciably from the reference standard in lightness of tint, and if a numerical rating is desired, prepare other pastes of the reference standard white pigment using different amounts of the tinting material. Make a draw-down of the sample paste with each standard as described in 7.3. Select the draw-down in which the standard pigment paste most closely matches the test pigment paste in lightness. Use the weight of tinting material in this method to calculate the tinting strength of the test pigment.

8. Procedure B—Automatic Muller

8.1 In order to minimize the effect of the difference in grinding of the area near the center of the plates as compared to the area near the periphery of the plates, draw two concentric circles under the base plate of the muller in such a way that they show clearly through the plate. These circles can be drawn either on a paper inserted under the plate, or drawn directly on the bottom of the plate. The inner circle should be 2 1/4 in. (63 mm) in diameter and the outer circle 4 1/2 in. (115.3 mm) in diameter.

8.2 Carefully weigh the pigment as described in 7.1, transferring the pigment to the base plate of the muller. Add the oil and work the pigment and oil into a paste with the spatula. Distribute the paste within the area between the two concentric circles on the plate, close the muller, and add a 50-lb (23-kg) weight (to make a total weight of 100 lb (45 kg)) pressing the plates together. Carry out three mulling stages of 50 revolutions each, collecting the paste from both plates with the spatula after each stage, spreading it around the path on the lower plate and wiping the spatula on the upper plate as before.

8.3 Continue the treatment of the paste of the test and standard pigments as described in 7.2, 7.3, and 7.4.

9. Calculation

9.1 Calculate the tinting strength of the sample as follows:

$$TS = (B/A) \times T$$

where:

TS = tinting strength of the test pigment, lbf (or N),
 A = weight of tinting material used with standard to give equality of tint, g,
 B = weight of tinting material used with test sample, g, and
 T = arbitrary tinting strength value given to the standard.

10. Precision and Bias

10.1 The precision and bias are to be determined.

11. Keywords

11.1 tinting strength; white pigment strength

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Standard Specification for Shellac Varnishes¹

This standard is issued under the fixed designation D 360; the number immediately following the designation indicates the year of original adoption 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 covers shellac varnish consisting of a solution or "cut" of a specified type and grade of dry lac resin in specially denatured alcohol. Ninety-five percent (95 proof) ethyl alcohol, specially denatured according to Formula No. 1 of the U. S. Bureau of Internal Revenue, is the solvent most commonly used. However, other specially denatured alcohols and proprietary solvents are also used for this purpose.

Referenced Documents

2.1 ASTM Standard:

D 29 Test Methods for Sampling and Testing Lac Resins²

D 207 Specification for Dry Bleached Lac²

D 237 Specification for Orange Shellac and Other Lacs²

3. Types, Grades, and Bodies

3.1 This specification covers two grades, A and B, of orange shellac varnish and two grades, regular and refined, of bleached lac varnish. Each type and grade may be furnished in very light, light, medium, heavy, very heavy, or extra heavy body varnish, as specified.

4. Color

4.1 The color of shellac varnish shall be no darker than the color of a standard varnish of the same nonvolatile matter content mutually agreed upon between the purchaser and the seller.

NOTE 1—Attention is called to the fact that the purchaser and the seller must agree upon one of the two methods for determining color appearing in Test Methods D 29.

5. Drying Time

5.1 Shellac varnish shall dry hard in 1 h. Shellac varnishes having a body greater than that for a light body varnish shall be thinned to the nonvolatile matter content of a light body

varnish. Special conditions for this test may be made by mutual agreement between the purchaser and the seller.

6. Body and Cut

6.1 The body and cut of the respective types and grades shall be based on the percent of nonvolatile matter, and shall conform to the following minimum requirements:

	Nonvolatile Matter, min, %	
	Orange, Grades A and B	Bleached, Regular and Refined
Very light body (3-lb (1.4-kg) cut) varnish	28.5	28.0
Light body (4-lb (1.8-kg) cut) varnish	35.0	34.5
Medium body (4.5-lb (2.0-kg) cut) varnish	37.5	37.0
Heavy body (5-lb (2.3-kg) cut) varnish	40.0	39.5
Very heavy body (6-lb (2.7-kg) cut) varnish	44.0	43.5
Extra heavy body (8-lb (3.6-kg) cut) varnish	51.0	50.5

NOTE 2—The term "cut" is defined as the number of pounds of dry lac resin (containing only that amount of moisture acceptable for proper cutting with the solvent to ensure a satisfactory varnish) that were added to 1 gal (3.8 L) of specified solvent in manufacturing the varnish.

Allowance has, therefore, been made for 2 % maximum moisture and other volatile matter and 3 % insoluble matter in orange shellac, and for 6 % moisture and other volatile matter in bleached lac, in arriving at a minimum value. Varnishes of the above specified bodies or "cuts" should accordingly conform to the respective minimum requirements specified for nonvolatile matter.

7. Properties of Dry Lac

7.1 The nonvolatile matter in the varnish shall conform to the Properties section of Specification D 207 and Table 1 of Specification D 235.

8. Packaging and Marking

8.1 Shellac varnish shall be packaged in standard commercial containers of the type and size mutually agreed upon between the purchaser and the seller. The label of the container shall show the type, grade, body or cut, and date of manufacture of the varnish contained therein.

9. Test Methods

9.1 The sampling and methods of testing shall be conducted in accordance with Test Methods D 29.

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

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² Annual Book of ASTM Standards, Vol 06.02.

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Standard Test Methods for Soluble Nitrocellulose Base Solutions¹

This standard is issued under the fixed designation D 365; the number immediately following the designation indicates the year of original adoption 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, including the title, in March 1989.

Scope

1.1 These test methods cover the testing of soluble nitrocellulose base solutions that are made by dispersing various grades and concentrations of soluble nitrocellulose (cellulose nitrate) in various solvent mixtures.

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 11.*

Referenced Documents

- 2.1 *ASTM Standards:*
 D 301 Test Methods for Soluble Cellulose Nitrate²
 D 333 Test Methods for Clear and Pigmented Lacquers³
 D 1193 Specification for Reagent Water⁴
 D 1200 Test Method for Viscosity by Ford Viscosity Cup³
 E 300 Practice for Sampling Industrial Chemicals⁵

Significance and Use

3.1 Since the desired specifications and compositions of soluble nitrocellulose base solutions vary greatly, these methods are used to establish whether limits that shall be as agreed upon between the producer and the user have been met.

Sampling

4.1 Select the sampling method from those listed in Practice E 300.

CONSISTENCY (VISCOSITY)

5. Consistency Tests

5.1 *For Consistencies from 3 to 500 s*—Determine the consistency by falling-ball consistency test described in Method D 301 for those solutions having a consistency from 3 to 500 s when tested in that apparatus.

5.2 *For Consistencies Less than 3 s*—Determine the consistency by Test Method D 1200 for those solutions having a consistency of less than 3 s when tested in the falling-ball apparatus referred to in 5.1.

5.3 *For Consistencies over 500 s*—Determine the consistency using the apparatus and procedure described in Sections 6 and 7 for those solutions having a consistency greater than 500 s when tested in the falling-ball apparatus referred to in 5.1.

6. Apparatus

6.1 The consistency test apparatus, shown in Fig. 1, shall consist of the following:

6.1.1 *Glass Tube* (preferably heat-resistant glass), $2 \pm \frac{1}{32}$ in. (50 ± 1.5 mm) in inside diameter and 10 in. (255 mm) in length, with marks $5 \pm \frac{1}{16}$ in. (177 ± 1 mm) apart, the upper one being 3 in. (75 mm) from the top of the tube.

NOTE 1.—The steel ball can be removed (in order to leave the same material in the tube for a check run) by removing the lower stopper. However, a small air bubble is usually introduced in this way. It is preferable to invert the tube, removing the guide to get the ball out. It is often necessary to put a few drops of solvent in the guide lip to loosen it from the tube on account of the solution drying at the edge of the tube. When the latter method is used for removing the ball, a larger bubble traverses the tube than when the former method is used, but a large bubble moves sufficiently fast, even in a very viscous solution, to escape at the top in a few minutes, whereas small bubbles take hours to escape.

6.1.2 *Steel Ball*, 0.625 ± 0.001 in. (15.88 ± 0.02 mm) in diameter, and weighing 16.536 ± 0.10 g.

6.1.3 *Aluminum Guide Cone* of light gage aluminum (approximately 0.02 in. (0.5 mm) in thickness) as shown in Fig. 1. The orifice of the guide cone shall be $\frac{7}{8}$ in. (22 mm) in diameter, the conical portion 1 in. (25 mm) in height, the cylindrical portion $\frac{1}{2}$ in. (12.7 mm) in height, and the outside diameter shall be slightly under 2 in. (50 mm) so as to fit snugly into the viscosity tube.

6.1.4 *Stoppers*, made preferably of rubber and covered with tin foil.

7. Procedure

7.1 Fill the tube in any convenient manner whereby bubbles do not form and no appreciable amount of solvent is lost. One method is to immerse the lower end of the open tube in the solution and to apply suction at the upper end of the tube. In this manner the tube can be filled in from 5 to 10 s without the introduction of air bubbles or an appreciable

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

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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 06.03 and 11.01.

⁵ Annual Book of ASTM Standards, Vols 06.03 and 15.05.

⁶ Borosilicate glass is satisfactory for this purpose.

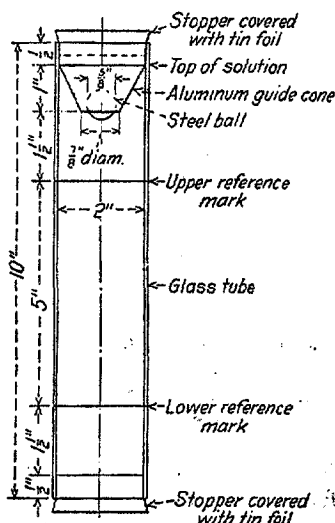


FIG. 1 Apparatus for Consistency (Viscosity) Test of Solutions Having Consistencies over 500 s

loss of solvent. Close the bottom of the tube with a stopper covered with tin foil. Push the aluminum guide cone slowly into the top of the tube and insert a stopper covered with tin foil into the top of the guide.

7.2. Bring the tube and its contents to a temperature of $25 \pm 0.1^\circ\text{C}$ by placing in a suitable bath. Allow at least 30 min for the solution to reach temperature equilibrium. For accurate measurements keep the tube during the determination either in a thermostat or suspended within a considerably larger cylinder of water at the specified temperature.

7.3 Remove the upper stopper only long enough to place the ball in the center of the tube; this can be done conveniently with crucible tongs. The principal value of the guide cone is to retard the ball sufficiently at the start of its fall so that the solution will close over the ball and not leave on the top of the ball a large "trailer bubble." If the guide is not used a big trailer bubble usually accompanies the ball, retarding its fall and, if the bubble is off center on the ball, pulling the latter away from the center of the tube.

7.4 Measure the time of fall in seconds from the instant the bottom of the ball is level with the upper reference mark on the tube until it reaches the lower mark on the tube.

NONVOLATILE MATTER

8. Procedure

8.1 Determine the percent of nonvolatile matter in accordance with the procedure described in the Nonvolatile Matter section of Test Methods D 333.

APPEARANCE OF SOLUTION

9. Procedure

9.1 Compare the appearance of the soluble nitrocellulose base solution with a reference standard agreed upon between the purchaser and the seller (both the reference standard and sample solutions shall be thoroughly agitated before making

observations) in similar bottles for turbidity, hair, grain, and insoluble matter.

DEPTH OF COLOR

10. Apparatus

10.1 The apparatus used for the preparation of the color standards and for the depth of color determinations shall consist of the following:

10.1.1 *Light*—Source of transmitted light.

NOTE 2—Not absolutely necessary but will increase accuracy and be more convenient.

10.1.2 *Bottles*, three dozen 2-oz (60 mL) screw-cap, square, glass.

10.1.3 *Flasks*, several, 1-L, volumetric.

10.1.4 *Burets*, two, 50-mL.

10.1.5 *Analytical Balance*.

10.1.6 *Colorimeter*—Dubosq colorimeter (see Note 2).

10.1.7 *Color Glass*—Yellow glass about 25 mm square and 10 mm thick.⁷

11. Reagents and Materials

11.1 *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.

11.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

11.3 *Potassium Chloroplatinate*, (K_2PtCl_6).

NOTE 3: *Warning*—This material is hazardous. Carefully review Material Safety Data Sheets supplied by manufacturers for handling and first aid instruction.

11.4 *Cobalt Chloride* ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$). (Warning—See Note 3.)

11.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). (Warning—See Note 3.)

11.6 *Caramel* (sugar coloring).

11.7 *Phenol*. (Warning—See Note 3.)

12. Preparation of Color Standards

12.1 *Platinum-Cobalt Color Standards*—Prepare the platinum-cobalt color standards as follows: Weigh out on an analytical balance exactly 1.245 g of K_2PtCl_6 and 1.000 g of crystallized cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$). Dissolve in distilled water, add 100 mL of HCl (sp gr 1.19), and dilute to 1000 mL with distilled water. This solution is color standard No. 10.⁹ Prepare color standards Nos. 1 to 10, inclusive, by

⁷ Corning glass, signal yellow No. 330 has been found satisfactory and is available from the Corning Glass Works, Corning, NY.

⁸ "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, NY., and the "United States Pharmacopeia."

⁹ This solution has an assigned value of 500 on the Hazen color scale (see *American Chemical Journal*, Vol XIV, p. 300).

TABLE 1 Platinum-Cobalt Color Standards

Platinum-Cobalt Color Standards	Quantity of No. 10 Color Standard, mL	Quantity of Distilled Water, mL
No. 1	5	45
No. 2	10	40
No. 3	15	35
No. 4	20	30
No. 5	25	25
No. 6	30	20
No. 7	35	15
No. 8	40	10
No. 9	45	5
No. 10	50	0

accurately measuring from burets directly into the square 2-oz (60-mL) bottles the quantities shown in Table 1 of the color standard and distilled water. After preparation of the color standards seal the bottles with corks and paraffin before putting on the screw caps. The platinum-cobalt color standards are permanent for approximately six months. Make the color standards from No. 12 to No. 500, inclusive, from a caramel solution, but the color is based on the platinum-cobalt color standard.

12.2 Caramel Color Standards—Prepare the caramel color standards as follows: Dilute caramel (sugar coloring) in the ratio of about 1 mL of caramel to 100 mL of distilled water in a glass vessel. Add 0.5 % phenol and agitate thoroughly. Adjust the concentration of this solution so that when it is diluted in the ratio of 1 mL of solution to 49 mL of distilled water, to which has been added 0.5 % phenol, the solution will match color No. 10 of the platinum-cobalt color standard. This caramel solution before dilution is color No. 500 (Note 4). Prepare color standards No. 12 to No. 500, inclusive, by accurately measuring from burets directly into the square 2-oz (60-mL) bottles the quantities shown in Table 2 of color No. 500 caramel standard and distilled water to which has been added 0.5 % phenol. After preparation of the color standards, seal the bottles with corks and paraffin before putting on the screw caps. All of the caramel color standards are permanent for approximately one month but should be checked semi-monthly if frequently used.

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TABLE 2 Caramel Color Standards

Caramel Color Standards	Quantity of No. 500 Caramel Color Standard, mL	Quantity of Distilled Water plus Phenol, mL
No. 12	1.2	48.8
No. 15	1.5	48.5
No. 20	2.0	48.0
No. 25	2.5	47.5
No. 30	3.0	47.0
No. 35	3.5	46.5
No. 40	4.0	46.0
No. 45	4.5	45.5
No. 50	5.0	45.0
No. 60	6.0	44.0
No. 70	7.0	43.0
No. 75	7.5	42.5
No. 80	8.0	42.0
No. 90	9.0	41.0
No. 100	10.0	40.0
No. 125	12.5	37.5
No. 150	15.0	35.0
No. 175	17.5	32.5
No. 200	20.0	30.0
No. 250	25.0	25.0
No. 300	30.0	20.0
No. 350	35.0	15.0
No. 400	40.0	10.0
No. 450	45.0	5.0
No. 500	50.0	0.0

NOTE 4—It is more convenient to check the caramel color standard No. 500 against a colored glass that has been previously standardized against the platinum-cobalt color standard; a piece of signal yellow glass (see 10.1.7) about 25 mm square by 10 mm thick may be standardized and used as a check on the caramel solution. This must be done by use of a colorimeter.

13. Procedure

13.1 Fill a square 2-oz (60-mL) glass bottle with the material to be tested and match this against one of the color standards using either direct daylight or indirect artificial transmitted light, the latter being preferred. The number of the color standard matched is the depth of color of the material.

14. Precision and Bias

14.1 The precision and bias of these test methods is as described for each of the separate methods cited if available.



Standard Test Method for Color and Strength of Color Pigments with a Mechanical Muller¹

This standard is issued under the fixed designation D 387; the number immediately following the designation indicates the year of original adoption 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 4220.1, 4221 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 This test method is intended to be used to compare the color and strength of a pigment under test with a reference standard of the same type and grade.

1.2 This test method does not apply to white pigments.

NOTE 1—Test Method D 3022 is similar to this test method, but it utilizes a miniature sandmill rather than a mechanical muller, to disperse the color pigment.

NOTE 2—Test Method D 332 and Test Method D 2745 are similar to this test method, but they are intended for use with white pigments, rather than color pigments.

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. Specific hazard statements are given in Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

- D 332 Test Method for Relative Tinting Strength of White Pigments by Visual Observation²
- D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials³
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
- D 2745 Test Method for Relative Tinting Strength of White Pigments by Reflective Measurements²
- D 3022 Test Method for Color and Strength of Color Pigments by Use of a Miniature Sandmill²
- D 3964 Practice for Selection of Coating Specimens for Appearance Measurements⁴
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry³
- E 308 Test Method for Computing the Colors of Objects by Using the CIE System⁵

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 Aug. 26, 1986. Published October 1986. Originally published as D 387 - 34 T. Last previous edition D 387 - 81.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01 and 14.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 14.02.

3. Summary of Test Method

3.1 Pigments are dispersed in a suitable vehicle with a mechanical muller. Test and standard pigments are treated identically. Opaque drawdowns are made from the dispersions and compared, either visually or instrumentally, for color and strength differences.

4. Significance and Use

4.1 Color and tinting strength are the most important properties of a color pigment. This test method provides a means of testing these properties for quality control.

4.2 This test method is intended as a referee method so that such matters as the vehicle for preparing the dispersions and the white for making tints have been suggested. However, other vehicles and whites may be suitable for quality control purposes, and changes in this test method are allowed by agreement between the parties to a test.

4.3 It is assumed that the most exact comparison of mass color and tinting strength occurs when the pigment is completely dispersed. By following the procedure described in Annex A1, the conditions for achieving the maximum practical degree of dispersion with a mechanical muller may be determined. Color and strength tests should be carried out under these conditions.

4.4 The results obtained with a mechanical muller do not necessarily correlate directly with an industrial situation where different dispersing conditions exist. However, dispersion with a mechanical muller is a quick and inexpensive way of testing the color and strength of a pigment for routine quality control.

5. Apparatus

5.1 *Balances*—(1) A balance sensitive to 10 mg and (2) an analytical balance sensitive to 1.0 mg.

5.2 *Muller, Mechanical*,⁶ equipped with ground-glass plates to which a variable but known force may be added in 50-lbf (220-N) increments. The driven glass plate shall have a speed of rotation of between 70 and 120 rpm and the apparatus shall have an arrangement for pre-setting the number of revolutions in multiples of 50.

5.3 *Rubbing Surfaces*—The rubbing surfaces of the ground glass plates shall be kept sharp by removing them from the muller and grinding them face-to-face with No. 303 optical emery, or its equivalent, and water.

⁶ A satisfactory muller is supplied by the Hoover Color Corp., 13 Cordier St., Irvington, NJ 07111.

4.4 *Small Glass Slab* or other nonabsorbent material, suitable for weighing and mixing pigment pastes.

4.5 *Spatula*—A flexible spatula having a 3 to 6-in. (75 to 150 mm) blade.

4.6 *Paper Charts*,⁷ white with a black band and a surface impervious to paint liquids.

4.7 *Film Applicator*,⁸ at least 3 in. (75 mm) wide with a clearance of 4 mils (100 μ m) to produce wet films about 2 mils (50 μ m) thick.

4.8 *Color-Measuring Instrument*, meeting the requirements of Method D 2244.

5. Materials

5.1 *Reference Standard*—A standard pigment of the same type and grade as the pigment to be tested, as agreed upon between the purchaser and the seller.

5.2 *Vehicle*—A solvent-free vehicle, such as No. 1 lithographic varnish, with 0.8 % each of cobalt and manganese driers (6 % types).

5.3 *White Tinting Paste*—A white paint compatible with the dispersion vehicle, such as 57 parts of rutile titanium dioxide dispersed in 43 parts of the vehicle described in 5.2.

NOTE 3—Because the choice of vehicle and white tinting pigment may affect the results, they should be agreed upon between the purchaser and the seller.

6. Hazards

6.1 Some pigments may be potentially toxic and therefore should be handled with care. Obtain specific precautions from the manufacturer or supplier.

6.2 Many solvents and paint vehicles present explosion, fire, and toxicity hazards, and they should accordingly be handled with care. Again, obtain specific precautions from the manufacturer or supplier.

7. Dispersing Conditions

7.1 The conditions for dispersing the pigment on the mechanical muller should be such that the maximum tinting strength is developed. For each pigment and each dispersing vehicle the development of tinting strength by the mechanical muller is influenced by the force applied, the number of revolutions, the mass of the pigment, and the mass of the vehicle. The conditions for obtaining the maximum tinting strength with the mechanical muller can be determined by following the procedure in Annex A1.

7.2 If these conditions are known for a particular pigment with a particular vehicle, or if the purchaser and seller agree upon a particular set of conditions, there is no need to carry out the procedure in Annex A1.

8. Dispersion Procedure

8.1 Decide, by agreement or by experimentation, as discussed in Section 8, the following dispersing conditions:

8.1.1 Force applied to the muller plates;

8.1.2 Number of revolutions;

8.1.3 Mass of the pigment; and

8.1.4 Mass of the vehicle.

8.2 Applying these decisions, prepare a dispersion of the reference standard pigment. Weigh onto a glass slab to within 2 mg, the appropriate quantities of the standard pigment and the dispersing vehicle. Mix the pigment and vehicle together thoroughly with the spatula and transfer the mixture to the lower plate of the muller. Spread the mixture in a path approximately 100 mm wide and halfway between the center and rim of the lower plate, and clean the spatula as much as possible by wiping it on the upper plate of the muller. Close the plates and carry out the mulling stages of 50 revolutions; after each stage collect the paste from both plates with the spatula and spread it around the 100-mm path on the lower plate, wiping the spatula on the upper plate as before. When the mulling has been carried out for the prescribed number of revolutions, collect the paste and store it. Clean the glass slab, the muller plates, and the spatula, and repeat the procedure with exactly the same quantities of the test sample and vehicle. Collect the paste from this sample and store it. Clean the glass slab, the muller plates, and the spatula.

NOTE 4—The most common sources of error in this procedure are inaccurate weighing, incomplete transfer of the pigment and vehicle mixture, and contamination of the plates by previous samples.

9. Masstone Color Procedure

9.1 Draw down a portion of the test and standard pastes in juxtaposition on a paper chart over a vacuum-drawdown plate or other suitable plane surface with the film applicator. Make sure that the coating is opaque.

9.2 Immediately compare the colors visually while still wet, using Practice D 1729, and record the results. Set the drawdowns aside in a dust-free area to dry. When dry repeat the visual color difference evaluation and record the results. See Practice D 3964.

9.3 If desired, evaluate the color difference instrumentally using Method D 2244, and report the color difference in units as agreed upon between the purchaser and seller.

NOTE 5—Wet color difference evaluations may not agree with dry color difference evaluations because of such phenomena as flooding and flocculation. In the case of a difference between the wet and dry evaluations, the purchaser and the seller should agree upon which condition is the standard.

NOTE 6—Color difference measurements of wet paints may require a special adapter to protect the instrument from fouling. Because color difference-measuring instruments differ widely in their design, the user may have to develop his own adapter.

10. Tint Color Procedure

10.1 Determine by calculation the amount of white pigment paste that must be added to 0.5 g of the color pigment paste so that the mixture contains 1 part of dry color pigment to 10 parts of dry white pigment. For stronger or weaker pigments this ratio may be adjusted accordingly, for example, 1:20 or 1:5, respectively.

10.2 Weigh 500 ± 2 mg of the standard color pigment paste onto a glass slab. Then weigh the amount of white pigment paste determined in 10.1, and place it next to the color pigment paste on the glass slab. Thoroughly mix the two pastes together with the spatula until a uniform color is observed.

⁷ White charts with a black band are preferred for judging opacity. Satisfactory charts are available from the Morest Co., 211 Centre St., New York, NY 10073, and the Leneta Co., P.O. Box 576, Ho-Ho-Kus, NJ 07423.

⁸ Satisfactory film applicators are available from Bird and Son, Walpole, MA 02081 and Precision Gage & Tool Co., 28 Volkenand Ave., Dayton, OH 45410.

11.3 Prepare a tint mixture of the test color pigment paste and the white pigment paste on a separate glass slab by the procedure described in 11.2.

11.4 Draw a portion of the test and standard tint pastes down in juxtaposition on a paper chart as in 10.1. Evaluate the color difference visually as in 10.2 and, if desired, instrumentally as in 10.3. Clean the spatula blade and glass slabs.

12. Calculation of Tinting Strength

12.1 If the colors of the test tint paste and the standard tint paste are visually the same, the tinting strength of the test pigment is equal to that of the standard pigment, and the relative tinting strength of the test pigment is 100 %. However, if the test and standard colors are not the same, the difference may be due to tinting strength or hue (shade).

12.2 To determine the relative tinting strength of the test pigment, repeat the operations of Section 11, but this time use an amount of the test pigment paste that is estimated to give the closest color match to the standard pigment paste. Repeat this procedure until satisfied that the closest color match has been obtained. At this point any residual color difference between the test and the standard pigments is attributed to a shade difference, rather than a strength difference. Note and record this shade difference.

12.3 Calculate the relative tinting strength of the test pigment by dividing the mass of the standard paste by the mass of the test paste used to obtain the closest color match; multiply by 100 to express the result in percent.

12.4 If desired, the relative tinting strength of the sample pigment can be calculated from instrumental measurements (see Test Method E 97 or Practice E 308) using the following equation:

$$TS = [(1 - R_{\infty})^2 / 2R_{\infty}]_u / [(1 - R_{\infty})^2 / 2R_{\infty}]_s (T)$$

where:

TS = tinting strength of test pigment,

R_{∞} = measured reflectant factor (as a decimal),

T = assigned tinting strength of standard, usually 100 %, and subscripts "u" and "s" refer to the test and standard pigments, respectively.

12.4.1 For R_{∞} use the lowest reflectance value measured in the range 420 to 680 nm with a spectrophotometer or abridged spectrophotometer. This method of calculation is not valid when any tristimulus value or filter colorimeter reading is substituted for R_{∞} unless it can be demonstrated

that with such a substitution the function $(1 - R_{\infty})^2 / 2R_{\infty}$ still varies linearly with concentration for the pigments being tested over the range of concentrations of interest. Because the use of different parameters may give significantly different results for the tinting strength, the choice of the parameter to be used as R_{∞} shall be agreed on between the purchaser and the seller.

NOTE 7—This method is not applicable to metameric specimens, or to specimens exhibiting large shade differences, or to specimens differing in strength by more than about 30 %.

13. Report

13.1 Report the following information:

13.1.1 Type and identification of the test pigment, reference standard pigment, white tinting pigment, and dispersing vehicle.

13.1.2 Mass ratio of pigment to vehicle, and for tints mass ratio of color pigment mass to white pigment.

13.1.3 Manufacturer and model number of the mechanical muller employed.

13.1.4 Total force applied to the muller plates and total number of revolutions.

13.1.5 Results of the visual evaluation of the color difference (masstone and tint) in accordance with Practice D 1729.

13.1.6 If an instrument was used to evaluate the color difference, the results of the instrumental evaluation in accordance with Method D 2244.

13.1.7 Relative tinting strength and method by which it was determined (visual or instrumental). Also, for the instrumental method, the parameter used as the measure of R_{in} .

13.1.8 Any deviation, by agreement or otherwise, from the test procedure described above.

14. Precision

14.1 The precision of this test method depends on several factors such as the type of pigment, the level of tinting, and the magnitude and direction of the color difference. This point is illustrated by the results in Table 1(a), which contains the between-laboratories standard deviations obtained in an interlaboratory study involving five different laboratories and four different pigments. The dispersing conditions used to obtain these results are listed in Table 2.

14.2 Table 1(b) lists the maximum acceptable differences, calculated at the 95 % confidence level from the results in Table 1(a).

TABLE 1(a) Between-Laboratories Standard Deviations for Various Color Difference^a and Tinting Strength^b Parameters

Pigment Type	Masstone Color				Tint Color				Tinting Strength			
	Δa	Δb	ΔL	ΔE	Δa	Δb	ΔL	ΔE	Y	T	R	V
Yellow Iron Oxide	0.10	0.46	0.22	0.42	0.13	0.20	0.19	0.08	2.0	2.2	3.0	0.7
BON Red	0.12	0.37	0.20	0.28	0.29	0.31	0.07	0.24	0.7	1.8	1.1	2.5
Molybdate Orange	0.09	0.14	0.06	0.11	0.11	0.12	0.05	0.14	0.5	0.8	0.8	1.0
Phthalocyanine Green ^c	0.29	0.65	1.43	0.51	0.25	0.07	0.13	0.21	1.6	1.8	2.6	1.8

TABLE 1(b) Maximum Acceptable Differences for Various Color Difference^A and Tinting Strength^B Parameters

Pigment Type	Masstone Color				Tint Color				Tinting Strength			
	Δa	Δb	ΔL	ΔE	Δa	Δb	ΔL	ΔE	Y	T	R	V
Yellow Iron Oxide	0.28	1.30	0.62	1.19	0.37	0.57	0.54	0.23	5.7	6.2	8.5	2.0
BON Red	0.34	1.05	0.57	0.79	0.82	0.88	0.20	0.68	2.0	5.1	3.1	7.1
Molybdate Orange	0.25	0.40	0.17	0.31	0.31	0.34	0.14	0.40	1.4	2.3	1.7	2.8
Phthalocyanine Green ^C	0.82	1.84	4.04	1.44	0.71	0.20	0.37	0.59	4.5	5.1	7.4	5.1

^A Color difference values were calculated with the CIE 1976 L*a*b* (CIELAB) equation.

^B Tinting strengths were calculated four different ways with the equation in 12.2: Y, based on Y tristimulus value; T, based on lowest tristimulus value; R, based on lowest reflectance factor between 420 nm and 680 nm; and V, based on visual observation.

^C Severe bronzing occurred with the masstone of this pigment (more in the batch than the standard), which probably affected the color difference measurements made with different types of instruments.

TABLE 2 Dispersing Conditions Used in Interlaboratory Study

Pigment type	Phthalocyanine Green	Yellow Iron Oxide	BON Red	Molybdate Orange
Force applied to the muller plates, lb (N)	100 (440)	100 (440)	100 (440)	100 (440)
Total number of revolutions	400 (8 × 50)	100 (2 × 50)	200 (4 × 50)	100 (2 × 50)
Mass of color pigment, g	0.75	1.0	0.6	2.0
Mass of dispersing vehicle, g	1.8	1.7	1.4	1.0

ANNEX

(Mandatory Information)

A1. DISPERSING CONDITIONS FOR MAXIMUM TINTING STRENGTH

A1.1 The following describes a test method for determining the conditions for achieving the maximum level of tinting strength with the mechanical muller.

A1.2 Determine the appropriate ratio of color pigment to dispersing vehicle by performing the following operations: Tare off the weight of a glass slab on a balance. Weigh 1.00 ± 0.01 g of the standard pigment on to the glass slab. Add dispersing vehicle to the pigment in small amounts and mix them together with the spatula. Keep adding the vehicle and mixing the paste until the pigment is completely wetted and a workable paste is obtained. At this point the consistency of the paste should be such that a dab of the paste will drop from the spatula when it is gently tapped with the finger. Weigh the paste, and subtract the mass of the pigment to determine the mass of the vehicle. Calculate the pigment to vehicle mass ratio. Repeat the operations described above for the test pigment.

A1.3 Determine the appropriate amount of pigment to use by estimating, to within 0.2 mL, the volume of that paste prepared in A1.2 that has the smallest pigment-to-vehicle mass ratio. Calculate the masses of pigment and vehicle needed to give a paste having a volume of about 2.0 mL. Round the amount of pigment down and the amount of vehicle up to the nearest 0.1 g.

A1.4 Apply 100 lbf (440 N) to the muller plates and prepare a tint of the standard pigment in accordance with the procedure in Sections 9 and 11. Use the amounts of the color pigment and dispersing vehicle determined in A1.3 and mull the paste for 100 revolutions in two stages of 50 revolutions each.

A1.5 Prepare three more specimens from the same sample following the procedure described in A1.4, but mull these specimens, in stages of 50 revolutions, for 200, 300, and 400 revolutions, respectively.

A1.6 Compare each of the four specimens, one to the other, for tinting strength using one of the methods described in Section 12, and determine the minimum number of revolutions necessary to achieve full tinting strength. If the tinting strength is still developing after 400 revolutions, repeat A1.4 to A1.6 with 50 lbf (220 N) more force on the mechanical muller plates.

A1.7 Record the appropriate amounts of pigment and vehicle (by A1.3), the force applied to the mechanical muller plates and the minimum number of revolutions required for maximum tinting strength.

A1.8 Table 2 lists, as examples, the dispersing conditions used in the interlaboratory study that established the precision given in Tables 1(a) and 1(b). The vehicle used was No. 1 lithographic varnish with 0.8 % each of cobalt and manganese driers (6 % types).

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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 Methods of Testing Shellac Used for Electrical Insulation¹

This standard is issued under the fixed designation D 411; the number immediately following the designation indicates the year of original adoption 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 methods cover tests for shellac in the dry or powdered form to be used for electrical insulating purposes.

1.2 The values stated in inch-pound units are to be regarded as the standard. The SI equivalents of inch-pound units may be approximate.

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 29 Test Methods for Sampling and Testing Lac Resins²
E 1 Specification for ASTM Thermometers³

3. General Tests

3.1 Each of the following tests shall be made in accordance with the procedures described in Test Methods D 29:

- 3.2 Sampling,
- 3.3 Insoluble Matter,
- 3.4 Iodine Number,
- 3.5 Moisture Content,
- 3.6 Wax,
- 3.7 Ash, and
- 3.8 Orpiment (Native arsenic trisulfide).

POLYMERIZATION TIME

4. Significance and Use

4.1 Polymerization time provides a measure of time during which shellac retains its plasticity and flow properties at the specified temperature before gelling or polymerizing to the tough rubbery insoluble form. This test is important in determining the quality of different lots, useful shelf-life, batch uniformity, and processing characteristics of shellac.

5. Apparatus

5.1 *Test Tubes and Wire Rack*—Two 18-mm ($\frac{3}{4}$ -in.) outside diameter glass test tubes, supported by a wire rack to a depth of 100 mm (4 in.) when placed in an oil bath. The

test tubes must be supported and maintained in a vertical position. The rack shall be constructed so as to permit free circulation of oil around the test tubes.

5.2 *Oil Bath*—An oil bath of such construction as to permit the maintaining of a test temperature of $150 \pm 1^\circ\text{C}$ ($300 \pm 2^\circ\text{F}$). It is essential to use a mechanical stirring device in the oil bath to maintain a uniform temperature. The oil used shall be a mineral oil, or its equivalent, having a Saybolt Universal viscosity of approximately 150 St ($0.015 \text{ m}^2/\text{s}$) at 100°C (212°F).

5.3 *Oil Bath Heater*—A thermostatically controlled heater capable of maintaining the oil bath temperature within the specified limits.

5.4 *Glass Rod*—A smooth glass rod about 3 mm (0.12 in.) in diameter and approximately 200 mm (8 in.) long. The end of the glass rod which is to be immersed in the shellac shall be fire polished to a smooth round end.

NOTE 1—A smooth glass rod about 5 mm (0.2 in.) in diameter and approximately 200 mm (8 in.) long with an indicator mark at the top to indicate movement may be used. Flatten slightly the diameter of the glass rod for approximately 3 mm (0.12 in.) length at the end which is immersed in the shellac. The purpose of the flattened rod is to provide a more positive feel of the twist back and rubbery character at the end point. If this alternative is used, it must be specified in the report.

5.5 *Thermometer*, suitable to indicate the test temperature.

5.6 *Stopwatch*, suitable to indicate elapsed time in minutes and seconds.

6. Conditioning

6.1 Roll samples of shellac on clean paper, mix well, and then carefully dry as described in 6.2 before testing.

6.2 For each shellac to be tested, evenly spread $9 \pm 0.1 \text{ g}$ of the prepared shellac in a flat bottom dish about 50 mm (2 in.) in diameter. Desiccate over anhydrous calcium chloride (CaCl_2) for 24 h at room temperature or dry in a well-ventilated convection oven at a temperature of $41 \pm 2^\circ\text{C}$ ($105 \pm 4^\circ\text{F}$) for approximately 16 h (or overnight).

6.3 After drying, immediately transfer the sample to a clean, dry, tightly stoppered bottle, and allow to cool. Do not open the bottle except when a specimen is being removed for test.

7. Test Specimens

7.1 Each test specimen shall consist of $4 \pm 0.01 \text{ g}$ samples of dried shellac as specified in 6.2.

7.2 Test two specimens of each shellac sample.

8. Procedure

8.1 Transfer a specimen of shellac from the stoppered

¹ These methods are under the jurisdiction of ASTM Committee D-9 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.01 on Electrical Insulating Varnishes, Powders, and Encapsulating Compounds.

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² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 14.03.

to the 18-mm (¾-in.) glass test tube. Insert the tube and hold securely in a vertical position in the test rack. Place the rack in the oil bath having a test temperature of $150 \pm 1^\circ\text{C}$ ($300 \pm 2^\circ\text{F}$) maintained throughout the test.

8.2 Record the time when the test tube enters the oil bath.

8.3 Using the glass rod, stir the specimen gently for up to the first 3 min of the test so that the shellac melts rapidly. It is essential that there be no stirring after the first 3-min period.

8.4 At the end of each minute thereafter, give the glass rod a slight twist (turning approximately 90°). In the early stages of the test, and before the polymerization point is reached, the glass rod will remain in the position to which it has been turned. When, however, the shellac takes on a rubbery set, there will be a definite turning or twisting back of the rod following the 90° twist. This is the end point.

NOTE 2—In the early stages of the test there may be a tendency for the glass rod to move back slowly after twisting with the fingers. This slight movement should not be confused with the definite "twist-back" observed when the end point is reached.

9. Polymerization Time

9.1 Record the elapsed time in minutes, from the time of entry of the specimen into the bath and including the 3-min stirring period, until the first "twist-back" of the glass rod is noted, as the polymerization time.

10. Report

10.1 The report shall include the following:

10.2 The polymerization time to the nearest 1 min for each specimen, and

10.3 The average of the values in 10.2.

FLOW TEST

11. Summary of Methods

11.1 These tests determine the flow of shellac when subjected to $100 \pm 1^\circ\text{C}$ ($212 \pm 2^\circ\text{F}$) under the conditions of test specified herein. These methods consist of melting a sample of ground shellac in a graduated test tube and then tilting the tube to the specified angle while maintained at $100 \pm 1^\circ\text{C}$ ($212 \pm 2^\circ\text{F}$) in order to permit the shellac to flow down the tube.

11.2 Two methods are provided as follows:

11.2.1 *Method A*—In this method the time required for the shellac to flow specified distances along the test tube is observed, and

11.2.2 *Method B*—In this method the total distance the shellac flows along the test tube in a specified time is observed.

12. Significance and Use

12.1 The rate at which shellac flows down an inclined plane under standard conditions can be used to determine the flow variation that occurs between different types, grades, or lots of shellac.

12.2 Flow tests are sensitive to atmospheric conditions; the flow being greater under humid conditions and less if desiccated. It is essential to carry out the test as quickly as

possible so as to minimize the errors due to the effects of atmospheric conditions.

13. Apparatus

13.1 *Method A*—A suitable form of testing apparatus for use in Method A is shown and described in the Annex.

13.2 *Method B*—A suitable form of testing apparatus for use in Method B is shown and described in the Annex. Any apparatus that will provide for accurately maintaining the required test temperature and the required positions of the test tubes may be used in Method B.

14. Test Specimens

14.1 Each test specimen shall consist of 2 ± 0.1 g of shellac, coarsely ground (approximately to pass a No. 20 sieve). The specimens shall be well spread out in a shallow vessel and placed in a desiccator over a saturated solution of sodium dichromate with an excess of solid salt and left in this atmosphere (52% relative humidity) at room temperature for at least 24 h. The specimens shall be tested immediately upon removal from the desiccator.

14.2 Two specimens shall be tested.

15. Preparation of Specimens

15.1 The two specimens of shellac shall be placed in separate glass test tubes with care being taken that the specimen in each tube is at the bottom and that none of the powdered shellac adheres to the walls of the glass tube. The top level of the dry shellac in each tube shall be read on the millimetre graduated scale. The tubes containing the specimens shall then be clamped in place in the testing fixture (see Section 13 and Fig. A1.1).

15.2 The testing fixture, with the glass test tubes in a vertical position, shall be inserted in the oil bath maintained at the test temperature of $100 \pm 1^\circ\text{C}$ ($212 \pm 2^\circ\text{F}$).

15.3 The top surface of the specimen in each tube should be level and at right angles to the walls of the tube.

15.4 The specimens shall be allowed to melt for 3 min.

METHOD A

16. Procedure

16.1 At the end of the 3-min melting period, place each test tube at an angle of 15° from the horizontal, with the corked ends down (see 13.1 and Fig. A1.2), and with the breather tube extending above the level of the oil bath. Make the change from the vertical position to the flow position as quickly as possible. With the oil bath maintained at the test temperature of $100 \pm 1^\circ\text{C}$ ($212 \pm 2^\circ\text{F}$), record the total time required for the shellac in each tube to flow from the initial level of the shellac to each centimetre marking along the tube. Discontinue the test in each tube when the flow is 9 cm (3.5 in.) or the total time is 20 min.

17. Report

17.1 The report of tests by Method A shall include the following:

17.2 Time required for each centimetre distance of flow for each specimen,

17.3 Plot of curve showing the data reported in 17.2 with time plotted as abscissas and flow, in centimetres, as ordinates,

17.4 Angle of the test tubes during the flow period, and

17.5 Atmospheric temperature and humidity of the laboratory.

METHOD B

18. Procedure

18.1 At the end of the 3-min melting period, place each test tube at an angle of 15° from the horizontal, with the corked ends down (see 18.2 and Fig. A1.2) and with the breather tube extending above the level of the oil bath. Make the change from the vertical position to the flow position as quickly as possible. With the oil bath maintained at the test temperature of $100 \pm 1^\circ\text{C}$ ($212 \pm 2^\circ\text{F}$), allow the test tubes to remain in the bath in this position for exactly 12 min.

18.2 Remove the test tubes immediately, place in a vertical position, cool, wipe, and measure the flow of the shellac in each tube by reading the distance between the initial point (18.1) and the end of the flow tongue. Disregard the "feather" at the very tip of the tongue.

NOTE 3—This "feather," caused by separation of wax from the shellac, is more noticeable with some shellacs than with others. It can readily be distinguished from the main body of shellac as it is always of a different color.

19. Report

19.1 The report of tests by Method B shall include the following:

19.2 Flow expressed in millimetres for each specimen,

19.3 Average of the values in 19.2,

19.4 Angle of the test tube during the flow periods, and

19.5 Atmospheric temperature and humidity of the laboratory.

20. Precision and Bias

20.1 Shellac is a naturally occurring resin and its chemical and physical properties are highly variable, and dependent upon a number of factors. A standard reference material has not been established.

20.2 Because of the unavailability of round-robin data, no statement can be made as to the precision and bias of these methods for determining polymerization time and flow of shellac.

ANNEX

(Mandatory Information)

A1. DESCRIPTION OF APPARATUS FOR DETERMINING THE FLOW OF SHELLAC

A1.1 The apparatus shown in Figs. A1.1 and A1.2 consist of the following:

A1.1.1 *Test Tubes*—Two test tubes, *A*, of the following types should be used:

A1.1.1.1 For use with Method A, the tubes should be preferably a red-lined, heat-resistant glass, 13 cm in length, 2.5 cm in outside diameter, and 1.5 mm in wall thickness. They should be graduated in 5-mm divisions beginning 1.1-cm from the outside bottom and extending upward to 100 mm. Every 10-mm line should be numbered. These tubes are to be used for holding the specimen, *S*.

A1.1.1.2 For use with Method B, plain heat-resistant glass tubes 12.5 cm in length and 2.5 cm in outside diameter.

The test tubes should be stoppered with tightly fitting corks through which extend small breather tubes, *B*.

A1.1.2 *Support*—A fixture *C* for holding the glass tubes in the correct position. This consists of two disks of brass supported as a pendulum as indicated. The disks are free to turn on the supporting shaft and are held in the desired position by the pin *D*. The tubes are supported on narrow *V*

blocks, and held in position by coil springs *H*, attached to disks *C*.

A1.1.3 *Oil Bath*—A metal bath *E*, heated by a bunsen burner or an electric immersion heater. Oil having a Saybolt Universal viscosity of approximately 150 s at 212°F (100°C) is suitable. Glycerol or a clear oil should be used where visual observations during the flowing period are required as in Method A. It is essential to use a mechanical stirring device *F*, to maintain a uniformly distributed temperature. The use of a glass tank or container with an immersion heater is required for Method A, where it is desired to observe the flow during the test and to record the time for the shellac specimen to flow various stated distances. For Method B any suitable container may be used.

A1.1.4 *Thermometer*—A thermometer *G*, to indicate the test temperature. The ASTM Partial Immersion Thermometer graduated in either Celsius or Fahrenheit degrees as specified, the range being -5 to $+300^\circ\text{C}$ or $+20$ to $+580^\circ\text{F}$, and conforming to the requirements for Thermometer 2C or 2F respectively, as prescribed in Specification E 1, is satisfactory for this purpose.

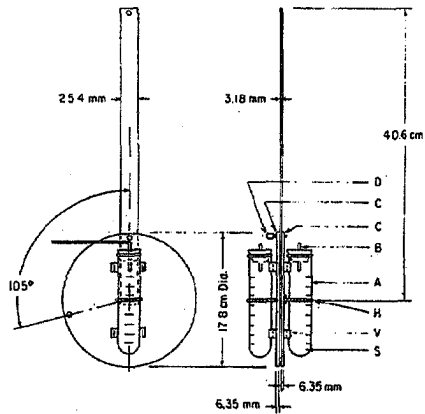


FIG. A1.1 Flow Test Fixture Showing Essential Parts with Test Tubes in Vertical Position

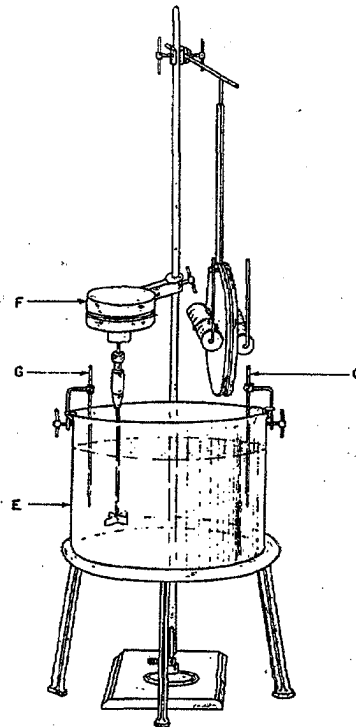


FIG. A1.2 Apparatus Arranged with Tubes in Inclined Position for Immersion in Bath for Flow Test

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 Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow)¹

This standard is issued under the fixed designation D 444; the number immediately following the designation indicates the year of original adoption 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 7211 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 These test methods cover procedures for the chemical analysis of the pigment known commercially as "zinc yellow" or "zinc chromate yellow."

1.2 The analytical procedures appear in the following order:

	Sections
Moisture and Other Volatile Matter	7
Combined Water	8
Chromium:	
Dichromate Method	9 to 11
Thiosulfate Method	9, 12, and 13
Zinc:	
Hydroxyquinoline Method	9, 14, and 15
Ferrocyanide Method	9, 16, and 17
Alkaline Salts	18 and 19
Sulfates	20 and 21
Chlorides	22 and 23
Matter Insoluble in Dilute Acetic Acid	24
Coarse Particles	25

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 Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

D 478 Specification for Zinc Yellow (Zinc Chromate)³

D 1193 Specification for Reagent Water⁴

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

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 D 01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 444 - 37. Last previous edition D 444 - 81(1987)¹.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

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 14.02.

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁶

3. Significance and Use

3.1 This test method has been developed to standardize the chemical analysis of zinc chromate yellow pigment and to provide alternate methods of analysis for chromium and zinc.

4. Preparation of Sample

4.1 Mix the laboratory sample thoroughly. Take a sufficient quantity for the chemical analyses and pass it through a 180- μ m (No. 80) sieve, grinding in a mortar if necessary.

NOTE 1—Detailed requirements for this sieve are given in Specification E 11.

5. Reagents

5.1 *Purity of Reagents*—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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water for use in the preparation of reagents and in analytical procedures shall conform to Type II reagent water, in Specification D 1193.

6. Precision

6.1 Precision statements have not been established.

MOISTURE AND OTHER VOLATILE MATTER

7. Procedure

7.1 Determine moisture and other volatile matter in accordance with Test Method A of Test Method D 280.

⁶ Annual Book of ASTM Standards, Vol 03.05.

⁷ "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."

COMBINED WATER

Procedure

8.1 Weigh to 0.1 mg 3 g of the oven-dried material from the determination of moisture and other volatile matter (Notes 2 and 3), and place in a porcelain boat. Introduce the boat with the charge into a refractory combustion tube in an electrically-heated combustion furnace of the type used for the determination of carbon in steel by direct combustion (Note 4). Place the boat at the center of the combustion tube maintained at a temperature of 1000°C for 4 h. Draw a current of pure dry air or dry nitrogen through the tube to sweep the evolved moisture into a previously weighed absorption tube containing anhydrous magnesium perchlorate ($\text{Mg}(\text{ClO}_4)_2$) or other efficient desiccant. The weight increase of the absorption tube represents the "combined water."

NOTE 2—Loss on ignition of the pigment does not suffice for the determination of combined water in zinc yellow.

NOTE 3—If the pigment contains an organic treating agent, first remove this treating agent by washing with ether or chloroform.

NOTE 4—See Apparatus No. 1 for the determination of total carbon by direct combustion as described in Practices E 50.

SPECIMEN SOLUTION FOR DETERMINATION OF CHROMIUM AND ZINC

9. Procedure

9.1 Weigh to 0.1 mg about a 4-g specimen and mix with 50 mL of cold sulfuric acid (H_2SO_4) (1+5). It should dissolve completely at this stage (Note 5). Dilute the solution to 500 mL in a volumetric flask.

NOTE 5—A cloudy solution may result if the pigment contains a surface-treating agent. In this case, it usually can be cleared by cooling in an ice-bath and filtering through a medium porosity fritted disk. If the solution is not clarified by this treatment, extract a portion of the original sample with a solvent such as chloroform before the analysis is begun.

CHROMIUM BY THE DICHROMATE TEST METHOD

10. Reagents

10.1 *Ferrous Ammonium Sulfate Solution*—Dissolve 80 g of ferrous ammonium sulfate ($\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$) in 50 mL of H_2SO_4 (sp gr 1.84) and enough water to make 1 L of solution. Mix thoroughly before use. This solution is approximately 0.2 N.

10.2 *Ortho-Phenanthroline Indicator Solution*—0.5 % in water.

10.3 *Potassium Dichromate, Standard Solution* (0.1 N)—Dissolve 4.9035 g of dried potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in water and dilute to 1 L in a volumetric flask.

11. Procedure

11.1 Pipet 50 mL of the solution of the specimen (Section 9) into a 600-mL beaker. Add 200 to 250 mL of water and 10 mL of H_2SO_4 (sp gr 1.84). Cool to 35°C or below.

11.2 Add an excess of ferrous ammonium sulfate solution and back-titrate with 0.1 N $\text{K}_2\text{Cr}_2\text{O}_7$ solution, using orthophenanthroline indicator. Carry out a blank titration of the same amount of ferrous ammonium sulfate solution at the same time and in the same manner.

11.3 *Calculation*—Calculate the percent of chromium C as CrO_3 , as follows:

$$C = (B_1 - V_1)N_1 \times 0.03334/S_1 \times 100$$

where:

B_1 = $\text{K}_2\text{Cr}_2\text{O}_7$ solution required for titration of the blank, mL,

V_1 = $\text{K}_2\text{Cr}_2\text{O}_7$ solution required for back-titration of the specimen, mL,

N_1 = normality of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution, and

S_1 = specimen in the aliquot used, g.

0.03334 = milliequivalents weight of CrO_3 .

11.4 Alternatively, the solution of the specimen may be titrated directly with the ferrous ammonium sulfate solution, using an electrometric titration assembly to detect the end point. Standardize the ferrous solution against $\text{K}_2\text{Cr}_2\text{O}_7$.

CHROMIUM BY THE THIOSULFATE TEST METHOD

12. Reagents

12.1 *Potassium Iodide Solution* (150 g/L)—Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 L.

12.2 *Sodium Thiosulfate, Standard Solution* (0.1 N)—Dissolve 24.8 g of sodium thiosulfate of ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 1 L of freshly boiled and cooled water contained in a sterile glass bottle. If sulfur precipitates during preparation or upon subsequent use, discard the solution and prepare a new one. Standardize against iodine.

12.3 *Starch Indicator Solution*—Stir up 2 to 3 g of potato starch with 100 mL salicylic acid solution (1 %), and boil the mixture until the starch is practically dissolved, then dilute to 1 L with water.

13. Procedure

13.1 Pipet a 25-mL aliquot of the solution of the specimen (Section 9) into a 500-mL glass-stoppered Erlenmeyer iodimetric flask or other suitable glass-stoppered bottle containing 200 mL of H_2SO_4 (1+39). Add 20 mL of KI solution (150 g/L), stopper, and allow the solution to stand for approximately 5 min.

13.2 Titrate the liberated iodine with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution at room temperature until the reddish brown iodine color becomes quite faint. Add 5 mL of starch solution and continue the titration until the final color change becomes pale green with no blue tinge. Titrate this final titration by swirling the flask at least three times after each addition of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, being sure that there is no further color change, especially at the final stage of the titration. The green end point is definite and sharp.

13.3 *Calculation*—Calculate the percent of chromium C as CrO_3 as follows:

$$C = [(V_2N_2 \times 0.03334)/S_2] \times 100$$

where:

V_2 = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the specimen, mL

N_2 = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, and

S_2 = specimen in the aliquot used, g.

ZINC BY THE HYDROXYQUINOLINE TEST METHOD (Suitable if No Interfering Substances Are Present)

14. Reagents

14.1 *Acetone Solution of 8-Hydroxyquinoline* (50 g/L)—Dissolve 5 g of 8-hydroxyquinoline in 100 mL of acetone.

15. Procedure

15.1 Pipet 50 mL of the solution of the specimen (Section 9) into a 250-mL beaker and dilute to 100 mL with water. Add 5 to 10 g of ammonium chloride (NH_4Cl) and heat to boiling. Add a slight excess of ammonium hydroxide (NH_4OH) and let stand a few minutes to allow any precipitate to coagulate. Filter through an ashless, rapid paper into a 400-mL beaker and wash.

15.2 Heat the filtrate to boiling and add 5 mL of NH_4OH (sp gr 0.90). Add dropwise 10 mL of the acetone solution of 8-hydroxyquinoline (Note 6). Let stand 10 to 20 min and filter through a medium-porosity sintered-glass crucible. Wash well with water.

NOTE 6—The reagent is used in acetone solution rather than alcohol solution to eliminate the danger of reducing some of the chromate by alcohol. Avoid adding an excess of reagent and lengthy boiling after its addition. Ten millilitres of hydroxyquinoline (50 g/L) is sufficient for a normal zinc yellow. The solution can be tested for complete precipitation, but since the reagent itself is rather insoluble, the results may be misleading. The insoluble reagent will dissolve in a hot solution and also in an excess of alcohol or acetone, whereas the zinc oxyquinolate will not.

15.3 Dry the precipitate at 165°C for at least 2 h and weigh as zinc oxyquinolate.

15.4 *Calculation*—Calculate the percent zinc A as zinc oxide (ZnO), as follows:

$$A = [(P \times 0.2303)/S_3] \times 100$$

where:

P = zinc oxyquinolate, g, and

S_3 = specimen in aliquot used, g.

0.2303 = $\text{ZnO}/\text{zinc oxyquinolate} = 81.38/353.37$

ZINC BY THE FERROCYANIDE TEST METHOD

16. Reagents

16.1 *Methyl Orange Indicator Solution*—Dissolve 0.1 g of methyl orange in 100 mL of water.

16.2 *Potassium Ferrocyanide, Standard Solution*—Dissolve 22 g of potassium ferrocyanide ($\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$) in water and dilute to 1 L. To standardize, transfer 0.2 g of metallic zinc or freshly ignited ZnO to a 400-mL beaker. Dissolve in 10 mL of hydrochloric acid (HCl , sp gr 1.19) and 20 mL of water. Drop in a small piece of litmus paper, add ammonium hydroxide (NH_4OH) until slightly alkaline, then add HCl until just acid, and then 3 mL more of HCl . Dilute to about 250 mL with hot water and heat nearly to boiling. Run in the $\text{K}_4\text{Fe}(\text{CN})_6$ solution slowly from a buret, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator solution shows a brown tinge after standing 1 min. Do not allow the temperature of the solution to fall below 70°C during the titration. Run a blank using the same amounts of reagents and water as in the standardization. The standardization must be made under the same conditions of temperature, volume, and

acidity as obtained when the specimen is titrated. Calculate the strength of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution in terms of grams of zinc as follows:

$$Z = W/(V_3 - B_2)$$

where:

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL,

W = zinc used (or equivalent to the ZnO used), g,

V_3 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the standard, g, and

B_2 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the blank, mL.

16.3 *Thymol Blue Indicator Solution* (0.5 g/L)—Dissolve 0.1 g of thymol blue indicator in 200-mL of methanol, ethanol, or isopropanol.

16.4 *Uranyl Acetate Indicator Solution* (50 g/L)—Dissolve 5 g of $\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot \text{H}_2\text{O}$ in water made slightly acid with acetic acid and dilute to 100 mL.

17. Procedure

17.1 Pipet 50 mL of the solution of the specimen (Section 9) into a 400-mL beaker. Add 35 mL of H_2SO_4 (1+5) and 3 to 4 drops of thymol blue indicator solution. First add NH_4OH (sp gr 0.90) and finally NH_4OH (1+10) until the color of the indicator changes to a salmon shade intermediate between pink and yellow. This gives a pH of about 2.4. Dilute the solution to 300 mL and heat to just under boiling. Pass in H_2S at a moderate rate for 40 min. Allow the precipitate to settle for 1 h and filter. Wash the filter ten times with water saturated with hydrogen sulfide gas (H_2S).

17.2 Dissolve the precipitate in hot HCl (1+3) and wash the filter paper well with hot water. Boil out the H_2S , neutralize to methyl orange with NH_4OH , and dilute to 300 mL. Add 2 g of NH_4Cl and 3 mL of HCl (sp gr 1.19), and heat to boiling.

17.3 Titrate the hot solution with $\text{K}_4\text{Fe}(\text{CN})_6$ solution (16.2), using uranyl acetate as an external indicator on a spot plate or 1 to 2 drops of ferrous ammonium sulfate solution as an internal indicator.

17.4 *Calculation*—Calculate the percent zinc A as ZnO , as follows:

$$A = (V_4 Z/S_4) \times 100$$

where:

V_4 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the specimen, mL,

Z = ZnO equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL, and

S_4 = specimen in the aliquot used, g.

ALKALINE SALTS

18. Reagents

18.1 *Gelatin Solution* (0.2 g/L)—Dissolve 0.2 g of low-ash gelatin in water and dilute to 1 L.

18.2 *Lead Acetate Solution* (100 g/L)—Dissolve 117 g of $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ in water and dilute to 1 L.

19. Procedure

19.1 Dissolve exactly 1 g (Note 7) of a specimen in 10 mL of acetic acid (1+1) and add 25 mL of water. Heat until dissolved. Dilute to 250 mL and heat to boiling. Add 20 mL of lead acetate solution (100 g/L) and allow the precipitate to

16. Filter and wash the precipitate with hot water.

NOTE 7—The specimen should not be greater than 1 g because above the loss in alkali metals due to adsorption on the precipitate becomes large.

19.2 Saturate the filtrate with hydrogen sulfide (H_2S) for 15 min. Add 10 mL of gelatin solution (0.2 g/L) and stir vigorously. Filter and wash with H_2S water acidified with a few drops of H_2SO_4 (1+1).

19.3 Add 5 mL of H_2SO_4 (1+1) to the filtrate and boil to a volume of 50 mL. Transfer to a silica dish and evaporate to dryness. Ignite gently.

19.4 Leach the residue with hot distilled water, transferring the entire contents of the silica dish to a small beaker. Saturate with H_2S for about 15 min (Note 8). Add 10 mL of gelatin solution (0.2 g/L) and stir vigorously. Filter and wash the precipitate with H_2S water, catching the filtrate in a tared silica dish.

NOTE 8—A second treatment with H_2S is necessary because some of the heavy metals pass through to the alkali metal filtrate.

19.5 Add about 2 mL of H_2SO_4 (1+1) to the filtrate, evaporate to dryness, and again ignite gently. During the ignition process add small portions of solid ammonium carbonate ($(NH_4)_2CO_3$). Cool in a desiccator and weigh.

19.6 Test the residue for calcium. If present, determine the amount, calculate to calcium sulfate ($CaSO_4$), and deduct from the weight of the ignited residue.

19.7 *Calculation*—Calculate the percent of alkaline salts as potassium oxide (K_2O), as follows:

$$D = [(R - W_2) \times 0.541] / S_3 \times 100$$

where:

R = ignited residue (see 16.5), g,

W_2 = $CaSO_4$ (if any) (see 16.6), g, and

S_3 = sample used, g.

SULFATES

20. Reagent

20.1 *Barium Chloride Solution* (100 g/L)—Dissolve 117 g $BaCl_2 \cdot H_2O$ in water and dilute to 1 L.

21. Procedure

21.1 For this determination take about 20 g of Type I pigment or about 5 g of Type II pigment as defined in Specification D 478. Weigh the specimen into a 1-L beaker. Add 75 mL of HCl (sp gr 1.19). Boil the solution under a hood until the rapid evolution of chlorine has subsided. Add 5 mL of methyl alcohol and boil to a volume of about 35 mL (Note 9). Add 25 mL of HCl and continue boiling. Add 5 mL of methyl alcohol and again boil to a volume of about 35 mL.

NOTE 9—If the volume is reduced below 35 mL, a precipitate may form that will not dissolve upon dilution. It is imperative, however, that all the chromium be reduced.

21.2 Dilute the solution to 400 mL with water. If the solution is not clear at this point, filter it. Heat to boiling and add 50 mL of glacial acetic acid. Add 10 mL of $BaCl_2$ solution (100 g/L) dropwise, while stirring. Boil for 30 min and allow to stand overnight (Note 10). Filter through a tared Gooch crucible and wash with hot water.

NOTE 10—The acetic acid is necessary to minimize the tendency of sulfate to form an inner complex with the trivalent chromium. It is necessary that the solution stand overnight to ensure the precipitation of all the sulfate.

21.3 Dry the precipitate in an oven, and ignite at $900^\circ C$ to constant weight. Weigh as barium sulfate ($BaSO_4$).

NOTE 11—If the wet precipitate has a yellow appearance, all the chromate was not previously reduced. The results will be high and the specimen should be discarded. Greater attention should then be given to the reduction of the chromium. A slight greenish color cannot be avoided but does not indicate a serious error.

21.4 *Calculation*—Calculate the percent sulfates E as sulfur trioxide (SO_3), as follows:

$$E = [(P_2 \times 0.343) / S_6] \times 100$$

where:

P_2 = $BaSO_4$, g, and

S_6 = sample used, g.

CHLORIDES

22. Reagent

22.1 *Silver Nitrate Solution* (17 g/L)—Dissolve 17.0 g of $AgNO_3$ in water and dilute to 1 L.

23. Procedure

23.1 Weigh a 10-g specimen into a 600-mL beaker. Add 200 mL of water and 50 mL of nitric acid (HNO_3) (2+3). Warm just enough to dissolve the specimen. Filter to remove insoluble material. Add a slight excess of $AgNO_3$ solution (17 g/L) (Note 12). Boil for 5 min and let stand for about 2 h.

NOTE 12—If care is exercised in adding but a slight excess of $AgNO_3$ solution, no silver chromate (Ag_2CrO_4) will form. About 10 to 15 mL of $AgNO_3$ solution (17 g/L) is usually not too much.

23.2 If no crystals of Ag_2CrO_4 are present, filter at room temperature through a tared, fine-porosity sintered-glass crucible, using suction. Wash the precipitate free of $AgNO_3$ with HNO_3 (1+99). To be sure that an excess of $AgNO_3$ was used, test the filtrate by adding a few drops of HCl (2+3). Dry the precipitate at $105 \pm 2^\circ C$ for 2 h, cool, and weigh as silver chloride ($AgCl$).

23.3 If crystals of Ag_2CrO_4 are present (Note 13), filter the solution through a fine-porosity sintered-glass crucible and discard the filtrate containing most of the chromium. Dissolve the $AgCl$ precipitate by pouring 100 mL of hot ammonium hydroxide (NH_4OH) (1+5) slowly through the crucible while applying gentle suction. Catch the filtrate in a clean flask, taking care not to lose any of it. Wash with a few millilitres of HNO_3 (sp gr 1.42) and then with a little more NH_4OH (1+5).

NOTE 13—Silver chromate is difficult to dissolve completely by washing with dilute HNO_3 ; hence, in such cases, it is necessary to dissolve in NH_4OH and reprecipitate.

23.4 Transfer the solution to a beaker and make it faintly acid by adjusting with either HNO_3 (1+5) or NH_4OH (1+5) as required. Add a few drops of $AgNO_3$ solution (17 g/L) and boil for 5 min. Let stand at least 2 h in a dark place. Filter through a tared, fine-porosity sintered-glass crucible, using suction.

23.5 Wash the precipitate free of AgNO_3 with HNO_3 (1+99), and dry at $105 \pm 2^\circ\text{C}$ for 2 h. Cool and weigh as AgCl .

23.6 *Calculation*—Calculate the percent chlorides E as chlorine, as follows:

$$E = [(P_3 \times 0.247)/S_7] \times 100$$

where:

P_3 = AgCl , g,

S_7 = specimen used, g, and

$0.247 = \text{Cl}/\text{AgCl} = 34.45/143.32$.

MATTER INSOLUBLE IN DILUTE ACETIC ACID

24. Procedure

24.1 Weigh to 0.1 mg about a 10-g specimen (Note 14) and place in a 600-mL beaker. Add 300 mL of acetic acid (1+9). Heat the mixture to 80°C and maintain at $80 \pm 5^\circ\text{C}$, while stirring, until nothing further dissolves. Filter while hot through a tared Gooch crucible. Wash the insoluble residue on the filter with hot water.

NOTE 14—If the pigment contains an organic treating agent, first remove this treating agent by washing with ether or chloroform.

24.2 Dry the crucible at $105 \pm 2^\circ\text{C}$ and weigh.

24.3 *Calculation*—Calculate the percent of matter insoluble in acetic acid (1+9) F as follows:

$$F = (R_1/S_8) \times 100$$

where:

R_1 = residue, g, and

S_8 = specimen used, g.

COARSE PARTICLES

25. Procedure

25.1 Determine the percent of coarse particles in the pigment as received, in accordance with Test Methods D 185.

INDEX TERMS

26. Index Terms

26.1 These test methods are indexed under the following terms: zinc chromate; pigments—zinc chromate.

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Standard Specification for Pure Para Red Toner Pigment¹

This standard is issued under the fixed designation D 475; the number immediately following the designation indicates the year of original adoption 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 specification covers the red pigment commercially known as pure para red toner. It is available in two types: Type I, light, and Type II, dark.

Referenced Document

1 *ASTM Standard:
D 970 Test Methods for Para Red and Toluidine Red
Pigments*²

Composition and Properties

3.1 Dry Pigment:

3.1.1 *Type I*—The pigment shall be in a soft dry form and shall consist of para red toner, light (1-(*p*-nitro-phenyl-azo)-2-naphthol) free of any substrate.

3.1.2 *Type II*—The pigment shall be in a soft dry form and shall consist of para red toner, light (1-(*p*-nitro-phenyl-azo)-2-naphthol) mixed with a small quantity of 1-(*p*-nitro-phenyl-azo)-2-naphthol-7-sulfonic acid free of any substrate.

3.2 The pigments shall conform to the following requirements:

	Type I	Type II
Ash, max, %	3.0	3.0
Moisture and other volatile matter, max, %	1.0	1.0
Coarse particles (total residue retained on a 45-μm (No.325) sieve), max, %	1.5	1.5
Solubility in chloroform	complete	...
Resistance to acids	to pass test	to pass test
Resistance to alkalis	to pass test	equal to standard
Identity test	to pass test	to pass test

3.3 The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (5 000 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with Test Methods D 970.

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 D01.31 on Pigment Specifications.

Current edition approved Nov. 30, 1967. Originally published as D 475 - 38 T. Last previous edition D 475 - 49.

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

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Standard Specification for Titanium Dioxide Pigments¹

This standard is issued under the fixed designation D 476; the number immediately following the designation indicates the year of original adoption 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 specification covers four types of titanium dioxide pigments:

- 1.1.1 *Type I*—Anatase, free chalking.
- 1.1.2 *Type II*—Rutile, medium chalk resistant (low).
- 1.1.3 *Type III*—Rutile, medium chalk resistant (high).
- 1.1.4 *Type IV*—Rutile, highly chalk resistant.

2. Referenced Documents

2.1 ASTM Standards:

- D 34 Practice for Chemical Analysis of White Pigments²
- D 153 Test Methods for Specific Gravity of Pigments²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 1394 Test Methods for Chemical Analysis of White Titanium Pigments²
- D 2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment²
- D 3720 Test Method for Ratio of Anatase to Rutile in Titanium Dioxide Pigments by X-Ray Diffraction²

3. Composition and Properties

3.1 *Titanium Dioxide Pigments*,⁴ shall conform to the requirements for composition prescribed in Table 1. They shall be chemically prepared pigments consisting of anatase or rutile titanium dioxide with or without modifications with water-insoluble oxides of aluminum, silicon, zinc, etc., or other agents; these reagent materials are introduced specifically to improve those properties for which the pigment is used. The titanium dioxide pigments shall be free of ex-

tenders such as barium sulfate, clay, magnesium silicate, whiting, etc.

3.2 The desired properties of the pigment, other than as herein indicated, shall be subject to mutual agreement between the purchaser and the seller and shall be based on a satisfactory match between the submitted pigment sample and a previously agreed upon reference sample (see 5.1.6).

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or as a composite sample formed by blending in equal quantities the samples from the same production unit.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.1.1 *Chemical Analysis*—Practice D 34 or Test Methods D 1394.

5.1.2 *Specific Gravity*—Test Methods D 153.

5.1.3 *Coarse Particles*—Test Methods D 185.

5.1.4 *Moisture*—Test Method D 280.

5.1.5 *Matter Soluble in Water, Specific Resistance*—Test Method D 2448. The measured specific resistance of the aqueous leachate from the pigment is an index of water-soluble salts.

5.1.6 *Chalk Resistance*—It is recommended that purchaser and seller agree upon standards and methods of test suitable for their requirements (see 3.2). Comparison in a good quality exterior air-dry alkyd enamel (trade sales or industrial) and exposure in Florida at 45° facing south are recommended. Chalking differences are minimized by less durable vehicles and required exposure times are prolonged by more durable vehicles. Once the reference sample agreed upon by the purchaser and seller has qualified, subsequent shipments of that product may be compared with the reference sample by a mutually agreed upon accelerated weathering test.

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 D01.31 on Pigment Specifications.

Current edition approved Nov. 14, 1984. Published January 1984. Originally published as D 476 - 38. Last previous edition D 476 - 73 (1979).

² Annual Book of ASTM Standards, Vol 06.02.

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

⁴ The Titanium Dioxide Pigments specified by type in 3.1 are indexed by type in the National Paint and Coatings Association, Raw Materials Index, Pigment Section.

TABLE 1 Requirements for Composition of Titanium Dioxide Pigments

	Type I	Type II	Type III	Type IV
	Anatase ^a Free Chalking	Rutile—Medium Chalk Resistant	Rutile—Medium Chalk Resistant	Rutile—Highly Chalk Resistant
Typical Paint Applications	white exterior house paints; interior uses	low-medium % PVC enamels and lacquers	medium-high % PVC enamels; alkyd and emulsion flat wall paints	exterior coatings requiring excellent durability and gloss retention
Titanium dioxide (TiO ₂), min, %	94	92	80	80
Insoluble in water, specific resistance, ohm-cm	5000 ^c	5000	3000	3000
Unburned and other volatile matter (loss at 550°C, max, % ^e)	0.7	0.7	1.5	1.5
Specific gravity	3.8 to 4.0	4.0 to 4.3	3.6 to 4.3	3.6 to 4.3
Agglomerates (total residue retained on a No. 325 sieve), max, %	0.2	0.2	0.2	0.2

Identification of anatase or rutile or mixtures thereof is made by X-ray analysis (Test Method D 3720).

Conditioned overnight at 50 % relative humidity at 25°C (for referee purposes only).

1000 permissible for water dispersible types only.

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Last ASTM Designation: D 477 - 78

Standard Specification for Zinc Sulfide Pigments

This specification covers zinc sulfide pigments.

Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials, this specification was discontinued in 1988.

Standard Specification for Zinc Yellow (Zinc Chromate) Pigments¹

This standard is issued under the fixed designation D 478; the number immediately following the designation indicates the year of original adoption 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.

Scope

- 1.1 This specification covers the pigments commercially known as zinc yellow (zinc chromate).
- 1.2 Two types are included:
 - 1.2.1 *Type I*—High-purity, low sulfate and chloride content.
 - 1.2.2 *Type II*—Regular grade.

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 Muller³
 - D 444 Test Methods for Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow)³

3. Significance and Use

- 3.1 Zinc yellow is used in rust-inhibiting protective coatings and metal primers for ferrous and non-ferrous metals.

4. Composition and Properties

4.1 *Dry Pigment*—The pigment shall be a reaction precipitate of soluble chromates and a suitable zinc compound and shall be free of extenders, carbonates, and organic color in any form. The pigment shall conform to the requirements for composition prescribed in Table 1.

4.2 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

5. Sampling

5.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

6. Test Methods

6.1 Tests shall be conducted in accordance with the following ASTM test methods:

6.2 *Chemical Analysis*—Test Methods D 444.

6.3 *Coarse Particles*—Test Methods D 185.

6.4 *Mass Color and Tinting Strength*—Test Method D 387.

7. Keywords

7.1 chromatic; inhibitor metal primer; pigment; zinc chromate; zinc yellow

TABLE 1 Composition Requirements

	Type I	Type II
Zinc (calculated as ZnO), %	35 to 40	35 to 40
Chromium (calculated as CrO ₃), min, %	41	41
Sulfates (calculated as SO ₃), max, %	0.20	3.0 ^A
Chlorides (calculated as Cl), max, %	0.10	0.8 ^A
Alkaline salts (calculated as K ₂ O), max, %	13	13
Matter insoluble in acetic acid (1+9) at 80°C, max, %	0.3	0.3
Moisture and other volatile matter, max, %	1.0	1.0
Coarse particles (total residue retained on No. 325 (45-μm) sieve), max, %	1.0	1.0

^A The maximum percent of sulfates or chlorides given in the table for Type II pigment apply if only one or the other is present (for this purpose amounts of less than 0.05 % shall be disregarded). If both sulfates and chlorides are present, the sum of the percentages of each divided by its respective maximum permissible percentage (3.0 for SO₃ and 0.8 for Cl) shall not exceed 1.0. For example, if a sample contains 1.8 % SO₃ (0.6 of the maximum for sulfates) and 0.3 % chlorides (0.4 of the maximum for chlorides), the sample just conforms to the maximum requirements of the specification in this respect.

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 D01.31 on Pigment Specifications.

Current edition approved April 25, 1986. Published June 1986. Originally published as D 478 - 38 T. Last previous edition D 478 - 49 (1981)^{e1}.

² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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Standard Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes¹

This standard is issued under the fixed designation D 480; the number immediately following the designation indicates the year of original adoption 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 4451.1, 7233 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.

Scope

1. These test methods cover procedures for sampling, qualitative analysis, and physical testing of flaked aluminum powders and pastes (leafing and nonleafing) for coatings.
2. These test methods apply equally to leafing and nonleafing flaked aluminum powders and pastes, except where noted to the contrary.
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 Note 1.*

Referenced Documents

- 2.1 *ASTM Standards:*
 - D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
 - D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Drycleaning Solvent)³
 - D 329 Specification for Acetone³
 - D 962 Specification for Aluminum Powder and Paste Pigments for Paints⁴
 - D 3980 Practice for Interlaboratory Testing of Paint and Related Materials²
 - E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵
 - E 34 Test Methods for Chemical Analysis of Aluminum and Aluminum Alloys⁶
 - E 607 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique, Nitrogen Atmosphere⁷
 - E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁸

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 D01.31 on Pigment Specifications.

Current edition approved July 29, 1988. Published December 1988. Originally published as D 480 - 38. Last previous edition D 480 - 70 (1976).

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 06.02.

⁵ Annual Book of ASTM Standards, Vol 14.02.

⁶ Annual Book of ASTM Standards, Vol 03.05.

⁷ Annual Book of ASTM Standards, Vol 03.06.

⁸ Annual Book of ASTM Standards, Vols 06.03 and 14.02.

3. Significance and Use

3.1 Flaked aluminum pigments are produced in a variety of forms. These test methods allow the user to determine the applicability of a given product to this use.

4. Sampling

4.1 Sampling is subject to mutual agreement between the seller and the purchaser. Place each sample in a clean, dry metal or glass container which shall be nearly filled, then close with a tight cover, seal, mark, and send to the laboratory for testing.

4.2 When requested, duplicate samples may be taken from the same container and delivered to the seller, and the inspector may take a third set of samples to hold for test in case of disagreement.

5. Qualitative Analysis

5.1 *Significance and Use*—This test method determines if there are chemical impurities, other than fatty and oily matter, as specified in Specification D 962, to ensure the absence of fillers or extender pigments.

5.2 Procedure:

5.2.1 *Total Impurities*—Determine the total impurities by weight in accordance with Test Methods E 34 and E 607 or by atomic absorption.

5.2.2 *Total Aluminum*—Determine the total aluminum by weight difference with elemental impurities in accordance with 5.2.1.

6. Leafing Properties

6.1 The leafing test conditions specified are selected arbitrarily, and even though the numerical leafing value obtained by this test method appears to be low, the pigment may give substantially perfect leafing under the conditions of practical application in a paint.

6.2 *Significance and Use*—This leafing test method has been established to determine the percent of leafed aluminum flakes at the surface of a simulated paint formula to ensure a bright metallic luster. Minimum leafing characteristics must conform to Specification D 962.

6.3 Apparatus:

6.3.1 *Leafing Spatula*, nonmagnetic, stainless steel spatula having the following dimensions:

Length of blade min, in. (mm) 5.5 (139.7)

Width of blade, in. (mm) 0.540 $\pm$ 0.002 (13.72 $\pm$ 0.05)

Thickness of blade, in. (mm):

10 mm from tip	0.013 ± 0.003 (0.33 ± 0.08)
100 mm from tip	0.038 ± 0.004 (0.97 ± 0.10)
Shape of tip	rounded or straight

The surface of the spatula shall be polished with 3/0 emery or silicon carbide metallographic paper wet with mineral spirits; polishing shall be repeated for 1-min intervals until a consistent minimum leafing result is obtained in a typical leafing test.

6.3.2 *Test Tube*, 6 in. (152 mm) in length by 0.75 in. (19.0 mm) in diameter.

6.3.3 *Glass Cylinder*, 8 in. (203 mm) in length and 1.5 to 2.0 in. (38.1 to 50.8 mm) in inside diameter. A stopper shall be used and the spatula shall be attached to the stopper so as to hang vertically in the cylinder with the tip about 1 in. (25 mm) from the bottom.

6.4 Reagents:

6.4.1 *Coumarone-Idene Resin*—The form commercially known as "chipped" and having the following properties:

Softening point, °C	109 to 117
Specific gravity	1.12 to 1.16
Acid value, max	0.5
Cloud point, °C, max	-7

6.4.2 *Petroleum Spirits*—Conforming to Specification D 235 and having the following additional properties:

Specific gravity at 60°F (16°C)	0.800 to 0.810
Surface tension at 70°F (21°C), N/m, min	24.5
Kauri butanol value	42 to 44
Aniline point, °F (°C)	105 to 115 (40 to 45)

6.5 *Preparation of Leaf-Testing Vehicle*—Prepare solution by dissolving 30.0 g of resin, in the form of small chips, in 100 mL of petroleum spirits, while heating gently at about 140°F (60°C). The specific gravity of the solution shall be between 0.877 and 0.881 at 60°F (16°C). Allow the solution to settle and retain the clear portion for use.

6.6 *Aliquot*—Unless otherwise specified, the aliquot weight shall be as follows:

Classification	ASTM Designation	Class	Aliquot Weight, g
Flaked aluminum powder	D 962, Type 1	A	1.0
		B	1.0
		C	2.0
Aluminum paste	D 962, Type 2	A	1.5
		B	1.5
		C	3.0

6.7 *Procedure*—Perform the test at a room temperature of 25 ± 2°C. Place approximately 5 mL of the leaf-testing vehicle in the glass cylinder and cover with a watch glass. Transfer 25.0 ± 1.0 mL of the vehicle to a clean container. Weigh the required aliquot of aluminum powder or paste to the nearest 0.01 g and transfer to a small dish. Add about 2.5 mL of the 25-mL quantity of vehicle and mix to a stiff paste with a spatula or small stiff brush. Add approximately 5 mL more of the vehicle and stir to a smooth mixture, then add the remainder of the vehicle and continue stirring to obtain complete dispersion. Transfer the mixture to the test tube to give a depth of about 110 mm when the specified spatula is immersed. Avoid formation of bubbles. Dip the spatula to the bottom of the mixture and rotate it gently through an arc of about 90° for 10 s. Withdraw the spatula at a uniform rate of about 2 in. (50 mm)/s. The spatula should not touch the

wall of the test tube above the liquid level. Immediately suspend the spatula vertically in the glass cylinder with leafing liquid in it and allow to remain for 3 min. Measure the height of immersion and the height of complete leafing on both sides of the spatula, measuring to the bottom of the meniscus in each case. Clean the spatula by rinsing it in petroleum spirits and wipe dry with a clean cloth. Stir (do not shake) the mixture in the test tube and repeat the determination.

6.8 *Calculation*—Calculate the leafing value L as follows:

$$L = A/B \times 100 = A \times 100/B$$

where:

A = height of complete leafing, in. (mm), and

B = height of immersion, in. (mm).

7. Coarse Particles

7.1 This section provides two test methods for determining the percent residue retained on a sieve by wet-screening techniques.

7.2 *Significance and Use*—These procedures determine the percent of coarse particles present in aluminum pigments to ensure a smooth appearance, as required in Specification D 962.

7.3 Procedure A:

7.3.1 *Aluminum Powder*—Fill two "straight wall" containers not less than 12 in. (305 mm) in diameter to a depth of not less than 4 in. (102 mm) with petroleum spirits, and fill a third similar container to a depth of not less than 3 in. (76 mm) with acetone. The petroleum spirits used for the test shall conform to Specification D 235, and the acetone shall conform to Specification D 329. Weigh 5 g of the aliquot, transfer to a 250-mL beaker, and add in small portions a total of about 150 mL of petroleum spirits, mixing thoroughly to a uniform consistency after each addition. Clamp a standard sieve 8 in. (203 mm) in diameter conforming to Specification E 11, just above the level of the petroleum spirits in the first container and pour the specimen over the sieve, using a 45-μm (No. 325) sieve for Classes A, B, and C powder and in addition a 150-μm (No. 100) sieve for Class C powder. Rinse the beaker with clear petroleum spirits and transfer the rinsings to the sieve. Holding the sieve at a slight angle to prevent air lock, shake it backward and forward working the sieve alternately just under and just above the level of the liquid with rotation, avoiding spillage from the screen, at a rate of approximately 60 to 100 cpm. It is important that the material be thoroughly sieved in each container. After the bulk of the aliquot has passed through the sieve (usually about 2 min) repeat the procedure in the second container for about 2 min. When it is evident that practically none of the residue on the sieve is passing through it, repeat the procedure in the acetone container for about 2 to 3 min until nothing more passes through the sieve. Rinse the sides of the sieve with a small stream of acetone, and collect the residue at one side of the sieve. Transfer the residue to a dried and weighed aluminum cup or small evaporating dish with a small stream of acetone, using not less than 15 to 25 mL. Place the aluminum cup or dish in an oven, evaporate to dryness at 105 to 100°C, and continue heating at this temperature for 15 min.

Warning—Do not close the door of the oven while evaporating acetone, as there is danger of explosion or flash of acetone.

Aluminum Paste—Determine the percent of coarse particles in accordance with the procedure described in 7.3.1 using a 45- μ m (No. 325) sieve for Classes A, B, and C paste and in addition a 150- μ m (No. 100) sieve for Class C paste.

Calculation—Calculate the weight percent of the residue retained on the sieve R from the increase in weight as follows:

$$R = [(A/W) \times CF] \times 100$$

weight of dried residue, g,
correction factor (1.0 for NIST sieves), and
aliquot weight, g.

NOTE 2—A correction factor shall be applied in the case of sieves used in the test that have not been certified by the National Institute of Standards and Technology. Sieves having a correction factor above 1.2 and below 0.8 should not be used. The correction factor may be calculated as follows: Determine the coarse particles of an aliquot on a master sieve certified by the National Institute of Standards and Technology following the procedure described in 7.3.1. Sieve the same aliquot on the unknown sieve by the same procedure. Calculate the correction factor, as follows:

$$CF = X/Y$$

where:

X = weight of residue on the NIST sieve, g, and
 Y = weight of residue on the unknown screen, g.

4 Procedure B—Test in accordance with Test Methods D 185 to determine conformance of flaked aluminum powders and pastes to Specification D 235.

8. Easily Extracted Fatty and Oily Matter

8.1 Easily extracted fatty and oily matter is determined by the following arbitrary test method. Details of the test procedure shall be strictly followed.

8.2 Significance and Use—This test method determines the percent of easily extracted fatty and oily matter in aluminum pigments in accordance with Specification D 962.

8.3 Procedure:

8.3.1 Aluminum Powder—Weigh accurately (to within 0.01 g) approximately 2 g of the aliquot. Transfer to a 200-mL volumetric flask and add about 50 mL of redistilled acetone conforming to Specification D 329. Heat the mixture to boiling while occasionally agitating. Cool, fill to the

200-mL mark with acetone, and mix. Allow the mixture to settle. Draw off approximately one half of the supernatant liquid and filter through dry paper. Discard the first 10 mL of the filtrate. Transfer 100 mL of the clear filtrate to a weighed dish. Evaporate the acetone at a temperature not above 75°C. Heat the dish for 15 min in an oven at 105 to 110°C. Cool and weigh the amount of fatty oily matter.

8.3.2 Aluminum Paste—Determine the percent of easily extracted fatty and oily matter in aluminum paste in accordance with the procedure described in 8.3.1.

8.4 Calculation—Calculate the weight percent of fatty and oily matter F as follows:

$$F = [2 \times (A/W)] \times 100$$

where:

A = weight of residue in 100 mL of acetone, g, and
 W = weight of aliquot, g.

9. Nonvolatile Matter

9.1 Determine the percent of nonvolatile matter in aluminum paste consisting of aluminum, fatty acids, additives, and their inter-reaction products.

9.2 Significance and Use—This test method determines the combined percent of aluminum and other nonvolatile matter, such as fatty acids and their inter-reaction products, in aluminum pigments and their conformance with Specification D 962.

9.3 Procedure—Aluminum Paste and Powder—Weigh into an evaporating dish 5 g of paste or powder to within 0.01 g. Evaporate in an oven at a temperature of 105 to 110°C for 3 h. Cool to room temperature and weigh.

9.4 Calculation—Calculate the weight percent of nonvolatile matter NV as follows:

$$NV = A/W \times 100$$

where:

A = weight of residue, g, and
 W = weight of aliquot, g.

10. Precision and Bias

10.1 Precision and Bias—Determine in accordance with Practices D 3980 and E 691.

11. Index Terms

11.1 These test methods are indexed under the following terms: flaked powders; leafing properties; aluminum powders and pastes.

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Standard Specification for Zinc Dust Pigment¹

This standard is issued under the fixed designation D 520; the number immediately following the designation indicates the year of original adoption 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 two types of zinc dust, for use as a pigment in paints. Type I is a general grade. Type II is a high-purity grade.

2. Referenced Documents

2.1 ASTM Standards:

- B 214 Test Method for Sieve Analysis of Granular Metal Powders²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 521 Test Methods for Chemical Analysis of Zinc Dust (Metallic Zinc Powder)⁴
- E 40 Test Methods for Chemical Analysis of Slab Zinc (Spelter)⁵

3. Composition and Properties

3.1 The pigments shall consist substantially of metallic zinc and shall conform to the requirements for composition prescribed in Table 1.

3.2 In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon between the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing

TABLE 1 Requirements for Composition

	Type I	Type II
Total zinc, calculated as Zn, min, %	97.5	98.0
Metallic zinc, min, %	94.0	94.0
Material other than metallic zinc, ZnO, and admixed CaO, where applicable max %	0.75	...
Calcium, calculated as CaO, max, %	0.7	0.7
Lead, calculated as Pb, max, %	...	0.01
Iron, calculated as Fe, max, %	...	0.02
Cadmium, calculated as Cd, max, %	...	0.01
Chlorine, calculated as Cl, max, %	...	0.01
Sulfur, calculated as SO ₂ , max, %	...	0.01
Moisture and other volatile matter, max, %	0.10	0.10
Oil or fatty matter, or both, max, %	...	0.05
Zinc oxide (ZnO), max, %	6.0	remainder
Coarse particles, max, %:		
Total residue retained on a 150- μ m (No. 100) sieve	none	0.1
Total residue retained on a 75- μ m (No. 200) sieve	...	0.8
Total residue retained on a 45- μ m (No. 325) sieve	4.0	3.0

between units of production appear, samples shall be taken from 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, two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by these ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Total and Metallic Zinc*—Methods D 521.

5.1.2 *Lead, Cadmium, and Iron*—Sections 8, 18.2, and 22, respectively, of Methods E 40.

5.1.3 *Oil or Fatty Matter, or Both*—Methods D 521.

5.1.4 *Coarse Particles*—Test Method B 214 or Test Methods D 185.

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 D01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vol 02.05.

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

⁴ Annual Book of ASTM Standards, Vol 06.02.

⁵ Annual Book of ASTM Standards, Vol 03.05.

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Standard Test Methods for Chemical Analysis of Zinc Dust (Metallic Zinc Powder)¹

This standard is issued under the fixed designation D 521; the number immediately following the designation indicates the year of original adoption 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. These test methods cover procedures for the chemical analysis of metallic zinc powder in the form commercially known as zinc dust for use as a pigment in paints.

2. The analytical procedures appear in the following order:

	Sections
Moisture and Other Volatile Matter	7
Coarse Particles	8
Matter Soluble in Hexane	9 and 10
Slab Zinc	11 and 12
Metallic Zinc	13 and 14
Zinc Oxide	15
Cadmium	16 and 17
Lead	18
Iron	19
Cadmium	20
Bromine	21 and 22
Mercury	23 and 24

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

2.1 ASTM Standards:

- B 214 Test Method for Sieve Analysis of Granular Metal Powders²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments⁴
- D 1193 Specification for Reagent Water⁵
- E 40 Test Methods for Chemical Analysis of Slab Zinc (Spelter)⁶
- E 68 Polarographic Determination of Lead and Cadmium in Zinc⁷

3. Significance and Use

3.1 These test methods compile procedures which can 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 D01.21 on Chemical Analysis of Paint and Paint Materials.

² Current edition approved May 25, 1990. Published July 1990. Originally published as D 521 - 39 T. Last previous edition D 521 - 81.

³ Annual Book of ASTM Standards, Vol 02.05.

⁴ Annual Book of ASTM Standards, Vols 06.01 and 06.02.

⁵ Annual Book of ASTM Standards, Vol 06.02.

⁶ Annual Book of ASTM Standards, Vol 06.03.

⁷ Annual Book of ASTM Standards, Vol 03.05.

⁸ Discontinued—See 1979 Annual Book of ASTM Standards, Part 12.

used to check the composition of purity of metallic zinc powder. This information is useful to both the formulator and users.

4. Treatment of Sample

4.1 Store the laboratory sample in a tightly stoppered bottle to protect it from oxidation. Mix the whole sample thoroughly before taking portions for analysis.

5. Purity of Reagents

5.1 *Purity of Reagents*—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.

5.2 *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. Precision

6.1 Precision statements have not been established.

MOISTURE AND OTHER VOLATILE MATTER

7. Procedure

7.1 Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280, except heat the sample for only 1 h.

COARSE PARTICLES

8. Procedure

8.1 Determine the percent of coarse particles in the pigment in accordance with Test Methods D 185 or B 214.

MATTER SOLUBLE IN HEXANE

9. Reagent

9.1 *Hexane*—Pure hexane or commercial hexane or petroleum ether of boiling point not higher than 75°C. Redistill before using.

10. Procedure

10.1 Place 100 g of the pigment in an extraction thimble

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."

in a Soxhlet extraction apparatus. Record the tare weight of the receiving flask. Charge the flask with a suitable volume of hexane and extract the sample for 4 h, subjecting the specimen to not less than 20 extractions in this time. Make a blank determination at the same time.

10.2 Remove the receiving flask, evaporate or distill off the hexane on a steam bath, and dry the flask at $105 \pm 2^\circ\text{C}$ for 1 h. Cool and weigh.

10.3 Calculate the percent of matter soluble in hexane, allowing for any material found in the blank.

TOTAL ZINC

11. Reagents

11.1 *Potassium Ferrocyanide, Standard Solution*—Dissolve 22 g of potassium ferrocyanide ($\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$) in water and dilute to 1 L. To standardize, transfer 0.2 g of metallic zinc or freshly ignited zinc oxide (ZnO) to a 400-mL beaker. Dissolve in 10 mL of hydrochloric acid (HCl), sp gr 1.19 and 20 mL of water. Drop in a small piece of litmus paper, add ammonium hydroxide (NH_4OH) until slightly alkaline, then add HCl until just acid, and then 3 mL more of HCl . Dilute to about 250 mL with hot water and heat nearly to boiling. Run in the $\text{K}_4\text{Fe}(\text{CN})_6$ solution slowly from a buret, while stirring constantly, until a drop tested on a white porcelain plate with a drop of the uranyl indicator solution shows a brown tinge after standing 1 min. Do not allow the temperature of the solution to fall below 70°C during the titration. Run a blank using the same amounts of reagents and water as in the standardization. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated. Calculate the strength of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution in terms of grams of zinc as follows:

$$Z = W/(V_1 - B) \quad (1)$$

where:

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL,

W = zinc used (or equivalent to the ZnO used), g,

V_1 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the standard, mL, and

B = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the blank, mL.

11.2 *Uranyl Nitrate Indicator Solution*—Dissolve 5 g of uranyl nitrate ($\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) in 100 mL of water.

12. Procedure

12.1 Transfer 0.25 g of the sample to a 400-mL beaker, moisten with alcohol, and dissolve in 10 mL of HCl (sp gr 1.19) and 20 mL of water.

12.2 Continue with the procedure used in standardizing the $\text{K}_4\text{Fe}(\text{CN})_6$ solution as described in 10.1, beginning with the addition of the litmus paper and the adjustment of the acidity with NH_4OH and HCl .

12.3 *Calculation*—Calculate the percent of total zinc, T , as follows:

$$T = [(V_2 - B_2)Z/S_1] \times 100 \quad (2)$$

where:

V_2 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the specimen, mL,

B_2 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the blank, mL,

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL, and

S_1 = sample used, g.

METALLIC ZINC

NOTE 1—Sections 12 and 13 cover a rapid method for determining metallic zinc, intended for routine analysis. The results are inclined to be somewhat low, and for highest accuracy and particularly for referee tests, the hydrogen evolution method should be used.⁹

13. Reagents

13.1 *Ferric Chloride Solution*—Prepare a solution containing 20 g of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and 20 mL of 20 % sodium acetate ($\text{NaC}_2\text{H}_3\text{O}_2$) solution per 100 mL. It is advisable to make up only one day's supply at a time (50 mL are required for each determination).

13.2 *Potassium Permanganate, Standard Solution* (0.1 N, 1 mL = 0.008 g TiO_2)—Dissolve 3.16 g of KMnO_4 in water and dilute to 1 L. Let stand 8 to 14 days, siphon off the clear solution (or filter through a medium porosity fritted disk), and standardize against the National Institute of Standards and Technology (NIST) standard chemical No. 40 of sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) as follows: In a 400-mL beaker dissolve 0.2500 to 0.3000 g of the NIST sodium oxalate in 250 mL of hot water (80 to 90°C) and add 15 mL of 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 solution shall not be below 60°C by the time the end point has been reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small hot plate during the titration. The use of a small nonmercury type thermometer as a stirring rod is most convenient.) Keep the KMnO_4 solution in a glass-stoppered bottle painted black to keep out light, or in a brown glass bottle stored in a dark place.

13.3 *Sodium Acetate Solution* (200 g/L)—Dissolve 200 g of sodium acetate ($\text{NaC}_2\text{H}_3\text{O}_2$) or 332 g of $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$, in water and dilute to 1 L.

13.4 *Zimmerman-Reinhardt Solution*—Prepare a solution containing 67 g of manganese sulfate ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$) 130 mL of H_2SO_4 (sp gr 1.84), and 138 mL of phosphoric acid (H_3PO_4) (85 %) per L.

14. Procedure

14.1 Weigh 0.2 g of the sample, transfer immediately to a dry, 600-mL heavy-wall Erlenmeyer flask, and add 50 mL of the FeCl_3 solution. Tightly stopper the flask and agitate constantly for approximately 15 min. As soon as the zinc dust is all dissolved, add 50 mL of the Zimmerman-Reinhardt solution and 250 mL of water. Titrate with 0.1 N KMnO_4 solution.

⁹ Wilson, L. A., "The Evaluation of Zinc Dust: A Proposed Method of Analysis," *Proceedings, ASTEA, Am. Soc. Testing and Mats.*, Vol 18, Part II, 1918, p. 220.

15.2—If preferred, the reduced iron may be titrated with 0.1 *N* dichromate ($K_2Cr_2O_7$) solution, using sodium diphenylamine in the presence of phosphoric acid (H_3PO_4) as an internal indicator.¹⁰

Blank—Make a blank determination, following the procedure and using the same amounts of all reagents as the titration. (The blank is usually 0.1 to 0.2 mL of $KMnO_4$ solution.)

Calculation—Calculate the percent of metallic zinc, *Z*, as follows:

$$M = [(V_3 - B_3)(N \times 0.0327)/S_2] \times 100 \quad (3)$$

V_3 = $KMnO_4$ solution required for titration of the specimen, mL,

B_3 = $KMnO_4$ solution required for titration of the blank, mL,

N = normality of the $KMnO_4$ solution,

S_2 = sample used, g, and

M = milliequivalent weight of Zn.

ZINC OXIDE

15. Calculation

15.1 Calculate the percent of zinc oxide (ZnO), *Z*, as follows:

$$Z = (A - C) \times 1.2447 \quad (4)$$

where:

A = total zinc, % (Section 10), and

C = metallic zinc, % (Section 12).

CALCIUM

16. Reagents

16.1 *Ammonium Oxalate, Saturated Solution*—Mix 50 g of ammonium oxalate and 1 L of water.

16.2 *Ammonium Oxalate Solution* (10 g/L)—Dissolve 10 g of ammonium oxalate in water and dilute to 1 L.

16.3 *Potassium Permanganate, Standard Solution* (0.1 *N*)—See 13.2.

17. Procedure

17.1 Transfer 10 g of the sample, weighed to 0.1 g, to a 400-mL beaker. Dissolve in 30 mL of HCl (1+1), keeping the beaker covered. Dilute to 200 mL. The solution at this point should be clear and transparent. Neutralize with NH_4OH . (Some zinc, because of its high concentration, will precipitate out as hydroxide at this point.) Add NH_4OH (sp gr 0.90) dropwise, stirring until the solution becomes clear. Heat nearly to boiling and add 75 mL of saturated ammonium oxalate solution. Boil until the precipitate assumes a dense crystalline appearance. Allow to settle on a steam bath for 20 min and then cool. Filter on close-grained paper and wash five times with a cold ammonium oxalate solution (10 g/L).

17.2 Carefully dissolve the precipitate from the filter paper, using alternately hot HCl (1+3) and hot water. Catch in a clean 400-mL beaker, dilute (if necessary) to a volume of about 250 mL, and add 25 mL of saturated ammonium

oxalate solution. Bring to a boil, and add NH_4OH in slight excess. Boil until the precipitate becomes crystalline and dense. Let stand 1 h on a steam bath. Allow to cool. Filter and wash with small amounts of hot water until all ammonium oxalate is removed.

17.3 Remove the paper from the funnel and spread it out on the inside of a 600-mL beaker above 300 mL of warm water to which has been added 20 mL of H_2SO_4 (1+1). Rinse the precipitate off the paper with water from a wash bottle and warm the solution to 80°C. Titrate at this temperature with $KMnO_4$ solution. As soon as a persistent pink end point is obtained, drop the paper into the liquid, rinse the side of the beaker, and quickly complete the titration.

17.4 **Calculation**—Calculate the percent of calcium, *C*, as CaO , as follows:

$$C = [(V_4 N \times 0.02804)/S_3] \times 100 \quad (5)$$

where:

V_4 = $KMnO_4$ solution required for titration of the sample, mL,

N = normality of the $KMnO_4$ solution, and

S_3 = specimen used, g.

LEAD

18. Procedure

18.1 Determine the lead content in accordance with Section 6 of Test Methods E 40.

NOTE 3—Lead may also be determined in accordance with Test Method E 68.

IRON

19. Procedure

19.1 Determine the iron content in accordance with Section 22 of Test Method E 40.

CADMIUM

20. Procedure

20.1 Determine the cadmium content in accordance with 18.1 and 18.3 of Test Method E 40.

NOTE 4—Cadmium may also be determined in accordance with Test Method E 68.

CHLORINE

21. Reagents

21.1 *Silver Nitrate Solution* (3.5 g/L)—Dissolve 3.5 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

21.2 *Sodium Chloride, Standard Solution* (0.01 *N*)—Dissolve 0.5850 g of pure sodium chloride ($NaCl$) in water and dilute to 1 L.

22. Procedure

22.1 Transfer 1.000 g of the sample to a 200-mL electrolytic beaker. Add 20 mL of water and then cautiously add 5 mL of nitric acid (HNO_3) (sp gr 1.42). Cover with a watch glass and heat on a steam bath with frequent stirring for 5 min, or until a clear solution results. Add 70 mL of water and cool to room temperature.

22.2 To the specimen and to a blank (prepared in similar fashion) add 5.0 mL of $AgNO_3$ solution (3.5 g/L) and stir. To

¹⁰ Kolthoff, I. M., and Sandell, E. B., *Textbook of Quantitative Inorganic Analysis*, 1945, p. 608.

the blank, add dropwise from a 10-mL buret, with thorough mixing, enough NaCl solution to develop a turbidity matching that of the sample. Keep the contents of both beakers well stirred and view against a black background in equal illumination.

22.3 *Calculation*—Calculate the percent of chlorine, C , as follows:

$$C = 0.035 V_s \quad (6)$$

where V_s = 0.01 N NaCl solution added to the blank, mL.

SULFUR

23. Reagents

23.1 *Barium Chloride Solution* (100 g/L)—Dissolve 117 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

23.2 *Bromine Water* (saturated).

24. Procedure

24.1 Transfer 20 g of the specimen, weighed to the nearest 0.1 g, to a 600-mL beaker. Cover the sample with 50 mL of saturated bromine water and then cautiously add HNO_3 (sp gr 1.42) until solution is complete.

24.2 Add 1 g of anhydrous sodium carbonate (Na_2CO_3) and boil down until salts just begin to separate, then add 75 mL of HCl (sp gr 1.19) and again boil down until salts begin to separate. Repeat this operation and, finally, dilute to 100

mL, heat until solution is complete, and filter into a 400-mL beaker through close-texture paper.

24.3 Wash the paper with hot water, make the filtrate alkaline with NH_4OH , and then just acid with HCl. Heat to boiling and hold at boiling temperature at least 5 to 10 min to drive out CO_2 , then slowly add with stirring 5 mL of BaCl_2 solution. Allow to stand at least 5 h (preferably overnight).

24.4 Filter on a weighed Gooch crucible, wash free from chlorides with hot water, dry, and ignite carefully at 900°C . Cool and weigh. The difference between the original and final weight is BaSO_4 .

24.5 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

24.6 *Calculation*—Calculate the percent of sulfur, S , as follows:

$$S = [(W_1 - B_4) \times 0.1374 / S_4] \times 100 \quad (7)$$

where:

W_1 = BaSO_4 , g,

B_4 = correction for blank, g,

S_4 = sample used, g, and

$0.1374 = S/\text{BaSO}_4 = 32.06/233.42$.

25. Keywords

25.1 calcium; chlorine; potassium ferrocyanide; zinc dust; zinc powder

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Standard Specification for Carbon Black Pigment for Paint¹

This standard is issued under the fixed designation D 561; the number immediately following the designation indicates the year of original adoption 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 specification covers the pigment commercially known as carbon black, which is suitable for use in the manufacture of protective or decorative coatings.

Referenced Documents

ASTM Standards:

- D 305 Test Method for Solvent-Extractable Material in Black Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1506 Test Method for Carbon Black—Ash Content³
- D 1509 Test Method for Carbon Black—Heating Loss³
- D 1514 Test Method for Carbon Black—Sieve Residue³

Composition and Properties

1.1 The pigment shall be made by burning natural gas (Type I) or oil (Type II) in such a manner as to form a deposit of carbon. It shall be free of adulterants and be in the form of powder or dustless pellets and shall conform to the requirements specified in Table 1.

1.2 The mass color and character of the tint and tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller when tested in accordance with Test Method D 387.

NOTE 1—For the tinting strength test a ratio of 100 parts of white to 1 part of black is usually suitable.

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 D01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 09.01.

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4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken.

4.2 At the option of the purchaser, the samples may be tested separately or, after blending the samples from the same production unit in equal quantities, tested as a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

- 5.1.1 Solvent Extractable Material—Test Method D 305.
- 5.1.2 Color and Tinting Strength—Test Method D 387.
- 5.1.3 Carbon Black—Ash Content—Test Method D 1506.
- 5.1.4 Heating Loss—Test Method D 1509.
- 5.1.5 Sieve Residue—Test Method D 1514.

TABLE 1 Composition and Properties

	Type I	Type II	ASTM Test Method
Ash, max, %	0.2 ^A	1.0 ^A	D 1506
Acetone extract, max, %	0.5 ^A	1.0 ^A	D 305
Moisture (loss at 105°C) max, %	8.0 ^B	8.0 ^B	D 1509
Coarse particles (total residue retained on 45- μ m (No. 325) screen, max), %	0.2	0.2	D 1514
Organic dyes	none	none	D 50

^A When mutually agreed upon by the purchaser and the seller, higher maximum ash and acetone extract values may be allowed if final product requirements necessitate the use of additional treating agents.

^B It may be necessary for the purchaser and the seller to agree upon a higher maximum moisture content in high-color black. High-color blacks are very hygroscopic and should be protected against moisture during storage.

...tared, using the alcohol-benzene wash solution for transferring the precipitate and washing the reaction flask. Wash the precipitate with successive portions of alcohol-benzene wash solution until a few millilitres of washings collected in a second suction flask are no longer alkaline to phenolphthalein. (Normally about 75 mL of wash solution is sufficient.) Do not allow air to be drawn through the crystals, as they are hygroscopic. Finally pour 25 mL of ether into the crucible and draw through the precipitate with the aid of suction.

6.4 Wipe the outer surface of the crucible with a clean cloth and place in a gravity convection oven at 60°C for 1 h (see 3). Cool to room temperature in a desiccator, and weigh.

NOTE 3—The precipitate is the alcoholate ($C_6H_4(COOH)_2 \cdot C_2H_5OH$), and the alcohol of crystallization will be slowly driven off on prolonged heating. It is safe, however, to dry the alcoholate at temperatures up to 60°C for as long as 1 h.

6.5 *Correction for Carbonates*—Coprecipitation of potassium carbonate (K_2CO_3) with the potassium alcohol phthalate may be a source of error. If a correction for K_2CO_3

is desired, proceed as follows: Dissolve the weighed precipitate in about 50 mL of distilled water that has been neutralized to phenolphthalein. Add 3 to 4 drops of phenolphthalein indicator solution, and if the solution is alkaline, titrate with 0.1 N HCl.

7. Calculation

7.1 Calculate the percent of phthalic anhydride A in the specimen as follows:

$$K = VN \times 0.1382$$

$$A = [(P - K) \times 0.5136/S] \times 100$$

where:

K = correction for K_2CO_3 (if determined), g,

V = HCl used for titration (see 6.5), mL,

N = normality of HCl,

P = potassium alcohol phthalate (see 6.5), g, and

S = specimen used, g.

8. Precision and Bias

8.1 Precision and bias are being determined.

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Standard Specification for Barium Sulfate Pigments¹

This standard is issued under the fixed designation D 602; the number immediately following the designation indicates the year of original adoption 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 specification covers the barium sulfate pigments commercially known as barytes and blanc fixe.

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 715 Test Methods for Analysis of Barium Sulfate Pigment³
- D 1208 Test Methods for Common Properties of Certain Pigments³

3. Composition and Properties

3.1 The pigment shall consist of barium sulfate (BaSO_4) without any admixture of other materials in the case of blanc fixe, and without any admixture of other materials not naturally occurring in the barite ore in the case of barytes, and which conform to the following requirements:

	Barytes	Blanc Fixe
Barium sulfate, min, %	94	97
Ferric oxide, max, %	0.05	0.02
pH, min	3.5	3.5

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 D01.31 on Pigment Specifications.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 602 - 41 T. Last previous edition D 602 - 42 (1975).

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

	Barytes	Blanc Fixe
Matter soluble in water, max, %	0.2	0.2
Moisture and other volatile matter, max, %	0.5	0.5
Coarse particles (total residue retained on No. 325 (45- μm sieve), max, %	0.5	0.5
Free silica (quartz, clays, or other foreign materials), max, %	2.0	2.0

3.2 In such physical properties as are specified by the purchaser, the pigment shall satisfactorily match a reference sample mutually agreed upon by the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples of each 10 000 lb (5000 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.2 *Coarse Particles in Pigment*—Test Methods D 185.

5.3 *Moisture*—Test Methods D 280.

5.4 *Barium Sulfate, Ferric Oxide, and Free Silica*—Test Methods D 715.

5.5 *Hydrogen Ion Concentration and Matter Soluble in Water*—Test Methods D 1208.

6. Keywords

6.1 barium sulfate; barytes; blanc fixe; pigments

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Standard Specification for Aluminum Silicate Pigments (Hydrous)¹

This standard is issued under the fixed designation D 603; the number immediately following the designation indicates the year of original adoption 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 covers the white pigments that consist substantially of natural hydrous aluminum silicate² of the 1:1 layer type), and are restricted to those minerals which conform to the chemical limits prescribed herein and which can be suitably processed to what is commercially known as paint pigment quality.

Referenced Documents

- 2.1 *ASTM Standards*:
D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out³
D 422 Method for Particle-Size Analysis of Soils⁴
D 718 Test Methods for Analysis of Aluminum Silicate Pigment³
D 1208 Test Methods for Common Properties of Certain Pigments³
D 1483 Test Method for Oil Absorption of Pigments by Gardner-Coleman Method³
D 2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment³
E 70 Test Method for pH of Aqueous Solution with the Glass Electrode⁵

3. Composition of Properties

3.1 *Preparation*—The pigment shall be made by grinding, milling, washing, purifying, size-fractionating, or otherwise processing, natural hydrous aluminum silicates, and shall conform to the composition requirements (weight percent) given in Table 1.

3.2 *pH*—The pH of a water slurry of the pigment shall be within a range as agreed upon between the purchaser and the seller.

3.3 *Water-Soluble Matter*—The water-soluble matter shall be not more than 0.50 %.

3.4 *Wet-Sieve Residue*—The pigment shall contain no more than 0.5 % wet-sieve residue retained on a 45- μ m (No. 325) sieve ("grit" or "coarse particles") except as may be agreed upon by the purchaser and the seller.

3.5 *Color*—The color (brightness, reflectance) shall con-

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 D01.31 on Pigment Specifications.

Current edition approved Sept. 20, 1966. Originally issued 1941. Replaces D 603 - 42 (1955).

² Synonymous terms are china clay and kaolinite.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ *Annual Book of ASTM Standards*, Vol 04.08.

⁵ *Annual Book of ASTM Standards*, Vol 15.05.

TABLE 1 Pigment Composition Requirements

	Ideal	Typical	Range	Max
Aluminum oxide, Al_2O_3 , %	39.50	38.8	37 to 42 ^A	...
Silicon dioxide, SiO_2 , %	46.54	45.4	48 to 43 ^B	...
Iron oxide, Fe_2O_3 , %	...	0.3	...	0.5
Titanium dioxide, TiO_2 , %	...	1.5	...	2.0
Calcium oxide, CaO , %	...	0.1	...	0.2
Sodium oxide, Na_2O , %	...	0.1	...	0.3
Potassium oxide, K_2O , %	...	0.1	...	2.0
Other oxides, %	...	trace	...	0.1
Free moisture (105°C), %	...	...	...	1.0
Loss on ignition (1000°C), %	13.96	13.8	...	15.0

^A Permitting up to 5 % excess Al_2O_3 , for example as allophane.

^B Permitting up to 5 % excess SiO_2 , for example as quartz.

form to the following requirements:

3.5.1 The color shall be equal, within agreed upon tolerances, to that of a reference standard agreed upon between the purchaser and the seller, or

3.5.2 The color shall be not less than a guaranteed minimum expressed as percent reflectance of standard illuminant C at 457 nm compared to a freshly smoked standard magnesium oxide surface by means of an accepted integrating sphere reflectance spectrophotometer⁶ or a monochromatic reflectance meter,⁶ or

3.5.3 The color shall be specified by actual determination of dominant wavelength, hue, and spectral efficiency as can be calculated from the tristimulus integration of the reflectance curve.

3.6 *Oil Absorption*—The oil absorption shall be equal, within agreed upon tolerances, to that of a reference standard agreed upon between the purchaser and the seller.

3.7 Aluminum silicate pigments may be furnished in several types or grades whose various properties are dependent in part on average size or particle size distribution about the average, or both. The particle size shall be equal within agreed upon tolerances to that of a reference standard agreed upon between the purchaser and the seller.

4. Number of Tests

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4540 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after

⁶ The General Electric Reflectance Spectrophotometer and the General Electric Reflectance Meter, respectively, have been found satisfactory for this purpose.

blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods, where applicable. Test procedures not covered by ASTM test methods shall be agreed upon between the purchaser and the seller, except as follows:

5.1.1 *Hydrogen Ion Concentration (pH)*—Prepare a

sample having 20 % solids, but otherwise in accordance with Test Methods D 1208. Determine the pH in accordance with Test Method E 70 except to maintain sufficient agitation of the slurry such that all particles are in suspension at time of measurement.

5.1.2 *Water-Soluble Matter*—Test Method D 2448.

5.1.3 *Coarse Particles*—Test Methods D 718.

5.1.4 *Oil Absorption*—Test Method D 281 or D 1483.

5.1.5 *Particle Size Distribution*—Method D 422.

5.1.6 *Chemical Analysis*—Test Method D 718.

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Standard Specification for Diatomaceous Silica Pigment¹

This standard is issued under the fixed designation D 604; the number immediately following the designation indicates the year of original adoption 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 specification covers two types of diatomaceous pigments for use in paints:

- 1 Type A—Standard fineness for general paint use.
- 2 Type B—Extra fine, for special uses.

NOTE 1—Silica pigments originating from crystalline rocks or sands are not covered by this specification.

NOTE 2—For additional information, see Test Methods D 719.

Referenced Documents

- 1 ASTM Standards:
 - D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
 - D 719 Test Methods for Analysis of Diatomaceous Silica Pigment³
 - D 1208 Test Methods for Common Properties of Certain Pigments³

Composition and Properties

3.1 The pigment shall be a diatomaceous silica especially produced by chemical, heat, and grading processes, and selected for use in paints and shall conform to the following requirements:

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 D01.31 on Pigment Specifications.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 604 - 41 T. Last previous edition D 604 - 42 (1975).

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

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Loss on ignition, max., %	1.0
Matter soluble in HCl (1+2), max, %	3.0
Moisture and other volatile matter, max, %	1.0
Volume of settled pigment in petroleum spirits after 1 h, min, mL:	
Type A	35
Type B	25
Coarse particles (total residue retained on a 45- μ m (No. 325) sieve), %:	
Type A	5.0 to 15.0
Type B	1.0 max

3.2 The color shall be within mutually agreed upon limits of a reference sample acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4540 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Test shall be conducted in accordance with the following ASTM test methods, where applicable. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.2 Coarse Particles—Test Methods D 185.

5.3 Loss on Ignition and Moisture—Test Methods D 1208.



Standard Specification for Magnesium Silicate Pigment (Talc)¹

This standard is issued under the fixed designation D 605; the number immediately following the designation indicates the year of original adoption 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 Fed. Spec. TT-P-403A. 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 covers pigments that consist substantially of natural hydrous magnesium silicate, and is restricted to those minerals that conform to the chemical limits prescribed herein and can be suitably processed to what is commercially known as paint pigment quality.

1.2 The following hazard caveat applies to the test method portion of this specification only. *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 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out²
- D 562 Test Method for Consistency of Paints Using the Stormer Viscometer³
- D 717 Test Methods for Analysis of Magnesium Silicate Pigment²
- D 1208 Test Methods for Common Properties of Certain Pigments²
- D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems³

3. Composition and Properties

3.1 The pigment shall be made by grinding or otherwise processing natural, hydrous magnesium silicate and shall conform to the following requirements:

	Weight %	
	min	max
Combined magnesium and calcium silicates (MgO plus SiO ₂ plus CaO)	88	...
Calcium oxide (CaO)	...	10
Aluminum and iron oxides (R ₂ O ₃)	...	6
Loss on ignition	...	7
Moisture and other volatile material	...	1

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 D01.31 on Pigment Specifications.

Current edition approved June 25, 1982. Published August 1982. Originally published as D 605 - 41 T. Last previous edition D 605 - 69 (1976).

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.01.

3.2 *Color*—The color shall be equal, within agreed upon tolerances, to that of a reference sample agreed upon between the purchaser and the seller.

3.3 *Coarse Particles*—The pigment shall contain no more than 2 % of coarse particles, retained on a 45- μ m (No. 325 sieve except as may be agreed upon between the purchase and the seller.

3.4 *Water-Soluble Matter*—The pigment shall contain no more than 1 % water-soluble matter except as may be agreed upon between the purchaser and the seller.

3.5 *Oil Absorption*—The oil absorption shall be equal within agreed upon tolerances, to that of a reference sample agreed upon between the purchaser and the seller.

3.6 *Consistency*—When consistency is included in the purchaser's specification, it shall be equal, within agreed upon tolerances, to that of a reference sample agreed upon between the purchaser and the seller.

3.7 *Fineness*—The paint fineness shall be equal, within agreed upon tolerances, to that of a reference sample agreed upon between the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguish between units of production appear, samples shall be taken from 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, two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities, the samples from the same production unit form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods, where applicable. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Color, Composition, and Coarse Particles*—Test Methods D 717.

5.1.2 *Water-Soluble Matter and Loss on Ignition*—Test Methods D 1208.

5.1.3 *Oil Absorption*—Test Method D 281.

5.1.4 *Consistency*—Use the pigment to prepare a mixture according to the following directions: Weigh 78 g of petroleum spirits and 80 g of linseed oil (viscosity, 21 to 24 P; ac number, 4 to 8) into a pint can and thoroughly mix with spatula. Weigh in 160 g of magnesium silicate and stir with spatula to wet all lumps. Stir with a motor-driven propeller

stirrer for 5 min at approximately 800 r/min. Weigh in additional 180 g of linseed oil and stir an additional 2 min with the electric stirrer. Determine the consistency in accordance with Test Method D 562.

5.1.5 *Fineness*—Test Method D 1210. The test mixture may be the same as that used for the consistency test.

5.1.6 *Moisture*—Test Methods D 280.

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Standard Specification for Wet Ground Mica Pigments¹

This standard is issued under the fixed designation D 607; the number immediately following the designation indicates the year of original adoption 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—Paragraph 4.1 was changed editorially in May 1987.

1. Scope

1.1 This specification covers two types of finely divided muscovite mica, commercially known as wet ground mica, suitable for use in the manufacture of protective coatings.

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under Test Conditions) in Pigment³
- D 716 Test Methods for Evaluating Mica Pigment³
- D 1208 Test Methods for Common Properties of Certain Pigments³

3. Composition and Properties

3.1 The pigments shall be made by wet grinding muscovite mica and shall conform to the requirements for properties prescribed as follows:

	Types	
	A Regular 325 M Grade	B Fine Grade
Apparent density, max, lb/ft ³ (g/cm ³)	12.0 (0.2)	12.0 (0.2)

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 D01.31 on Pigment Specifications.

Current edition approved June 25, 1982. Published August 1982. Originally published as D 106 - 41 T. Last previous edition D 106 - 75.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

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	Types	
	A Regular 325 M Grade	B Fine Grade
Moisture and other volatile matter, max, weight %	0.5	0.5
Grit, max, weight %	0.5	0.5
Coarse particles, max, weight %:		
Total residue retained on a No. 140 (106- μ m) sieve	0.1	0.01
Total residue retained on a No. 325 (45- μ m) sieve	12.0	3.0
Ignition loss, max, weight % (dry basis)	5.0	5.0

3.2 The color shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM methods. Test procedures not covered by ASTM methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Apparent Density*—Test Method D 716.

5.1.2 *Moisture*—Test Method D 280.

5.1.3 *Grit*—Test Method D 716.

5.1.4 *Coarse Particles*—Test Method D 185.

5.1.5 *Ignition Loss*—Test Method D 1208.

Standard Specification for Pure Toluidine Red Toner¹

This standard is issued under the fixed designation D 656; the number immediately following the designation indicates the year of original adoption 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 specification has been approved for use by agencies of the Department of Defense to replace Fed. Spec. TT-P-453. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

Scope

1.1 This specification covers the red pigment commercially known as pure toluidine red toner. The pigment may be purchased in the dry form or as a paste in oil.

Referenced Documents

1.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller³
- D 970 Test Methods for Para Red and Toluidine Red Pigments³

2. Composition and Properties

2.1 *Dry Pigment*—The pigment shall consist of toluidine red toner (1-(2-nitro-*p*-tolylazo)-2-naphthol) with or without admixture with a small quantity of (1-(2-nitro-*p*-methoxyphenyl-azo)-2-naphthol). It shall be free of any substrate and shall conform to the following requirements:

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 D01.31 on Pigment Specifications.

Current edition approved June 26, 1987. Published August 1987. Originally published as D 656 - 42 T. Last previous edition D 656 - 81.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

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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Ash, max, %	1.0
Moisture and other volatile matter, max, %	1.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	1.0
Paranitraniline red	none
Solubility in chloroform	complete
Resistance to acids and alkalis	to pass test
Identity test	to pass test

3.2 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate test methods. Test procedures not covered by ASTM methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Coarse Particles*—Test Methods D 185.

5.3 *Moisture*—Test Methods D 280.

5.4 *Color and Tint*—Test Method D 387.

5.5 *Solubility*—Test Methods D 970.

5.6 *Identity*—Test Methods D 970.

5.7 *Ash*—Test Methods D 970.



Standard Test Methods for Analysis of Barium Sulfate Pigment¹

This standard is issued under the fixed designation D 715; the number immediately following the designation indicates the year of original adoption 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, and Specification E 832 replaced Specification D 1100.

1. Scope

1.1 These test methods cover the analysis of barium sulfate pigment.

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

D 1193 Specification for Reagent Water⁴

D 1208 Test Methods for Common Properties of Certain Pigments³

E 832 Specification for Laboratory Filter Papers⁵

3. Significance and Use

3.1 These test methods are used to determine the purity of barium sulfate and to determine the concentration of known impurities. This information is useful to producers and users as an aid in the manufacture of coatings.

4. Purity of Reagents

4.1 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.

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

BARIUM SULFATE

5. Reagents

5.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

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

5.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

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

5.5 *Methyl Red Indicator Solution*—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

5.7 *Sodium Carbonate Solution* (30 g/L)—Dissolve 30 g of Na_2CO_3 in water and dilute to 1 L.

6. Procedure

6.1 Weigh to 0.1 mg approximately 0.5 g of the sample into a platinum crucible, add 3 g of Na_2CO_3 , mix thoroughly, and fuse until the melt is clear. Allow the melt to cool and then leach in a platinum dish with hot water until it is entirely disintegrated. Filter on a close-texture paper and wash thoroughly with hot Na_2CO_3 solution (30 g/L).

6.2 Transfer the filter paper containing the insoluble carbonates to a 250-mL beaker and acidify with concentrated HCl (sp gr 1.19). Wash the fusion crucible with HCl (sp gr 1.19) so that no barium is lost. Boil the solution, filter into a 600-mL beaker, and wash well with water. Add methyl red indicator solution, and add NH_4OH (sp gr 0.90) until the solution is slightly alkaline. Add 6 mL of HCl (1+1), and dilute to 300 mL.

6.3 Heat the solution to boiling, and add 5 g of $(\text{NH}_4)_2\text{SO}_4$ dissolved in 40 mL of water. If low-grade material is being analyzed, the $(\text{NH}_4)_2\text{SO}_4$ solution should be added drop by drop from a buret to minimize inclusion. If the barytes is a rather pure product (95 to 99 % BaSO_4), this is not necessary, since the only nonvolatile constituents of the solution will be barium salts. Allow the precipitate of BaSO_4 to digest for 4 h or overnight, and filter through a close-texture paper. Wash the precipitate with as little cold water as possible (consistent with the purity of the precipitate), ignite in an oxidizing atmosphere, and weigh as BaSO_4 .

7. Calculation

7.1 Calculate the percent of BaSO_4 as follows:

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 Committee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Sept. 26, 1986. Published November 1986. Originally published as D 715 - 43. Last previous edition D 715 - 75 (1981)¹.

² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

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 14.02.

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."

$$\text{BaSO}_4, \% = (P/S_1) \times 100$$

where:

P = BaSO_4 , g, and
 S_1 = sample used, g.

FERRIC OXIDE

Apparatus

9.1 *Colorimetric Apparatus*—Nessler type or other similar 100-mL colorimetric tubes.

Reagents

9.1 *Ammonium Thiocyanate Solution* (76.1 g/L)—Dissolve 76 g of ammonium thiocyanate (NH_4CNS) in water and dilute to 1 L.

9.2 *Iron, Standard Solution* (100 mL = 0.00002 g Fe)—Dilute and divide a ferric solution of known iron content so as to obtain 0.4 mg of iron. Dilute the solution to 2 L with water containing 200 mL of iron-free H_2SO_4 .

9.3 *Potassium Permanganate Solution* (0.1 g/L)—Dissolve 0.1 g of potassium permanganate (KMnO_4) in water and dilute to 1 L.

9.4 *Standard Color Solution*—Mix thoroughly 10 parts by volume of NH_4CNS and 90 parts by volume of standard iron solution (100 mL = 0.02 mg Fe). One hundred millilitres of the solution will thus contain 0.000018 mg of Fe.

9.5 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

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

10. Procedure

10.1 Dissolve the soluble portion of a 1-g specimen in H_2SO_4 (1+1), filter, and wash, keeping the volume of the solution under 100 mL. Oxidize any iron present in the filtrate by adding potassium permanganate (KMnO_4) until a faint pink color is obtained. Dilute the solution to 100 mL and pour into a buret graduated in 0.1-mL divisions.

10.2 Pour 100 mL of the standard color solution into a 100-mL colorimetric tube. Into a second colorimetric tube pour 10 mL of concentrated H_2SO_4 (sp gr 1.84) and 10 mL of NH_4CNS , dilute to 60 or 70 mL and then add the test solution from the buret until the depth of color thus produced on dilution to 100 mL exactly matches that of the standard. Record the number of millilitres required.

11. Calculation

11.1 Calculate the percent of Fe_2O_3 as follows:

$$\text{Fe}_2\text{O}_3, \% = [(I \times 1.4298)/S_2] \times 100$$

where:

I = iron in standard, g,
 $1.4298 = \frac{\text{Fe}_2\text{O}_3}{\text{Fe}} = \frac{159.694}{111.694}$, and
 S_2 = sample used, g.

HYDROGEN ION CONCENTRATION

12. Procedure

12.1 Determine the pH in accordance with Test Methods D 1208.

MATTER SOLUBLE IN WATER

13. Procedure

13.1 Determine the amount of water soluble material present in accordance with Test Methods D 1208.

MOISTURE AND OTHER VOLATILE MATTER

14. Procedure

14.1 Determine the moisture and other volatile matter in accordance with Test Method D 280.

COARSE PARTICLES

(Total Residue Retained on No. 325 (45- μm) Sieve)

15. Procedure

15.1 Determine the coarse particles in accordance with Test Methods D 185.

FREE SILICA

16. Reagent

16.1 *Heavy Liquid* (sp gr 2.9)—Dissolve 4 parts by weight of potassium iodide (KI) in 6 parts of warm water, and add, while stirring constantly, 5 parts of mercuric iodide (HgI_2). Evaporate over a hot plate or gas flame, protected by screen cloth, until a light crystalline scum forms. Cool, and filter through heavy paper. The solution will be a clear, deep, amber color of about 3.2 specific gravity. All filter washings showing a yellow color should be saved and evaporated. Determine the 3.2 specific gravity accurately by means of a specific gravity bottle, and correct the solution to a specific gravity of 2.9 at room temperature by adding water. Prepare approximately 200 mL of the solution.

17. Procedure

17.1 Carefully weigh 10 g of the finely ground dry barium sulfate pigment and transfer to a carefully dried 125-mL clear glass separatory funnel of the long narrow type. Add 100 mL of the heavy liquid (sp gr 2.9), stopper, and shake well to liberate any air bubbles that might adhere to the particles, and also to break up all agglomerates. Set the separatory funnel aside and allow to stand in a perpendicular position for 2 h, or for a sufficient time for the particles to rise or sink.

17.2 Draw off the heavy particles through the stopcock into a beaker along with 75 mL of the liquid. Add 50 mL of fresh heavy liquid (sp gr 2.9) to the float particles and liquid remaining in the separatory funnel and repeat the procedure as described in 17.1.

17.3 Draw off the heavy particles along with 75 mL of the liquid and add to those drawn off in accordance with 17.1, leaving the float particles in the funnel. Wash the particles remaining in the funnel with fresh heavy liquid (sp gr 2.9), filter through a No. 2/0 paper (see Specification E 832), and wash free of solution with warm water.

17.4 Transfer the filter paper and silica to an ignition cup, dry, and ignite carefully in an electric furnace or over a flame to keep mechanical loss at a minimum. Weigh the floated material, and transfer to a tared platinum dish or crucible. Add 20 mL of hydrofluoric acid (HF) and 3 drops of H_2SO_4 (1+1), evaporate to dryness, and ignite carefully to expel all

fumes. Cool and weigh. The loss in weight represents the free silica in the floated material.

18. Calculation

18.1 Calculate the percent of free SiO_2 as follows:

$$\text{Free SiO}_2, \% = (A/S_3) \times 100$$

where:

A = SiO_2 , g, and

S_3 = sample used, g.

19. Precision and Bias

19.1 The precision and bias of these test methods has not been determined.

20. Keywords

20.1 analysis of pigment; barium sulfate; barium sulfate content; barium sulfate pigment; ferric oxide in barium sulfate; free silica in barium sulfate.

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Designation: D 716 - 86 (Reapproved 1991)^{e1}

Standard Test Methods for Evaluating Mica Pigment¹

This standard is issued under the fixed designation D 716; the number immediately following the designation indicates the year of original adoption 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 These test methods cover the evaluation of mica pigment.

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

3. Significance and Use

3.1 These test methods are used to determine apparent density of mica pigments and the grit level. This information is significant to the user of mica pigments for inclusion in coatings.

APPARENT DENSITY

4. Apparatus

4.1 *Volumeter*⁵—A Scott volumeter or similar apparatus modified as follows: The screen used shall conform to the requirements of a No. 40 (425- μ m) sieve as prescribed in Specification E 11. The funnel below the screen shall be replaced by a conical funnel having a bottom opening 1 in. (25 mm) in diameter. It may be found necessary to replace the top glass baffle with one that is longer to ensure that all of the sifted mica is caught.

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 Sept. 26, 1986. Published November 1986. Originally published as D 716 - 43. Last previous edition D 716 - 75 (1981)^{e1}.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ The volumeter is no longer generally available. As of June 1986 only one supplier, Sargent Welch Co., Skokie, IL, was known. The device is Scott-Schaeffer and White Volumeter listed on p. 932 of Catalog No. 133 (1985-86).

5. Procedure

5.1 Transfer convenient quantities of the mica pigment to the funnel of the modified volumeter and brush the pigment through the screen with a camel-hair brush until the receiver is slightly more than full. Scrape off the excess and weigh the pigment. Care must be taken not to jar the apparatus during the procedure.

6. Calculation

6.1 Calculate the apparent density of the mica pigment and convert to pounds per cubic foot.

7. Report

7.1 Report the mean of three determinations as the apparent density of the mica pigment.

8. Reproducibility of Results

8.1 The mean of the three determinations should check within 5 %.

MOISTURE AND OTHER VOLATILE MATTER

9. Procedure

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

GRIT

10. Apparatus

10.1 *Beaker*, 600-mL, low-form.

10.2 *Metal Tubing*—An L-shape metal tube (Fig. 1) $\frac{1}{4}$ in. (6.4 mm) in outside diameter with a foot 1 in. (25 mm) in length. The foot of the tube shall be drilled to 0.173 in. (4.4 mm) (No. 17 drill) in inside diameter for a depth of $\frac{1}{2}$ in. (12.7 mm). The tube shall be placed in the beaker with the long arm in the vertical position, and the foot of the L parallel to the bottom of the beaker and perpendicular to the radius of the bottom at a point such that there is about $\frac{1}{16}$ -in. (1.5-mm) clearance between the tubing and both the bottom and side of the beaker. The center of the foot shall be placed 90° from the lip of the beaker.

11. Procedure

11.1 Transfer 10 g of the sample to the 600-mL beaker, add 100 mL of water, and swirl the beaker until the mica is dispersed. Insert the metal tubing into the beaker as described in 10.2 and connect the upper end to a water faucet. Regulate the flow of water by a constant head to a rate of 800 mL/min over the lip of the beaker. When the elutriation has

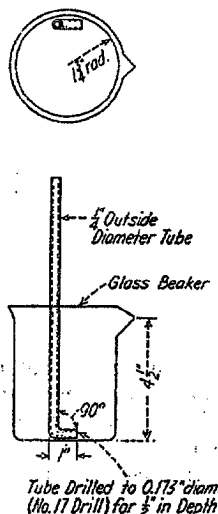


FIG. 1 Grit Test Apparatus

progressed so that the body of the water in the beaker is clear, stop the flow, and lower the water level to prevent spilling.

11.2 Swirl the beaker again to bring the remaining mica pigment into suspension and repeat the elutriation procedure as described in 11.1 three times. After final clearing, decant part of the water, and filter the residue. Transfer the paper and residue to a weighed crucible, and ash slowly, cool, and weigh.

12. Calculation

12.1 Calculate the percent grit, G , as follows:

$$G, \% = [(A - B)/S] \times 100$$

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where:

A = ash, g,

B = filter paper ash, g, and

S = sample used, g.

COARSE PARTICLES

13. Procedure

13.1 Determine the coarse particles in accordance with Test Methods D 185.

COLOR

14. Standard Pigments

14.1 Standard Mica Pigment.

14.2 Standard Zinc Oxide.

15. Procedure

15.1 Compare pastes made by rubbing a standard mica pigment and standard zinc oxide, mutually agreed upon by the purchaser and the seller, in linseed oil with a similar paste using the sample to be tested. Rub 9 g of zinc oxide, and 3 g of mica pigment on a glass plate with a glass muller or rubber spatula to avoid staining, with 4 mL of bleached linseed oil. Spread the pastes adjacently on a clear glass plate and determine the color by viewing the spreadouts on the glass.

16. Precision and Bias

16.1 Insufficient data are available to develop acceptable precision and bias statements. The information presented in Section 8 (Reproducibility of Results) was obtained in 1943, and no documentation is currently available. The information will remain in Section 8.

17. Keywords

17.1 grit, detection in mica pigment; mica pigment, analysis of; pigment

Standard Test Methods for Analysis of Magnesium Silicate Pigment¹

This standard is issued under the fixed designation D 717; the number immediately following the designation indicates the year of original adoption 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 January 1991.

1. Scope

1.1 These test methods cover the analysis of magnesium silicate pigment.

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 234 Specification for Raw Linseed Oil²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 718 Test Methods for Analysis of Aluminum Silicate Pigment³
- D 1193 Specification for Reagent Water⁴
- D 1208 Test Methods for Common Properties of Certain Pigments³
- D 2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment³
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵

3. Significance and Use

3.1 These test methods may be used to confirm the stated SiO_2 , CaO , and MgO content of magnesium silicate for quality control.

4. Purity of Reagents

4.1 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.

4.2 Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

5. Apparatus

- 5.1 *Platinum Crucible.*
- 5.2 *Electric Furnace, capable of 1200°C.*

SILICON DIOXIDE

6. Reagents

- 6.1 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).
- 6.2 *Hydrochloric Acid* (1+20)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 20 volumes of water.
- 6.3 *Hydrofluoric Acid* (48 %)—Concentrated hydrofluoric acid (HF).
- 6.4 *Sodium Carbonate* (Na_2CO_3).
- 6.5 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

7. Procedure

7.1 Transfer 1 g of the sample weighed to 0.1 mg to a platinum crucible and fuse with 5 g of Na_2CO_3 until the entire contents of the crucible are in a molten state. Continue heating for 20 min. Keep a close-fitting platinum cover on the crucible during the fusion. When the fusion is complete, allow the crucible and contents to cool, and transfer to a 600-mL porcelain casserole containing 200 mL of water (Note 1). Boil until the melt is disintegrated.

NOTE 1—If, during the cooling period, the crucible is partially immersed several times in cold water to chill the outer portions of the melt, the subsequent removal of the melt is facilitated. Do not allow the water to enter the crucible while the contents are hot to avoid spattering.

7.2 Remove crucible and lid, being careful to scrub and rinse out any adhering particles of the melt. Carefully acidify the contents of the casserole with concentrated HCl (sp gr

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.

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² *Annual Book of ASTM Standards*, Vol 06.03.

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*, Vols 06.01 and 14.02.

⁶ "Reagent Chemicals, American Chemical Society Specification," 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."

1.19); introduce the HCl in small portions, keeping a watch glass over the crucible to avoid loss by spattering. Add 30 mL of HCl in excess and evaporate to dryness on a steam bath; take care to break up any crusts that form. When the material appears completely dry, and no odor of HCl can be detected, remove the casserole from the steam bath, and allow to cool.

7.3 Wash down the sides of the casserole with 20 mL of HCl (sp gr 1.19) and then with water. Repeat the evaporation as described in 7.2, then bake for 1 h in an oven at 105°C. Cool the residue, drench with 25 mL of HCl (sp gr 1.19), add 175 mL of water, and warm, while stirring, until all soluble salts are dissolved. Filter off the silica on a close-texture paper, wash five times with HCl (1+20), wash five times with hot water, and reserve the filtrate for determination of other oxides (Section 9).

7.4 Transfer the paper and washed silica to a clean platinum crucible, ignite, first gently until the filter paper is consumed, and then at 1200°C for 20 min, cool, and weigh. Moisten the residue with water, add 5 drops of H₂SO₄ (sp gr 1.84), and 15 mL of HF. Evaporate to dryness on a steam bath, heat gently until H₂SO₄ has been expelled, and ignite at 1200°C for 5 min. Cool and weigh. The loss in weight represents the SiO₂.

8. Calculation

8.1 Calculate the percent of silica as follows:

$$\text{SiO}_2, \% = (P/S) \times 100$$

where:

P = SiO₂, g, and

S = sample used, g.

AMMONIUM HYDROXIDE GROUP (Alumina and Iron Oxide)

9. Reagents

9.1 *Ammonium Chloride Solution* (2 g/100 mL)—Dissolve 2 g of ammonium chloride (NH₄Cl) in 100 mL of water.

9.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

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

9.4 *Methyl Red Indicator Solution*—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

9.5 *Potassium Pyrosulfate* (K₂S₂O₇).

10. Procedure

10.1 If an appreciable residue remains after the treatment with HF in accordance with 7.4, fuse the residue with a small amount of K₂S₂O₇ until it is dissolved. Leach the pyrosulfate melt out of the crucible with water and combine the solution with the filtrate reserved in accordance with 7.3.

10.2 Using the methyl red indicator solution, neutralize the combined solutions from the silica determination with NH₄OH and add an excess of 2 drops. Bring to a boil adding NH₄OH 1 drop at a time if necessary to maintain a slight alkalinity. Allow the precipitate to settle (not more than 5 min) and filter. Wash four times with hot NH₄Cl solution.

10.3 Set aside the filtrate and transfer the precipitate and filter paper to the same beaker in which the first precipitation

was effected. Dissolve the precipitate with 40 mL of hot HCl (1+3), dilute the solution to about 100 mL, and reprecipitate the hydroxides as described in 10.2. Filter and wash the precipitate four times with hot NH₄Cl. Combine the filtrate and washings with the filtrate set aside and reserve for the determination of CaO (Section 10).

10.4 Place the precipitate in a weighed platinum crucible, heat slowly until the papers are charred, and finally ignite to constant weight at 1050 to 1100°C with care to prevent reduction. Cool and weigh as R₂O₃ (aluminum and iron oxides).

11. Calculation

11.1 Calculate the percent of R₂O₃ as follows:

$$\text{R}_2\text{O}_3, \% = (P_2/S) \times 100$$

where:

P_2 = R₂O₃, g, and

S = sample used, g.

CALCIUM OXIDE

12. Reagents

12.1 *Ammonium Hydroxide* (1+1)—Mix 1 volume of concentrated NH₄OH (sp gr 0.90) with 1 volume of water.

12.2 *Ammonium Oxalate Solution*—((NH₄)₂C₂O₄·H₂O), saturated.

12.3 *Ammonium Oxalate Solution* (1 g/L)—Dilute 1.0 g of ammonium oxalate ((NH₄)₂C₂O₄·H₂O) in 1 L of water.

12.4 *Hydrochloric Acid* (1+3)—See 9.3.

12.5 *Hydrochloric Acid* (1+1)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1 volume of water.

12.6 *Methyl Red Indicator Solution*—See 9.4.

13. Procedure

13.1 Acidify the combined filtrates obtained in the precipitations of the ammonium hydroxide group (Section 10) with HCl and evaporate them to a volume of about 300 mL. Add 5 mL of HCl (1+1), a few drops of methyl red indicator solution, and 30 mL of warm ammonium oxalate solution (saturated). Heat the solution to 70 to 80°C and add NH₄OH (1+1) dropwise while stirring, until the color changes from red to yellow. Allow the solution to stand without further heating for 1 h (no longer) with occasional stirring during the first 30 min. Filter and wash moderately with cold dilute ammonium oxalate solution. Reserve the filtrate and washings.

13.2 Transfer the precipitate and filter paper to the beaker in which the precipitate was effected. Dissolve the oxalate in 40 mL of hot HCl (1+3) and macerate the filter paper. Dilute to 200 mL with water, add a few drops of methyl red indicator solution and 5 mL of ammonium oxalate solution (saturated). Heat the solution nearly to boiling, and precipitate calcium oxalate again by neutralizing the acid solution with NH₄OH as described in 13.1. Allow the solution to stand 1 to 2 h and wash as before. Combine the filtrate with that already obtained and reserve for the determination of MgO (Section 16).

13.3 Dry the precipitate in a weighed covered platinum crucible. Char the paper without flaming, burn the carbon at as low a temperature as possible, and, finally, heat with the

sample tightly covered in an electric furnace or over a blast lamp at a temperature of 1100 to 1200°C. Cool in a desiccator and weigh as CaO. Repeat the ignition to a constant weight.

Calculation

14.1 Calculate the percent of CaO as follows:

$$\text{CaO, \%} = (P_3/S) \times 100$$

where:

P_3 = CaO, g, and

S = sample used, g.

MAGNESIUM OXIDE

Reagents

15.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

15.2 *Ammonium Hydroxide* (5+95)—Mix 5 volumes of concentrated NH₄OH (sp gr 0.90) with 95 volumes of water.

15.3 *Diammonium Phosphate Solution* (250 g/L)—Dissolve 250 g of diammonium phosphate (NH₄)₂HPO₄ in enough water to yield 1 L of solution.

15.4 *Hydrochloric Acid* (1+3)—See 9.3.

Procedure

16.1 Acidify the filtrates reserved in the determination of CaO (Section 13) with HCl and concentrate to about 400 mL. Add to this solution about 50 mL of (NH₄)₂HPO₄ (250 g/L) and if the solution becomes alkaline, add HCl (1+3) to keep the solution acidic. Cool the solution by placing the beaker in a cold water trough or in a tray of ice water. After cooling add NH₄OH drop by drop, while stirring constantly, until the crystalline magnesium ammonium phosphate begins to form, and then in moderate excess (5 to 10 % of the volume of the solution), the stirring being continued for several minutes. Set the solution aside for at least 8 h in a cool atmosphere.

16.2 Filter the solution on a close-texture paper and wash with NH₄OH (5+95). Unfold the filter paper and, using hot water, wash the precipitate into the beaker in which the precipitation was effected. Rinse the filter paper with hot HCl (1+3) and again with hot water; if necessary add more hot HCl (1+3) to dissolve the precipitate. Dilute the solution to about 250 mL, add 1 mL of (NH₄)₂HPO₄ solution, and then add concentrated NH₄OH drop by drop, while stirring constantly, until the precipitate is again formed as described in the NH₄OH is in moderate excess. Cool and allow to stand for about 4 h.

16.3 Filter the solution on a close-texture paper and wash six times with NH₄OH (5+95). Transfer the precipitate wrapped in the moist paper to a weighed platinum or porcelain crucible, slowly char the paper without allowing it to ignite and carefully burn off the carbon over a gradually increasing flame which shall never heat the crucible to more than the faintest red. Finally ignite at 1000 to 1100°C to constant weight, taking care to avoid melting the pyrophosphate.

Calculation

17.1 Calculate the percent of MgO to 0.1 as follows:

$$\text{MgO, \%} = [(P_4 \times 0.3623)/S] \times 100$$

where:

P_4 = Mg₂P₂O₇, g, and

S = sample used, g.

$$\frac{\text{molecular weight } 0.2 \text{ MgO}}{\text{molecular weight } \text{Mg}_2\text{P}_2\text{O}_7} = \frac{2 \times 40.32}{222.60} = 0.3623$$

17.2 Consider the sum of MgO, CaO, and SiO₂ as combined magnesium and calcium silicate.

LOSS ON IGNITION

18. Procedure

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

MOISTURE AND OTHER VOLATILE MATTER

19. Procedure

19.1 Determine the moisture and other volatile matter by Test Methods D 280.

COARSE PARTICLES

(Total Residue Retained on a No. 325 (45-μm) Sieve)

20. Procedure

20.1 Determine the coarse particles in accordance with Test Methods D 718, except that the specimen weight should be adjusted to provide appropriate sensitivity for the residual weight determination. Any difficulty encountered in wetting-out the specimen can be overcome by prewetting with a 50+50 mixture of acetone or alcohol and water.

MATTER SOLUBLE IN WATER

21. Procedure

21.1 Determine the matter soluble in water by Test Method D 2448 or alternatively by Test Methods D 1208.

COLOR

22. Standard Pigment

22.1 *Standard Extender Pigment.*

22.2 *Standard Zinc Oxide.*

23. Procedure

23.1 Carefully weigh out the proportions of the standard extender pigment and standard zinc oxide mutually agreed upon, and rub up to a fairly stiff paste with a glass muller on a glass plate or stone slab with raw linseed oil conforming to Specification D 234. Note the volume of the oil required. Prepare a similar paste with the sample, using the same weight of pigment, volume of oil, and number of strokes of the glass muller as used in the preparation of the paste of the standard pigments. Spread the pastes adjacently on a microscope slide, draw a scraper lightly across them so as to present them on an even plane, and judge the color immediately.

24. Dry Brightness

24.1 The test sample and reference sample shall be prepared into suitable smooth, dry, packed surfaces in

accordance with accepted practice and tested for reflectance using the green filter in accordance with Test Method E 97.

PRECISION

25. Precision and Bias

25.1 Data are not available to determine the precision and bias of these test methods. There are no plans at present to

obtain such information. The methods have been in use for several years and are considered acceptable.

26. Keywords

26.1 calcium oxide in magnesium silicate; magnesium oxide in magnesium silicate; magnesium silicate pigment, analysis of; silicon dioxide, analysis of

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Standard Test Methods for Analysis of Aluminum Silicate Pigment¹

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^{e1} NOTE—Keywords were added editorially in January 1991.

Scope

- 1.1 These test methods cover the analysis of aluminum silicate pigment.
- 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.*

Referenced Documents

- 2.1 *ASTM Standards:*
 - D 234 Specification for Raw Linseed Oil²
 - D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
 - D 717 Test Methods for Analysis of Magnesium Silicate Pigment³
 - D 1193 Specification for Reagent Water⁴
 - D 1208 Test Methods for Common Properties of Certain Pigments³
 - D 2448 Test Methods for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment³
 - E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁵

3. Significance and Use

3.1 These test methods may be used to confirm the stated aluminum oxide and SiO₂ content of aluminum silicate for quality control.

4. Purity of Reagents

4.1 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,⁶

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 April 25, 1986. Published June 1986. Originally published as D 718 - 43. Last previous edition D 718 - 73 (1979)^{e1}.

² *Annual Book of ASTM Standards*, Vol 06.03.

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 14.02.

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."

where such specifications are available. 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 Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

5. Apparatus

- 5.1 *Platinum Crucible.*
- 5.2 *Electric furnace* (or gas burner), capable of 1050 to 1100°C.
- 5.3 *Volumetric flask*, 100 and 250 mL.
- 5.4 *Colorimeter*, with transmission range from 400 to 550 nm.
- 5.5 *High Silica Crucible.*

SILICON DIOXIDE

6. Procedure

- 6.1 Determine the silicon dioxide content in accordance with Test Methods D 717.

ALUMINUM OXIDE

7. Reagents

- 7.1 *Ammonium Acetate* (20 %)—Dissolve 200 g of ammonium acetate (NH₄C₂H₃O₂) in 1 L of distilled water.
- 7.2 *Ammonium Chloride Solution* (20 g/L)—Dissolve 20 g of ammonium chloride (NH₄Cl) in water and dilute to 1 L.
- 7.3 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).
- 7.4 *Diphenylamine Indicator Solution* (1 g/100 mL)—Dissolve 1 g of diphenylamine in 100 mL of concentrated sulfuric acid (H₂SO₄, sp gr 1.84).
- 7.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).
- 7.6 *Hydrochloric Acid* (1+3)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 3 volumes of water.
- 7.7 *Hydrofluoric Acid* (48 %)—Concentrated hydrofluoric acid (HF).
- 7.8 *Hydrogen Peroxide* (H₂O₂, 3 %, freshly prepared)—Mix 1 volume of H₂O₂ (30 %) with 9 volumes of distilled water.
- 7.9 *Hydroxylamine Hydrochloride* (10 %)—Dissolve 10 g of NH₂OH·HCl in 100 mL of distilled water. Prepare fresh weekly.
- 7.10 *Iron Standard*—Dissolve 0.1 g of analytical grade iron wire in 10 mL of HCl (1+1) and dilute to 1 L. Each millilitre contains 0.1 mg of Fe.
- 7.11 *Mercuric Chloride Solution* (HgCl₂), saturated.

7.12 *Methyl Red Indicator Solution*—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

7.13 *Potassium Dichromate, Standard Solution* (0.05 N)—Dissolve 2.457 g of potassium dichromate ($K_2Cr_2O_7$) in water and dilute to 1 L. Standardize against National Bureau of Standards' standard sample No. 27b of Sibley iron ore, using such an amount as to give approximately the same titration as the sample to be analyzed.

7.14 *Phosphoric Acid* (H_3PO_4 , 85 %)—Concentrated phosphoric acid.

7.15 *Potassium Pyrosulfate* ($K_2S_2O_7$).

7.16 *o-Phenanthroline* (0.1 %)—Dissolve 1.0 g of o-phenanthroline in 1 L of hot distilled water.

7.17 *Stannous Chloride Solution* (5 g/100 mL)—Dissolve 5 g of stannous chloride ($SnCl_2 \cdot 2H_2O$) in 10 mL of concentrated HCl (sp gr 1.19) and dilute to 100 mL with water. Add scraps of iron-free granulated tin, and boil until the solution is clear. Keep the solution in a closed dropping bottle containing metallic tin.

7.18 *Sulfuric Acid* (1+1)—Add carefully 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) to 1 volume of distilled water.

7.19 *Sulfuric Acid* (1+9)—Add carefully 1 volume of concentrated H_2SO_4 (sp gr 1.84) to 9 volumes of distilled water.

8. Procedure

8.1 If an appreciable residue remains after the treatment with HF in accordance with 7.4 of Test Methods D 717, fuse the residue with a small amount of $K_2S_2O_7$ until it is dissolved. Leach the pyrosulfate melt out of the crucible with water and combine the solution with the filtrate reserved in accordance with 7.3 or Test Methods D 717.

8.2 Bring the volume of the combined solution to 250 mL, and, if necessary, add HCl in order to ensure a total of 10 to 15 mL of HCl, add a few drops of methyl red indicator solution, and heat to boiling. Add concentrated NH_4OH (sp gr 0.90) dropwise until 1 drop changes the color of the solution to a distinct yellow. Reheat the solution containing the precipitated hydroxides to boiling, boil for 1 or 2 min, and filter. Wash the precipitate once by decantation and then slightly on the filter with hot NH_4Cl .

8.3 Transfer the precipitate and paper to the original beaker in which the precipitation was made. Dissolve the precipitate in hot HCl (1+3), dilute to 100 mL, and precipitate again as described in 8.2. After filtering, wash the precipitate ten times with small portions of hot NH_4Cl solution. Transfer the precipitate to a weighed platinum crucible, heat slowly until the paper is charred, and finally ignite to constant weight at 1050 to 1100°C in an electric furnace or over a burner, taking care to avoid reduction. Weigh the precipitate as $Al_2O_3 + TiO_2 + Fe_2O_3$.

8.4 Fuse the combined oxides from 8.3 with 9 to 10 g of $K_2S_2O_7$ in a platinum crucible, starting at low temperature and increasing the heat gradually until the oxides have all dissolved. Take up the melt with 5 mL of H_2SO_4 (1+1) in 150 mL of water and warm to effect solution. Transfer the solution to a 250-mL volumetric flask, dilute to volume, and mix well. Reserve for TiO_2 and Fe_2O_3 determinations.

8.5 Determine the percent of TiO_2 as follows: Pipet an aliquot containing 0.2 to 3.0 mg of TiO_2 into a 100-mL

volumetric flask. For samples containing 0.5 to 3 % TiO_2 , a 20-mL aliquot is suitable. Add 5 mL of H_2O_2 (3 %), dilute to volume with H_2SO_4 (1+9) and mix well. Obtain the colorimetric reading in a suitable colorimeter using a filter with transmission limits of 400 to 450 nm or at 410 nm with a prism or grating spectrometer. Compare the readings to a curve plotted from a set of TiO_2 standards similarly treated and read on the same instrument.

8.6 Determine the percent of Fe_2O_3 as follows: Pipet a 25-mL aliquot into a 100-mL volumetric flask. Add the following in the order given, mixing well after each addition: 2 mL of $NH_2OH \cdot HCl$ (10 %), 10 mL of $NH_4C_2H_3O_2$ (20 %), and 10 mL of o-phenanthroline (0.1 %). Roll a small piece of congo red paper into a ball and introduce into the flask. Add concentrated NH_4OH dropwise until indicator turns red and 1 drop in excess. Dilute to volume and let stand for 10 to 20 min. Obtain the colorimetric reading in a suitable colorimeter using a filter with transmission limits 485 to 550 nm, or at 510 nm with a prism or grating spectrometer. Compare the readings to a curve plotted from a set of Fe_2O_3 standards similarly treated and read on the same instrument.

8.7 Alternatively the Fe_2O_3 may be determined by titration as follows: Fuse 1 g of sample with 10 g of $K_2S_2O_7$ in a high-silica crucible starting at low temperature and increasing the heat gradually until the crucible glows with a dull red color and decomposition is judged to be complete. Leach the melt with 10 mL of concentrated HCl in 100 mL of water and digest at low heat to disintegrate the cake. Filter and wash free of chlorides with hot water. Save filtrate. Ignite the insoluble residue in a platinum crucible. Treat with 5 mL of H_2SO_4 (1+1) and 15 mL of HF (48 %), and heat until fumes of H_2SO_4 appear. Bring the residue into solution with the addition of a few drops more of H_2SO_4 (1+1) and combine the solution with the bulk of the iron. Add 5 mL of HCl to the combined solutions and evaporate to 50-mL volume.

8.8 To the hot solution add $SnCl_2$ solution, dropwise, while stirring, until the solution is decolorized, and then add 2 to 3 drops more. Cool the solution to room temperature, wash down the inside of the beaker with water, and add at one stroke 10 mL of cool $HgCl_2$ solution (saturated). Stir the solution vigorously for 1 min, add 5 mL of phosphoric acid (85 %) and 3 drops of diphenylamine indicator solution. Titrate with 0.05 N $K_2Cr_2O_7$ solution to an intense deep-blue end point that remains unchanged on further addition of $K_2Cr_2O_7$ solution.

8.9 Calculate the percent of Fe_2O_3 as follows:

$$Fe_2O_3 \% = [(N \times V \times 0.0798)/S] \times 100$$

where:

S = sample used, g,

V = $K_2Cr_2O_7$, mL, and

N = normality of the $K_2Cr_2O_7$.

8.10 Calculate the percent of Al_2O_3 as follows:

$$Al_2O_3 \% = 100(P/S) - C$$

where:

P = weight of combined oxides $Al_2O_3 + TiO_2 + Fe_2O_3$,

S = specimen weight, g, and

C = $Fe_2O_3 + TiO_2$, %.

LOSS ON IGNITION

Procedure

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

MOISTURE AND OTHER VOLATILE MATTER

10. Procedure

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

COARSE PARTICLES

(Total Residue Retained on a No. 325 (45- μ m) Sieve)

11. Apparatus

11.1 *Sieve*—A No. 325 (45- μ m) 3-in. (76 mm) diameter high form sieve conforming to Specification E 11, shall be used.

11.2 *Stirring Apparatus*,⁷ consisting of:

11.2.1 *Mixing Cup*—A stainless steel cup, about 7 in. (178 mm) deep, and slightly tapered from an outside diameter at the top of about 4 in. (102 mm) to about 2 3/4 in. (70 mm) at the bottom, such as is characteristic of a usual malted-milk type mixing cup.

11.2.2 *Mechanically Operated Stirring Device*, in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 r/min without load. The shaft shall be equipped with a replaceable stirring button not more than 1 in. (25 mm) in diameter, and of such length that the stirring button shall operate not less than 3/4 in. (19.0 mm) nor more than 1 1/2 in. (38 mm) above the bottom of the dispersion cup. A common malted milk-type mixer of high quality meets this specification.

12. Procedure

12.1 Dry a No. 325 (45- μ m) sieve in an oven at 105 $\pm$ 2°C, cool, and weigh accurately. Transfer 100 $\pm$ 0.5 g of the specimen to the mixing cup containing approximately 250 mL of water to which has been added about 2 drops of 0.5 % solution sodium silicate,⁸ and stir for about 10 min at high speed. Pour the resulting slurry slowly through the sieve and

⁷ The Hamilton Beach Mixer or equivalent has proven satisfactory for this purpose.

⁸ "O" Brand sodium silicate, manufactured by the Philadelphia Quartz Co., Public Ledger Building, Independence Square, Philadelphia, PA, or equivalent has been found satisfactory for this purpose.

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wash out the mixing cup carefully (a polyethylene squeeze-type wash bottle is very convenient) to ensure complete transfer of the specimen to the sieve. Spray the sieve surface gently with a low pressure fan-shaped spray (tap water at about 5 psig (30 kPa) back pressure) and continue washing until all the pigment has been washed through and the water passing the sieve is clear. Dry the sieve for 1 h at 105 to 110°C, cool and weigh.

12.2 Calculation:

Percent of coarse particles

$$= (\text{increase in weight of sieve/specimen weight}) \times 100$$

MATTER SOLUBLE IN WATER

13. Procedure

13.1 Determine the matter soluble in water by Test Method D 2448.

COLOR

14. Standard Pigment

14.1 *Standard Extender Pigment*.

14.2 *Standard Zinc Oxide*.

15. Procedure

15.1 Carefully weigh out the proportion of the standard extender pigment and standard zinc oxide mutually agreed upon, and rub up to a fairly stiff paste with a glass muller on a glass plate or stone slab with raw linseed oil conforming to Specification D 234. Note the volume of the oil required. Prepare a similar paste with the sample using the same weight of pigment, volume of oil, and number of strokes of the glass muller as used in the preparation of the paste of the standard pigments. Spread the pastes adjacently on a microscope slide, draw a scraper lightly across them to present them on an even plane, and judge the color immediately.

PRECISION AND BIAS

16. Precision and Bias

16.1 Data are not available to determine the precision and bias of these methods. There are no plans at present to obtain such information. The methods have been in use for several years and are considered acceptable.

17. Keywords

17.1 aluminum oxide in aluminum silicate pigment; aluminum silicate pigment, analysis of; pigment, aluminum silicate



Standard Test Methods for Analysis of Diatomaceous Silica Pigment¹

This standard is issued under the fixed designation D 719; the number immediately following the designation indicates the year of original adoption 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 analysis of diatomaceous silica pigment.

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 234 Specification for Raw Linseed Oil³

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)³

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments⁴

D 1208 Test Methods for Common Properties of Certain Pigments⁴

3. Significance and Use

3.1 These test methods are used to determine the purity and some physical properties of diatomaceous silica pigment. The information is significant to pigment producers and coatings manufacture.

MOISTURE AND OTHER VOLATILE MATTER

4. Procedure

4.1 Determine the moisture and other volatile matter by Test Methods D 280.

LOSS ON IGNITION

5. Procedure

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

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.

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² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.02.

MATTER SOLUBLE IN HYDROCHLORIC ACID

6. Procedure

6.1 Transfer about 2 g (weighed to 0.1 mg) of the dried material (Section 3) to a 150-mL flask or beaker. Add 45 mL of hydrochloric acid (HCl, 1+2) and boil for 5 min. Add 50 mL of water and boil for 5 min. Filter through a previously dried and weighed Gooch crucible. Wash the insoluble residue on the filter with hot water until free of chlorides and then with methanol. Dry the crucible and contents at 105 $\pm$ 2°C for 2 h.

7. Calculation

7.1 Calculate the percent of matter soluble in HCl as follows:

$$\text{Matter soluble in HCl, \%} = [(S - R)/S] \times 100$$

where:

S = sample used, g, and

R = residue, g.

VOLUME OF SETTLING IN PETROLEUM SPIRITS

8. Procedure

8.1 Transfer 3 g of the sample to a 100-mL, glass-stoppered, graduated cylinder. Add mineral spirits, conforming to Specification D 235 until a total volume of 100 mL is obtained. Disperse the mixture by inverting the cylinder 50 times, and then allow to stand for 1 h. Read the volume of the settled pigment.

COLOR

9. Standard Pigments

9.1 *Standard Extender Pigment.*

9.2 *Standard Zinc Oxide.*

10. Procedure

10.1 Carefully weigh out the proportions of the standard extender pigment and standard zinc oxide mutually agreed upon, and rub up to a fairly stiff paste with a glass muller on a glass plate or stone slab with raw linseed oil conforming to Specification D 234. Note the volume of oil required. Prepare a similar paste with the sample, using the same weight of pigment, volume of oil, and number of strokes of the glass muller as used in the preparation of the paste of the standard pigments. Spread the pastes adjacently on a microscope slide, draw a scraper lightly across them so as to present them on an even plane, and judge the color immediately.

COARSE PARTICLES

Procedure

- 1 Determine the percent of coarse particles in accordance with Test Methods D 185.

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12. Precision and Bias

12.1 Precision and bias for these test methods have not been determined.

13. Keywords

13.1 diatomaceous silica pigment, analysis of; pigment, diatomaceous silica



Standard Specification for Raw and Burnt Umber Pigments¹

This standard is issued under the fixed designation D 763; the number immediately following the designation indicates the year of original adoption 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 May 1988.

1. Scope

1.1 This specification covers the pigments commercially known as raw umber and burnt umber.

2. Referenced Documents

2.1 ASTM Standards:

- D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 *Dry Pigments*—The pigments shall conform to the following requirements:

3.1.1 *Raw Umber*—The pigment shall be in a soft, dry form and shall be a hydrated oxide of iron (together with such manganese that is naturally associated with it) permeating a siliceous base and shall be free of admixtures of other substances except carbon pigments. The pigment shall conform to the requirements for composition prescribed in Table 1.

3.1.2 *Burnt Umber*—The pigment shall be produced by the calcination of raw umber and shall be free of admixtures of other substances except carbon pigments. The pigment shall conform to the requirements for composition prescribed in Table 1.

3.2 *Paste in Oil*—For both raw and burnt umber, the paste in oil shall be made by thoroughly grinding the pigment with linseed oil (with or without a small amount of volatile thinner) together with (where necessary) small amounts of wetting or dispersing agents to produce a paste or semipaste of satisfactory consistency. As received, it shall not

be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min, %	55
Nonvolatile vehicle, min, %	80
Moisture by distillation, max, %	2.0
Coarse particles and skins (total residue retained on a No. 325 (45- μ m) sieve), max, % of dry pigment	1.0

3.3 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4540 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.2 *Chemical Analysis of Dry Pigment*—Methods D 50.

TABLE 1 Requirements for Composition of Raw and Burnt Umber

	Raw Umber	Burnt Umber
Iron oxide (Fe_2O_3), min, %	37	42
Calcium compounds (as CaO), max, %	5.0	5.0
Moisture and other volatile matter, max, %	5.0	5.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	2.0	2.0
Organic colors, max, %	none	none

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 D01.31 on Pigment Specifications.

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

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

Moisture in Paste in Oil—Test Method D 280, except the sample shall be weighed instead of measured and the result shall be expressed as weight percentage instead of percent.

5.5 Pigment and Linseed Oil in Paste in Oil—Test Methods D 1208.

5.6 Mass Color and Tinting Strength—Test Method D 387.

Coarse Particles—Test Methods D 185.

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Standard Specification for Raw and Burnt Sienna Pigments¹

This standard is issued under the fixed designation D 765; the number immediately following the designation indicates the year of original adoption 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 specification covers the pigments commercially known as raw sienna and burnt sienna.

2. Referenced Documents

2.1 ASTM Standards:

- D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods of Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 *Dry Pigments*—The pigments shall conform to the following requirements:

3.1.1 *Raw Sienna*—The pigment shall be in a soft, dry form and shall be a hydrated oxide of iron permeating a siliceous base. The pigment shall conform to the requirements for composition prescribed in Table 1.

3.1.2 *Burnt Sienna*—The pigment shall be produced by the calcination of raw sienna and shall conform to the requirements for composition prescribed in Table 1.

3.2 *Paste in Oil*—For both raw and burnt sienna, the paste in oil shall be made by thoroughly grinding the pigment with linseed oil (with or without a small amount of volatile thinner) together with (where necessary) small amounts of wetting or dispersing agents to produce a paste or semipaste of satisfactory consistency. As received, it shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with

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 D01.31 on Pigment Specifications.

Current edition approved June 26, 1987. Published August 1987. Originally published as D 765 - 44. Last previous edition D 765 - 80.

² Annual Book of ASTM Standards, Vol 06.02.

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

TABLE 1 Requirements for Composition of Raw and Burnt Sienna

	Raw Sienna	Burnt Sienna
Iron oxide (Fe ₂ O ₃), min, %	38	40
Calcium compounds (as CaO) max, %	5.0	5.0
Moisture and other volatile matter, max, %	4.0	4.0
Coarse particles (total residue retained on a No. 325 (45-μm) sieve), max, %	2.0	2.0
Organic colors, max, %	none	none

linseed oil, turpentine, or volatile petroleum spirits, or any mixture of these substances. The paste shall conform to the following requirements:

Pigment, min, %	60
Nonvolatile vehicle, min, of vehicle	80
Moisture by distillation, max, %	2.0
Coarse particles and skins (total residue retained on a No. 325 (45-μm) sieve), max, % of dry pigment	1.0

3.3 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.1.1 *Total Iron Oxide, Calcium Compound, and Organic Coloring Matter*—Methods D 50.

5.1.2 *Coarse Particles*—Methods D 185.

5.1.3 *Pigment and Linseed Oil in Paste in Oil*—Test Methods D 1208.

5.1.4 *Moisture in Paste in Oil*—Test Methods D 280, except that the sample shall be weighed instead of measured

The results shall be expressed as percentage by weight
of volume.

5 Mass Color and Tinting Strength—Test Method

6. Keywords

6.1 burnt sienna; iron; manganese; pigment; raw sienna

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Standard Specification for Yellow Iron Oxide Hydrated¹

This standard is issued under the fixed designation D 768; the number immediately following the designation indicates the year of original adoption 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 specification has been approved for use by agencies of the Department of Defense to replace Fed. Spec. TP-P-458. 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 in June 1987.

1. Scope

1.1 This specification covers the pigment commercially known as yellow iron oxide, hydrated.

2. Referenced Documents

2.1 ASTM Standards:

- D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 The pigment shall be a manufactured yellow iron oxide obtained by chemical reaction. It shall be a soft, finely powdered pigment, free of admixtures of other substances and shall conform to the following requirements:

Total hydrated oxide of iron, min, %	93
Total oxide of iron, ⁴ min, %	83
Loss on ignition, ⁴ max, %	13
Moisture and other volatile matter, max, %	1.0
Water soluble matter, max, %	0.50
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	0.5
Hydrogen ion concentration (pH value)	4.5 to 8.0

⁴ Total hydrated oxide of iron shall be the sum of iron oxide and loss on ignition. Loss on ignition shall be calculated on the dry material.

3.2 *Paste in Oil*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil (with or without a small amount of volatile thinner) together with (where necessary) small amounts of wetting or dispersing agents to a semipaste or fluid type consistency. As received, it shall not be caked in the container and shall

break up readily in oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits, or any mixtures of these substances. The paste shall conform to the following requirements:

Pigment, min, %	55
Nonvolatile vehicle, min, % of vehicle	80
Moisture by distillation, max, %	2.0
Coarse particles and skins (total residue retained on a No. 325 (45- μ m) sieve), max, percent of the dry pigment	1.0
Consistency by the Stormer viscometer:	
At shearing rate of 100 revolutions/30 s, min, g	700 ⁴
At shearing rate of 100 revolutions/35 s, max, g	1200 ⁵

⁴ Equivalent to a rating of 125 Krebs' Units.

⁵ Equivalent to a rating of 145 Krebs' Units.

3.3 The mass color character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 *General*—Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Chemical Analysis*—Methods D 50.

5.1.2 *Coarse Particles in Pigments*—Test Method D 185.

5.1.3 *Moisture in Pigments*—Test Method D 280.

5.1.4 *Water Soluble Matter*—Test Method D 1208.

5.1.5 *Hydrogen Ion Concentration*—Test Method D 1208.

5.1.6 *Pigment and Linseed Oil in Paste in Oil*—Test Methods D 1208.

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 D01.31 on Pigment Specifications.

Current edition approved March 2, 1981. Published May 1981. Originally published as D 768 - 44T. Last previous edition D 768 - 47 (1974).

² Annual Book of ASTM Standards, Vol 06.02.

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

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Standard Specification for Black Synthetic Iron Oxide¹

This standard is issued under the fixed designation D 769; the number immediately following the designation indicates the year of original adoption 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 specification covers the pigment commercially known as black synthetic iron oxide.

2. Referenced Documents

2.1 ASTM Standards:

- D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1208 Test Methods for Common Properties of Certain Pigments²
- D 3872 Test Method for Ferrous Iron in Iron Oxides²

3. Composition and Properties

3.1 The pigment shall be a manufactured ferrous-ferric oxide obtained by chemical reaction. It shall be a soft dry finely powdered pigment, free of admixtures of other substances and shall conform to the following requirements:

Total ferrous and ferric oxide, min %	93
Ferrous oxide (FeO), min, %	20
Water-soluble matter, max, %	0.5
Moisture and other volatile matter, max, %	1.0
Coarse particles (total residue retained on a No. 325 (45-μm) sieve), max, %	0.5
Hydrogen ion concentration (pH value)	4.5 to 8.5

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 D01.31 on Pigment Specifications.

Current edition approved May 29, 1987. Published July 1987. Originally published as D 769 - 44. Last previous edition D 769 - 81.

² Annual Book of ASTM Standards, Vol 06.02.

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

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3.2 The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 Total Iron Oxides—Methods D 50.

5.1.2 Ferrous Iron in Iron Oxides—Test Method D 3872.

5.1.3 Water Soluble Matter, Maximum, Percent—Test Methods D 1208.

5.1.4 Moisture and Other Volatile Matter, Maximum, Percent—Test Methods D 280.

5.1.5 Coarse Particles (Total Residue Retained on a 325-mesh sieve (45 μm), Maximum, Percent—Test Methods D 185.

5.1.6 Hydrogen Ion Concentration (pH value)—Test Methods D 1208.

5.1.7 Mass Color and Tinting Strength—Test Method D 387.

6. Keywords

6.1 iron oxide; manganese; pigment



Standard Specification for Orange Shellac and Other Indian Lacs for Electrical Insulation¹

This standard is issued under the fixed designation D 784; the number immediately following the designation indicates the year of original adoption 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 covers the requirements and methods of test for three types and four grades of orange shellac and other lacs, as follows:

1.1.1 *Type I*—Orange Flake Shellac, Grades A, B, C, and D.

1.1.2 *Type II*—Button Lac, and

1.1.3 *Type III*—Garnet Lac.

1.2 Stick-lac and seed-lac are not covered by this specification.

1.3 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

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 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 29 Test Methods for Sampling and Testing Lac Resins²

D 411 Test Methods for Shellac Used for Electrical Insulation³

¹ This specification is under the jurisdiction of ASTM Committee D-9 on Electrical and Electronic Insulating Materials and is the direct responsibility of Subcommittee D09.01 on Electrical Insulating Varnishes, Powders, and Encapsulating Compounds.

Current edition approved May 27, 1983. Published July 1983. Originally published as D 784 - 61. Last previous edition D 784 - 61 (1975).

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vols 06.02 and 10.01.

3. Description of Materials

3.1 Type I is the commercial rosin-free grade of orange flake shellac. Type II customarily occurs in the form of circular disks about 76 mm (3 in.) in diameter and 3 mm ($\frac{1}{8}$ in.) in thickness and is known to the trade as pure button lac. Type III is dark garnet in color and is known to the trade as pure garnet lac. Garnet lac is also manufactured in admixture with rosin and as a dewaxed lac, but these are not covered by this specification.

4. Ordering Information

4.1 Orders for material covered by this specification shall include the following:

4.2 Lot number and supplier's designation,

4.3 Type and grade,

4.4 Quantity in each bag or container, and

4.5 Total quantity.

5. Properties

5.1 The material shall conform to the requirements prescribed in Table 1.

6. Test Methods

6.1 Determine the material sampled and the properties enumerated in this specification in accordance with Test Methods D 29 and Methods D 411.

7. Packaging and Package Marking

7.1 Packaging and marking shall be as agreed upon between the purchaser and the supplier.

8. Certification

8.1 When specified in the purchase order or contract, a producer's or supplier's certification shall be furnished to the purchaser that the material complies with the requirements of this specification. A report of the test results shall be furnished when specified in the purchase order or contract.

TABLE 1 Requirements for Orange Shellac and Other Lacs

	Type I				Type II	Type III
	Grade A	Grade B	Grade C	Grade D		
Matter insoluble in specified hot solvents, max, %	1.00	1.25	1.50	3.00	1.00	0.50
Iodine number, max	15.0	15.0	15.0	15.0	15.0	18.0
Moisture, max, %	2.0	2.0	2.0	2.0	2.0	2.0
Wax, max, %	5.5	5.5	5.5	5.5	5.5	3.5
Orpiment, max, % (native arsenic trisulfide)	0.03	0.03	0.03	0.2	0.03	0.03
Matter soluble in water, max, %	0.5	0.5	0.5	0.5	0.5	0.5
Ash, max, %	1.0	1.0	1.0	1.0	0.5	0.5
Rosin	none	none	none	none	none	...
Color						when specified, the color shall be no darker than that of a sample agreed upon by the purchaser and the seller ^A
Polymerization time at 150 ±1°C						when specified, shall be within the maximum-minimum range agreed upon by the purchaser and the seller
Flow test at 100 ±1°C						when specified, shall be within the maximum-minimum range agreed upon by the purchaser and the seller ^A

^A Attention is called to the fact that the purchaser and the seller must agree upon one of the two methods for determining (1) color for Type I, appearing in Methods D 29 and (2) flow test in Methods D 411.

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Standard Test Methods of Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate¹

This standard is issued under the fixed designation D 817; the number immediately following the designation indicates the year of original adoption 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 procedures for the testing of cellulose acetate propionates and acetate butyrates. These esters may vary widely in composition and properties, so certain of the procedures can be used only in the ranges of composition where they are suitable.

2. The test procedures appear in the following sections:

	Sections
Propionyl or Butyryl Contents	28 to 37
Acetyl Content, Apparent	18 to 27
Acidity, Free	12 to 17
Color	7 to 10
Color and Haze	77 to 81
Heat Stability	57 to 65
Hydroxyl Content	38 to 44
Hydroxyl Content, Primary	46 to 50
Intrinsic Viscosity	67 to 71
Moisture Content	5 to 6
Sulfur or Sulfate Content	51 to 56
Viscosity	74 to 75
Limiting Viscosity Number	67 to 71

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 Standard:

- D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing²
- D 1343 Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method³
- D 2929 Test Method for Sulfur Content of Cellulosic Materials by X-Ray Fluorescence³

3. Reagents

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 Chem-

ical 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.

4. Conditioning

4.1 *Conditioning*—Condition the test specimens at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and 50 ± 5 percent relative humidity for not less than 40 h prior to test in accordance with Procedure A of Methods D 618, for those tests where conditioning is required. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and ± 2 percent relative humidity.

4.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and 50 ± 5 % relative humidity, unless otherwise specified in the test methods. In cases of disagreements, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and ± 2 % relative humidity.

MOISTURE CONTENT

5. Procedure

5.1 Transfer about 5 g of the sample to a tared, low, wide-form weighing bottle and weigh to the nearest 0.001 g. Dry in an oven for 2 h at $105 \pm 3^\circ\text{C}$. Remove the bottle from the oven, cover, cool in a desiccator, and weigh.

6. Calculation

6.1 Calculate the percentage of moisture as follows:

$$\text{Moisture, \%} = (A/B) \times 100 \quad (1)$$

where:

A = weight loss on heating, g, and

B = sample used, g.

ASH

7. Significance and Use

7.1 Ash content gives an estimate of the inorganic content of cellulose ester samples. The presence of high levels of inorganic content (ash) can be detrimental to the melt stability and optical clarity of a cellulose ester in melt processing or act as a potential source of insolubles when the ester is used in solution.

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 D01.36 on Cellulose and Cellulose Derivatives.

Current edition approved Feb. 22, 1991. Published April 1991. Originally published as D 817 - 44 T. Last previous edition D 817 - 72 (1983).

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ "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."

8. Procedure

8.1 Dry the sample for 2 h at $105 \pm 3^\circ\text{C}$ and weigh 10 to 50 g, to the nearest 0.01 to 0.1 g, depending on its ash content and the accuracy desired. Burn directly over a flame in a 100-mL tared platinum crucible that has been heated to constant weight and weighed to the nearest 0.1 mg. Add the sample in portions if more than 10 g is taken. The sample should burn gently and the portions should be added as the flame subsides. Continue heating with a burner only as long as the residue burns with a flame. Transfer the crucible to a muffle furnace and heat at 550 to 600°C for 3 h, or longer if required, to burn all the carbon. Allow the crucible to cool and then transfer it, while still warm, to a desiccator. When the crucible has cooled to room temperature, weigh accurately to the nearest 0.1 mg.

9. Calculation

9.1 Calculate the percentage of ash as follows:

$$\text{Ash, \%} = (A/B) \times 100 \quad (2)$$

where:

A = ash, g, and

B = sample used, g.

10. Precision and Bias

10.1 No statement on bias can be made as no reference material is available as a standard.

FREE ACIDITY

11. Significance and Use

11.1 Free acidity is a measure of unesterified organic acid in the ester. The presence of high levels of free acid is potentially detrimental to melt processing of the ester and can impact the odor of the ester.

12. Reagents

12.1 Acetone, neutral.

12.2 Methyl Red Indicator Solution (0.4 g/L)—Dissolve 0.1 g of methyl red in 3.72 mL of 0.1000 N NaOH solution and dilute to 250 mL with water. Filter if necessary.

12.3 Phenolphthalein Indicator Solution (1 g/100 mL)—Dissolve 1 g phenolphthalein in 100 mL of ethyl alcohol (95 %).

12.4 Sodium Hydroxide, Standard Solution (0.01 N)—Prepare and standardize a 0.01 N solution of sodium hydroxide (NaOH).

Test Method A—For Samples Containing Not More than About 30 % Propionyl or Butyryl

13. Procedure

13.1 Shake 5 g of the sample, corrected for moisture content if necessary, in a 250-mL Erlenmeyer flask with 150 mL of freshly boiled, cold water. Stopper the flask and allow it to stand for 3 h. Filter off the cellulose ester and wash it with water. Titrate the combined filtrate and washings with 0.01 N NaOH solution, using phenolphthalein indicator solution.

13.2 Run a blank determination on the water, using the same volume as was used in extracting the sample.

14. Calculation

14.1 Calculate the percentage of acidity as free acetic acid as follows:

$$\text{Free acetic acid, \%} = \{[(A - B)C \times 0.06]/W\} \times 100 \quad (3)$$

where:

A = NaOH solution used to titrate the sample, mL,

B = NaOH solution used to titrate the blank, mL,

C = normality of the NaOH solution, and

W = sample used, g.

Test Method B—For Samples Containing More than About 7 % Propionyl or Butyryl and Particularly Suitable for Samples Containing More than 30 % Propionyl or Butyryl

15. Procedure

15.1 Dissolve 10.0 g of the sample, corrected for moisture content if necessary, in 200 mL of neutral acetone plus 20 mL of water. When completely dissolved, add 50 mL of water and shake well to precipitate the ester in a finely divided form. Add 3 drops of methyl red indicator solution and titrate to a lemon-yellow end point and 0.01 N NaOH solution.

15.2 Make a blank determination on the reagents.

16. Calculation

16.1 Calculate the free acid content as acetic acid as directed in Section 14.

17. Precision and Bias

17.1 No statement on bias can be made as no reference material is available as a standard.

APPARENT ACETYL CONTENT

18. Scope

18.1 The test methods described in the following sections 20 to 26 cover the determination of the saponification value of the sample calculated as percentage of apparent acetyl, equivalent weight 43. This value is required in the calculation of acetyl and propionyl or butyryl contents in 36.1.

18.2 The test method used should be specified or agreed upon. The choice depends on the propionyl or butyryl content and the physical condition of the sample. Ordinarily, Test Method A is recommended for samples having less than about 35 % propionyl or butyryl and Test Method B for samples having more than that amount.

19. Significance and Use

19.1 Apparent acetyl content is a measure of the saponification value of the ester. Apparent acetyl value is required in the calculation of acetyl, propionyl, and butyryl content in 36.1.

Test Method A—For Samples Containing Less than About 35 % Propionyl or Butyryl

20. Apparatus

20.1 Weighing Bottle, glass-stoppered, 15-mL capacity, 25-mm diameter by 50 mm high.

20.2 Tray, copper or aluminum, approximately 137 mm (5½ in.) square, containing 25 compartments 25 mm (1 in.) square. Each compartment shall have the correct dimensions

strongly recommended. Solution is more rapid, titrations are more rapid, and the end point can be approached directly and without a back titration.

NOTE 3—It is important to correct all 1.0 N H_2SO_4 buret readings for temperature and buret corrections.

23. Calculation

23.1 Calculate the percentage by weight of acetyl as follows:

$$\text{Acetyl, \%} = [(D - C)N_a - (B - A)N_b + P] \times 0.04305 / W \times 100$$

$$P = (GH \times 1000) / 204.2 \quad (\text{Note 4}) \quad (4)$$

where:

A = NaOH solution required for titration of the sample, mL

B = NaOH solution required for titration of the blank, mL

N_b = normality of the NaOH solution,

C = H_2SO_4 required for titration of the sample, mL

D = H_2SO_4 required for titration of the blank, mL

N_a = normality of the H_2SO_4 ,

P = milliequivalents of potassium acid phthalate,

G = potassium acid phthalate used, g,

H = purity factor for potassium acid phthalate, and

W = sample used, g.

NOTE 4—When equal volumes of alkali or acid are added to samples and blank, these amounts cancel out. Thus only the amounts of each added in the titration enter into the calculations. Use of potassium acid phthalate in the blank is recommended. When it is not used, the term P drops out of the equation.

Test Method B—For Cellulose Esters Containing More than 30 % Propionyl or Butyryl, by Varying the Reagents⁵

24. Reagents

24.1 *Acetone-Alcohol Mixture*—Mix equal volumes of acetone and methyl alcohol.

24.2 *Hydrochloric Acid, Standard (0.5 N)*—Prepare and standardize a 0.5 N solution of hydrochloric acid (HCl).

24.3 *Phenolphthalein Indicator Solution (1 g/100 mL)*—Dissolve 1 g of phenolphthalein in 100 mL of ethyl alcohol (95 %).

24.4 *Pyridine-Alcohol Mixture*—Mix equal volumes of pyridine and methyl alcohol.

24.5 *Sodium Hydroxide, Aqueous Solution (20 g/L)*—Dissolve 20 g of sodium hydroxide (NaOH) in water and dilute to 1 L with water.

24.6 *Sodium Hydroxide, Methanol Solution (20 g/L)*—Dissolve 20 g of NaOH in 20 mL of water and dilute to 1 L with methyl alcohol.

25. Procedure

25.1 Dry the sample for 2 h at $105 \pm 3^\circ C$ and cool in a desiccator. Weigh 0.5-g portions of the sample to the nearest 0.005 g and transfer to 250-mL glass-stoppered Erlenmeyer flasks. Dissolve each sample in 100 mL of appropriate solvent (see 25.2 and 25.3) and prepare at least two blanks, which shall be carried through all steps of the procedure.

25.2 *Samples Containing 30 to 45 % Propionyl or*

Butyryl—Dissolve in 100 mL of the acetone-alcohol mixture. Add water and aqueous NaOH solution from a buret or pipet in the following order and swirl the contents of the flask vigorously during all additions: 10 mL of NaOH solution, 10 mL of water, 10 mL of NaOH solution, 5 mL of water, 20 mL of NaOH solution, and 5 mL of water. Stopper and allow to stand at room temperature for 16 to 24 h.

25.3 *Samples Containing More than 45 % Propionyl or Butyryl*—Dissolve in 100 mL of the pyridine-alcohol mixture. Add 30 mL of the methanol solution of NaOH from a pipet or buret slowly, with swirling. Add 20 mL of water slowly in about 2-mL portions, with swirling, and swirl the flask until the solution becomes turbid. Stopper and allow to stand overnight at room temperature.

25.4 Back-titrate the excess NaOH with 0.5 N HCl just to the disappearance of color, using phenolphthalein indicator solution.

26. Calculation

26.1 Calculate the apparent acetyl content as follows:

$$\text{Apparent acetyl, \%} = [(A - B)N_a \times 0.04305 / W] \times 100 \quad (5)$$

where:

A = HCl required for titration of the blank, mL

B = HCl required for titration of the sample, mL

N_a = normality of the HCl, and

W = sample used, g.

27. Precision and Bias

27.1 No statement on bias can be made as no reference material is available as a standard.

ACETYL AND PROPIONYL OR BUTYRYL CONTENTS

28. Scope

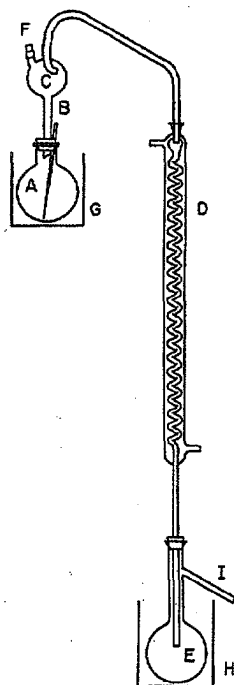
28.1 The test methods described in the following Sections 30 to 36 cover the determination of acetyl and propionyl or butyryl contents of cellulose mixed esters by calculation from the apparent acetyl content, determined in accordance with Sections 18 to 26, and the molar ratio of acetyl and propionyl or butyryl, determined in accordance with Sections 30 to 35. The molar ratio of acetyl and propionyl or butyryl is determined by saponifying, acidifying, vacuum distilling off the mixture of acids, and determining the distribution ratio of the acids between n -butyl acetate and water. The distribution ratios are also determined for acetic, propionic, and butyric acids, using samples of known high purity, and the molar ratio of the acids in the sample is calculated from these values.⁶

28.2 The saponification conditions are varied depending on the propionyl or butyryl content of the sample. Use Procedure A (Section 32) for samples containing less than about 35 % propionyl or butyryl, and use Procedure B (Section 33) for samples containing more than that amount.

28.3 Analyses for combined acetic, propionic, and butyric acids may be done by gas chromatographic methods. Diffi-

⁵ Malm, C. J., Genung, L. B., Williams, R. F., Jr., and Pile, M. A., "Analysis of Cellulose Derivatives: Total Acyl in Cellulose Organic Esters by Saponification in Solution," *Industrial and Engineering Chemistry, Analytical Edition*, IENAA, Vol. 16, 1944, pp. 501-504.

⁶ Malm, C. J., Nadeau, G. F., and Genung, L. B., "Analysis of Cellulose Derivatives: Analysis of Cellulose Mixed Esters by the Partition Method," *Industrial and Engineering Chemistry, Analytical Edition*, IENAA, Vol. 14, 1942, pp. 292-297. This reference may be consulted for application to other mixed esters and to three-component mixtures.



- A—Flask containing sample (500-mL, round-bottom).
- B—Capillary inlet tube.
- C—Kjeldahl distilling head.
- D—Condenser.
- E—Receiver (500-mL distilling flask).
- F—Opening for adding water.
- G—Water bath for heating sample.
- H—Cooling bath for receiver.
- I—Side arm, connected to vacuum line.

FIG. 1 Vacuum Distillation Apparatus for Mixed-Ester Analysis

culties encountered include ghosting in the columns, variation of factors with composition, and inconsistencies in the use of pure acids as standards. When such methods are used for this purpose, they shall be cross checked with the following partition method using suitable check batches to establish accuracy.

29. Significance and Use

29.1 Acetyl and propionyl or butyryl content is a measure of the amount of each of these acids esterified onto the cellulose backbone of the polymer. The amount of substitution of these esters has a very strong effect on the polymer's solubility and physical properties.

30. Apparatus

30.1 *Vacuum Distillation Apparatus*—The vacuum distillation apparatus shown in Fig. 1 will be required. The 500-mL round-bottom flask, A, shall be fitted with a stopper carrying a very small capillary inlet tube, B, and a Kjeldahl distilling head, C. The Kjeldahl distilling head shall be connected to a vertical condenser, D, having an outlet tube long enough to reach within 76.2 mm (3 in.) of the bottom of the 500-mL distilling flask, E, used as a receiver. The Kjeldahl distilling head shall be equipped with a funnel or

stoppered opening, F, for adding extra water during the distillation. A water bath, G, for heating the sample and a cooling bath, H, for cooling the receiver shall be provided.

31. Reagents

31.1 *Acetic, Propionic, and Butyric Acids*—Acetic, propionic, and butyric acids of tested purity.

31.2 *Bromocresol Green Indicator Solution* (0.4 g/L)—Grind 0.1 g of tetrabromo-*m*-cresolsulfonphthalein in a mortar with 14.3 mL of 0.01 *N* NaOH solution and dilute to 250 mL.

31.3 **n*-Butyl Acetate*—Prepare *n*-butyl acetate for use as an extraction solvent, free of acidity and water and containing not more than 2 % butyl alcohol. Check for acidity by shaking 60 mL of the *n*-butyl acetate with 30 mL of water in a 125-mL separatory funnel for about 1 min. Allow to settle, draw off the water layer, and titrate with 0.1 *N* NaOH solution, using phenolphthalein as the indicator. If this requires more than 0.02 mL of 0.1 *N* NaOH solution, the butyl acetate should be purified or a correction for acidity applied to each titration.

31.4 *Ethyl Alcohol*; Formula 2B, 3A, or 30 (denatured).

31.5 *Phosphoric Acid* (1 + 14)—Dilute 68 mL of phosphoric acid (H_3PO_4 , 85 %) to 1 L with water. Titrate the NaOH solution (20 g/L) with this acid to a yellow end point, using bromocresol green indicator solution, and calculate the volume of the acid (approximately 50 mL) required for 100 mL of the NaOH solution.

31.6 *Sodium Hydroxide Solution* (20 g/L)—Dissolve 20 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

31.7 *Sodium Hydroxide, Standard Solution* (0.1 *N*)—Prepare and standardize a 0.1 *N* solution of NaOH.

Isolation of the Mixed Acids

32. Procedure A—For Samples Containing Less than About 35 % Propionyl or Butyryl

32.1 Heat duplicate 3-g portions of the sample, not especially dried nor accurately weighed, with 100 mL of NaOH solution (20 g/L) in 500-mL, round-bottom, chemically resistant glass flasks in a water bath at 40°C for 48 to 72 h. At the end of this time add the required amount (approximately 50 mL) of H_3PO_4 (1+14) to each flask to form monosodium phosphate, which liberates the organic acids from their sodium salts.

32.2 Assemble the vacuum distillation apparatus as illustrated in Fig. 1. Heat the 500-mL round-bottom flask containing the sample in a water bath, and vacuum-distill the acid solutions to dryness, allowing a small stream of air bubbles to enter to avoid bumping. Keep the receiver cooled to 0°C. Add 25 mL of water to the residue in each flask and again distill to dryness. Repeat the distillation to dryness with a second 25-mL portion of water.

NOTE 5—In this operation it is not necessary to work with quantitative accuracy at all stages, but it is necessary to obtain water solutions of the acids in the same ratios as they occur in the esters. The volume of the distillate and rinsings is usually 200 to 250 mL, which in the majority of cases automatically adjusts the acidity of the distillate to 0.06 to 0.12 *N*, the range desired for subsequent extractions.

32.3 Continue as directed in Section 34.

33. Procedure B—For Samples Containing More than About 35 % Propionyl or Butyryl

33.1 Weigh duplicate 3-g samples, not especially dried nor accurately weighed, into 500-mL round-bottom flasks and add 100 mL of Formula 2B, 3A, or 30 denatured ethyl alcohol and 100 mL of NaOH solution (20 g/L) to each flask. Allow the samples to stand stoppered at room temperature for 48 to 72 h. At the end of this period, filter off the regenerated cellulose, collecting the filtrates in 500-mL round-bottom flasks.

33.2 Assemble the vacuum-distillation apparatus as illustrated in Fig. 1. Heat the flasks in the water bath and vacuum-distill off all the alcohol. After distilling to dryness, release the vacuum, rinse out the distillation heads, condensers, and receivers, and discard the distillates and rinsings.

33.3 Add the required amount, about 50 mL, of H_3PO_4 (1+14) to form monosodium phosphate, which liberates the organic acids from their sodium salts. Also add 100 mL of water to each flask and reassemble the distillation apparatus. Vacuum-distill the volatile acids as described in 32.2.

33.4 Continue as directed in Section 34.

Determination of the Molar Ratios of the Acids

34. Procedure

34.1 Titrate a 25-mL portion of the distillate (32.2) with 0.1 N NaOH solution, using phenolphthalein as the indicator. Designate the volume of NaOH solution required as M . Shake 30 mL of the distillate in a small separatory funnel with 15 mL of *n*-butyl acetate. Measure these volumes accurately using pipets and burets. Shake the mixture thoroughly for 1 min, allow the layers to separate for 2 min, and draw off the aqueous (lower) layer. Pipet out 25 mL of the solution and titrate with 0.1 N NaOH solution (Note 6). Designate the volume of NaOH solution required as M_1 . Calculate K , the percentage partition ratio of the acids in the distillate, as follows:

$$K = (M_1/M) \times 100 \quad (6)$$

NOTE 6—It should be kept in mind that all these determination are ratios and not quantitative; however, accuracy of duplication is very important. All measurements must be made as exactly as those made by standardizations of the solutions and equipment.

34.2 In the same manner determine the distribution ratios for acetic, propionic, and butyric acids. Dilute a sample of each acid of tested purity with water to give an approximately 0.1 N solution. Titrate 25-mL portions and extract 30-mL portions, following exactly the same procedure as used for the mixtures (34.1). Calculate the partition ratios for the pure acids, as decimal fractions, as follows (Note 7):

$$k = M_1/M \quad (7)$$

where:

k_a = distribution ratio for acetic acid under the conditions described,

k_p = distribution ratio for propionic acid under the conditions described, and

k_b = distribution ratio for butyric acid under the conditions described.

NOTE 7—The constants must be checked occasionally and must be determined by each operator for each supply of butyl acetate. Blanks

should be run on the butyl acetate, since it may develop acidity on standing, particularly if it contains a little water. All measurements should be made with good pipets or burets and extreme care and cleanliness observed during the whole operation. The accuracy of the procedure can be checked by testing an acid mixture of known composition.

35. Calculation

35.1 Calculate the molar ratios of acetic and propionic or butyric acids in the mixed acids as follows (Note 8):

$$P = (100k_a - K)/(k_a - k_p) \quad (8)$$

$$A = 100 - P \quad (9)$$

$$B = (100k_a - K)/(k_a - k_b) \quad (10)$$

$$A = 100 - B \quad (11)$$

where:

P = percentage of propionic acid, mol,

B = percentage of butyric acid, mol,

A = percentage of acetic acid, mol,

K = percentage distribution ratio of the acids in the distillate (34.1),

k_a = distribution ratio of acetic acid (34.2),

k_p = distribution ratio of propionic acid (34.2), and

k_b = distribution ratio of butyric acid (34.2).

NOTE 8—In order to evaluate two unknowns, two simultaneous algebraic equations involving the two unknown quantities are necessary. In the case of a binary acid mixture, the sum of the mol percentages of the acids present represents the total acidity, or 100 %. If A and B represent the mole percentages of acetic and butyric acids, respectively:

$$A + B = 100 \quad (12)$$

$$Ak_a + Bk_b = K \quad (13)$$

The distribution ratios k_a and k_b are known and refer to the pure individual acids, whereas the distribution ratio K refers to the binary mixture. By solving these equations for B , the equations given in this section may be derived.

Calculation of Acetyl, Propionyl, and Butyryl Contents

36. Calculation

36.1 Calculate the percentages by weight of acetyl, propionyl, and butyryl as follows:

$$\text{Acetyl, \%} = AC/100 \quad (14)$$

$$\text{Propionyl, \%} = (PC/100) \times (57/43) \quad (15)$$

$$\text{Butyryl, \%} = (BC/100) \times (71/43) \quad (16)$$

where:

A = percentage of acetic acid (Section 35), mol,

P = percentage of propionic acid (Section 35), mol,

B = percentage of butyric acid (Section 35), mol, and

C = percentages by weight of apparent acetyl (Sections 23 and 26).

36.2 Hydroxyl can be measured precisely, particularly at high degrees of esterification (Sections 38 to 44). It is therefore sometimes advantageous to base the calculation of weight percentages of acetyl, propionyl, and butyryl on hydroxyl content rather than on apparent acetyl as in 36.1. The equations for this calculation are as follows:

For cellulose acetate propionates:

$$\text{Acetyl, \%} = 9.15A(31.5 - h)/(786 - A) \quad (17)$$

$$\text{Propionyl, \%} = 2.93P(31.5 - h)/(786 - A) \quad (18)$$

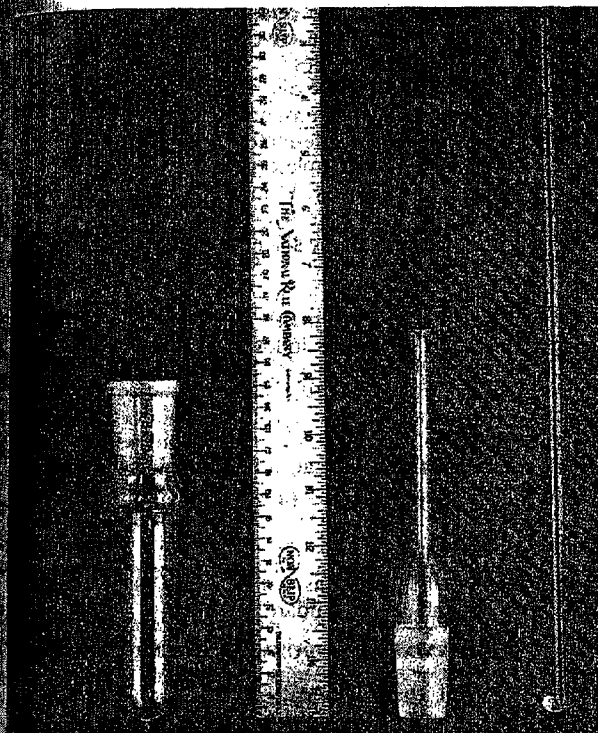


FIG. 2 Special Reflux Tube for Carbanilation

For cellulose acetate butyrates:

$$\text{Acetyl, \%} = 4.88A(31.5 - h)/(443 - A) \quad (19)$$

$$\text{Butyryl, \%} = 8.05B(31.5 - h)/(443 - A) \quad (20)$$

where, in addition to the definitions of terms in 36.1:
 h = weight percentage of hydroxyl (Section 44).

NOTE 9—This calculation involves the assumption that there are exactly three hydroxyls, free plus esterified, for each anhydroglucose unit of cellulose.

37. Precision and Bias

37.1 No statement on bias can be made as no reference material is available as a standard.

HYDROXYL CONTENT

38. Scope

38.1 This test method is applicable to pyridine-soluble cellulose esters and is especially useful when the hydroxyl content is low. (Samples containing plasticizer may be analyzed directly by this test method because the plasticizer is removed during washing of the carbanilate).

39. Summary of Test Method

39.1 Hydroxyl in cellulose esters is determined by reaction with phenyl isocyanate in pyridine solution under anhydrous conditions to form the carbanilate derivative. The derivative is then analyzed for its carbanilate content by ultraviolet absorption.

40. Significance and Use

40.1 Hydroxyl content is a measure of the free hydroxyl on the cellulose backbone of the polymer. Hydroxyl content has a strong effect on the polymer's solubility and physical properties. Hydroxyl content also impacts the propensity for this polymer to crosslink with various crosslinking agents.

41. Apparatus

41.1 *Spectrophotometer*,⁷ complete with hydrogen light source and a set of four 1.00-cm quartz cells or an equally suitable apparatus. The wavelength calibration, as checked against a mercury lamp, shall be within the manufacturer's tolerances. As a further check, measure the density of a potassium chromate (K_2CrO_4) solution prepared as follows: Dissolve 0.0400 g of K_2CrO_4 or 0.0303 g of potassium dichromate ($K_2Cr_2O_7$) in 0.05 *N* potassium hydroxide (KOH) solution and dilute to 1 L in a volumetric flask with 0.05 *N* KOH solution. Using the hydrogen lamp measure the absorbance at 280 nm of a silica cell filled with the K_2CrO_4 solution and also of the same cell filled with water. The absorbance of the solution minus that of the blank shall be 0.723 ± 0.023 .

41.2 *Bottles*, 112-g (4-oz), with screw caps, for washing the samples.

41.3 *Special Reflux Tubes* for the carbanilation, constructed as follows (see Fig. 2): Make a test tube approximately 20 by 150 mm from the outer part of a standard-taper 24/40 ground-glass joint by closing the open end in a blast lamp. Draw the tubing on the inner joint to a constriction just above the joint. Cut the glass at the point and seal on a short length of 8-mm tubing to provide a bearing for a glass stirrer. Make a stirrer of 4-mm glass rod with a semicircle at right angles to the shaft at the bottom and small enough to fit into the test tube. When properly constructed this unit acts as an air condenser, thus preventing the loss of solvent by evaporation.

41.4 *Pipet*, serological type, 5-mL capacity, graduated in 0.1-mL divisions.

41.5 *Büchner Funnel*, of a size accommodating 90-mm filter paper.

41.6 *Automatic Shaker*, with speed regulator mechanism.

41.7 *Electric Oven*, maintained at $105 \pm 3^\circ\text{C}$.

41.8 *Oil Bath*, equipped with a rack to hold several of the special reflux tubes. This bath shall be kept between 115 and 120°C .

42. Reagents

42.1 *Acetone*.

42.2 *Ethyl Alcohol*, Formula 2B, 3A, or 30 (denatured).

42.3 *Methylene Chloride-Methyl Alcohol Mixture*—Mix 9 parts by weight of methylene chloride with 1 part of methyl alcohol. This mixture should have an absorbance of less than 0.2 at 280 nm in a 1.00-cm silica cell measured against air. Pure methylene chloride has an absorbance of about 0.05, but the commercial product may have an absorbance as high as 1.00. The methylene chloride and methyl alcohol should

⁷ A Beckman Model DU Spectrophotometer has been found satisfactory for this purpose.

be selected to have low absorbance; otherwise, they should be redistilled.

42.4 Phenyl Isocyanate.⁸

42.5 Pyridine, redistilled, of low water content, preferably less than 0.05 %.

43. Procedure

43.1 In the following procedure the phenyl isocyanate reagent shall be used under anhydrous conditions. Therefore, the sample, containers, pipet, and all other equipment shall be thoroughly dried.

43.2 Place a 0.5-g sample in a special reflux tube and dry in an electric oven at $105 \pm 3^\circ\text{C}$ for 2 h. Remove the tube from the oven, add 5 mL of pyridine, assemble the reflux apparatus complete with glass stirring rod, and place in the 115 to 120°C oil bath. Stir occasionally until the sample is completely dissolved. Add 0.5 mL of phenyl isocyanate, stir thoroughly, and reflux in the oil bath for $\frac{1}{2}$ h to complete the reaction. Use 0.1 mL of phenyl isocyanate for each 1 % of estimated hydroxyl content, but never less than 0.5 mL.

43.3 At the end of the reaction time, remove the sample and dilute it with acetone to the proper viscosity for precipitation. The amount of acetone used to thin the solution is a critical factor in acquiring a good precipitate. Samples having low viscosity require little, if any, dilution. The average sample requires the addition of about an equal volume of acetone. Precipitate the carbanilate by pouring the solution into about 200 mL of ethyl alcohol, or if the ester contains more than 20 percent propionyl or butyryl, into the same volume of cold 80 % alcohol. Stir the alcohol vigorously during the precipitation. The precipitate should be fluffy and white. Sticky precipitates indicate too little dilution. Filter off the precipitate using paper on a Büchner funnel, with suction applied only as long as is necessary to remove the bulk of the solvent; prolonged suction may cause undesirable clumping together of the precipitate.

43.4 Wash the precipitate with alcohol, unless the sample was precipitated in cold 80 % alcohol. In this case, wash the precipitate in cold 90 % alcohol. Washing is best accomplished by transferring the precipitate to a 4-oz screw cap bottle containing about 75 mL of alcohol and shaking for $\frac{1}{2}$ h on an automatic shaker. Filter, pressing out as much liquid as possible with a glass stopper. Repeat the washing and filtering operations twice more.

NOTE 10—Samples of high hydroxyl content and large amounts of propionyl or butyryl may give gummy precipitates when poured into cold 80 % alcohol. Samples of this type give improved precipitates when precipitated in the reverse manner. Pour the diluted reaction solution into a 600-mL beaker, taking care to distribute the solution evenly on the bottom. Chill the beaker in a brine bath for 30 to 60 s. Pour about 200 mL of cold 80 % alcohol onto the chilled liquid. Wash the resulting precipitate and filter in the usual manner using cold 90 % alcohol.

43.5 Allow the precipitate to air-dry 1 to 2 h at room temperature with good ventilation or preferably overnight to ensure complete removal of the alcohol. (Samples wet with alcohol may sinter and stick to paper or glass when dried at

105°C .) Dry the sample at 105°C in the oven for 1 h and cool in a desiccator. Small manila envelopes are convenient for drying and cooling the samples.

43.6 Weigh 0.1231 g of the dry precipitate into a 100-mL volumetric flask fitted with a ground-glass stopper. Add 60 to 80 mL of methylene chloride-methyl alcohol mixture, and shake occasionally until complete solution occurs. Dilute to 100 mL and mix thoroughly. Using the spectrophotometer with a 1-cm silica cell, measure the absorbance of the solution at 280 nm against the solvent mixture as a reference.

44. Calculations

44.1 Calculate the percentage of carbanilate, c , for a sample weight of 0.1231 g as follows:⁹

$$\text{Carbanilate, \%} = A \times 17.1 \quad (21)$$

where:

A = absorbance.

44.2 Calculate the percentage of hydroxyl as follows:

$$\text{Hydroxyl, \%} = 14.3c/(100 - c) \quad (22)$$

45. Precision and Bias

45.1 No statement on bias can be made as no reference material is available as a standard.

PRIMARY HYDROXYL CONTENT

46. Summary of Test Method

46.1 The primary hydroxyl content of cellulose ester is determined by formation of the triphenylmethyl (trityl) ether and measurement of the trityl group by ultraviolet absorbance.⁹ Trityl chloride reacts preferentially with primary hydroxyls. Since there is also a slight reaction with secondary hydroxyls, standardized reaction conditions are important.¹⁰

47. Apparatus

47.1 See Section 41.

48. Reagents

48.1 Acetone.

48.2 Ethyl Alcohol, Formula 2B, 3A, or 30 (denatured).

48.3 Methylene Chloride-Methyl Alcohol Mixture—Mix 9 parts by weight of methylene chloride with 1 part of methyl alcohol. This mixture should have an absorbance of less than 0.2 at 259 nm in a 1-cm silica cell measured against air; otherwise, the solvents should be redistilled.

48.4 Pyridine, redistilled to a water content less than 0.05 %. The water content may be reduced further by storing over a suitable drying agent, such as a molecular sieve.¹¹

48.5 Trityl Chloride (Chlorotriphenylmethane or Triphenylmethyl Chloride).

⁹ Malm, C. J., Tanghe, L. J., Laird, B. C., and Smith, G. D., "Determination of Total and Primary Hydroxyl in Cellulose Esters by Ultraviolet Absorption Methods," *Analytical Chemistry*, ANCHA, Vol 26, 1954, p. 189.

¹⁰ Malm, C. J., Tanghe, L. J., and Laird, B. C., "Primary Hydroxyl Groups in Hydrolyzed Cellulose Acetate," *Journal of the American Chemical Society*, JACSA, Vol 72, 1950, p. 2674.

¹¹ Molecular Sieve Type 4A as manufactured by the Linde Air Products Co., has been found satisfactory for this purpose.

⁸ Phenyl isocyanate available as Eastman No. 553 has been found satisfactory for this purpose.

Procedure

The reagents must be used under anhydrous conditions. It is imperative that the sample and all equipment be thoroughly dry.

49.2 Place a 0.5-g sample in the test tube of the special apparatus and dry for 2 h at $105 \pm 3^\circ\text{C}$. Add 5 mL of pyridine, insert the top of the reflux apparatus and the thermometer, and heat with stirring in a 115 to 120°C oil bath. After the sample has dissolved, add 0.5 g of trityl chloride. If the total hydroxyl content exceeds 3 %, use an additional 0.5 g of trityl chloride for each additional 1 % hydroxyl. Stir the mixture thoroughly and reflux in the oil bath for 2 h at 115 to 120°C . Remove the tube and cool.

49.3 Dilute the sample with acetone to the proper viscosity for precipitation. The amount of acetone used to thin the solution is a critical factor in obtaining a good precipitate. Samples having low viscosity require little, if any, acetone. The average sample requires the addition of about an equal volume of acetone. Precipitate the trityl derivative by pouring the solution into about 200 mL of ethyl alcohol with vigorous stirring. The precipitate should be fluffy and white. Sticky precipitates indicate too little dilution. Separate the precipitate by filtering through paper on a Büchner funnel, with suction applied only as long as necessary to remove the bulk of the solvent; prolonged suction may evaporate the alcohol and cause the precipitate to partially redissolve in the remaining pyridine.

49.4 Wash the precipitate by transferring it to a 4-oz screw cap bottle containing 75 mL of ethyl alcohol, capping securely, and shaking for $\frac{1}{2}$ h on a shaker at medium speed. Again collect the precipitate on a Büchner funnel, pressing out as much liquid as possible with a glass stopper. Repeat this washing and filtering operation twice more, or until the absorbance of the filtrate at 259 nm is about the same as that of an alcohol blank. Allow the precipitate to air-dry on the filter paper for $\frac{1}{2}$ h at room temperature with good ventilation, or preferably overnight, to remove most of the alcohol. (Samples wet with alcohol may sinter or stick to paper or glass when dried at 105°C). Transfer the sample to a manila envelope, dry it for 1 h at 105°C , and cool in a desiccator.

49.5 Weigh a 0.1231-g sample of the dry trityl ether derivative into a 100-mL volumetric flask fitted with a ground-glass stopper, and dissolve in the methylene chloride-methyl alcohol mixture. Dilute to 100 mL and mix thoroughly. Measure the absorbance of this solution in a 1-cm silica cell using a spectrophotometer at 259 nm against the solvent as a reference.

50. Calculation

50.1 Calculate the trityl content, t , for this concentration of 0.1 g/100 g and with a correction of 0.015 for the absorbance of the cellulose acetate as follows:⁸

$$\text{Trityl, \%} = 25.25(A - 0.015) \quad (23)$$

where:

A = absorbance.

50.2 Calculate the weight percentage of primary hydroxyl as follows:

$$\text{Primary hydroxyl, \%} = 7.02 t / (100.4 - t) \quad (24)$$

50.3 Calculate the percentage primary hydroxyl of the total hydroxyl as follows:

$$\text{Primary hydroxyl of total hydroxyl, \%} = (B/C) \times 100 \quad (25)$$

where:

B = value of primary hydroxyl as determined in 50.2, and

C = value of total hydroxyl as determined in 44.2.

SULFUR OR SULFATE CONTENT

51. Summary of Test Method

51.1 The sulfur or sulfate content of cellulose acetate is measured by oxidizing the sample in a nitric acid-perchloric acid mixture and determined gravimetrically as barium sulfate. To determine combined sulfur the sample must first be reprecipitated into dilute acid to remove noncombined sulfur compounds.

51.2 The sulfur or sulfate content may also be determined by Test Method D 2929. The X-ray method shall be calibrated against the chemical method following in Sections 53 to 54, and the sample shall be treated in accordance with 53.1, if combined sulfur is to be determined.

52. Significance and Use

52.1 Sulfur and sulfate content indicates the amount of sulfur in the cellulose ester either as inorganic salts (usually sulfates) or as organic sulfate (usually as sulfate ester combined to the cellulose backbone). The presence of high levels of sulfur and sulfate can be detrimental to the melt stability of the ester.

53. Apparatus

53.1 *Funnel*, modified by cutting the stem off at the apex of the funnel and fire polishing.

53.2 *Crucibles*,¹² 30-mL, extra-fine porosity.

53.3 *Oven*, controlled at 120 to 125°C .

53.4 *Muffle Furnace*, controlled at $800 \pm 50^\circ\text{C}$.

54. Reagents

54.1 *Acetone*.

54.2 *Acetic Acid* (1+49)—Mix 1 volume of glacial acetic acid with 49 volumes of water.

54.3 *Barium Chloride Solution* (100 g/L)—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

54.4 *Hydrochloric Acid* (1+1)—Mix 1 volume of concentrated hydrochloric acid (sp gr 1.19) with 1 volume of water.

54.5 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

54.6 *Nitric Acid* (2+3)—Mix 2 volumes of concentrated nitric acid (sp gr 1.42) with 3 volumes of water.

54.7 *Nitric Acid-Perchloric Acid Mixture*—Mix 5 volumes of concentrated HNO_3 with 1 volume of concentrated perchloric acid (HClO_4 , 70 %).

54.8 *Phenolphthalein Indicator Solution* (1 g/100 ml)—Dissolve 1 g of phenolphthalein in 100 mL of ethyl alcohol (95 %).

54.9 *Silver Nitrate Solution* (50 g/L)—Dissolve 50 g of silver nitrate (AgNO_3) in water and dilute to 1 L.

54.10 *Sodium Carbonate* (Na_2CO_3).

54.11 *Sodium Hydroxide Solution* (400 g/L)—Dissolve

¹² Selas No. 3001 crucible has been found satisfactory for this purpose.

400 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

55. Procedure

Treatment Prior to Analysis

55.1 Remove uncombined sulfur as follows (Note 11): Dissolve 25 g of sample in approximately 300 mL of acetone, depending on the viscosity. If the sample is of too high acetyl content to be directly soluble in acetone, cool in a dry ice cabinet overnight; then allow to come to room temperature while tumbling or stirring. Filter the solution, if necessary, through felt or a coarse sintered-glass crucible. Precipitate with rapid stirring into a beaker or pail containing 2 to 3 L of acetic acid (1+49). Filter through a cloth bag or a Büchner funnel and give two 15-min washes with water using mechanical agitation. A little Na_2CO_3 may be added to the last wash to stabilize samples of high sulfur content. Filter and dry overnight at 60°C.

NOTE 11—To analyze for total sulfur content omit this treatment.

Decomposition

55.2 Weigh 10 ± 0.1 g of cellulose acetate and transfer to a clean wide-mouth, 500-mL Erlenmeyer flask. Add 50 mL of the HNO_3 - HClO_4 mixture to the flask, and swirl the flask gently to wet the sample thoroughly. Place the modified funnel in the mouth of the flask and heat the flask carefully on a hot plate in a fume hood.

NOTE 12: Precaution—Use the utmost care in handling the HNO_3 - HClO_4 mixture. If a spill occurs, wash down with plenty of water. Wear safety glasses or a face shield.

55.3 After the mixture becomes hot and less viscous, increase the heat of the hot plate. Continue the digestion until all the sample has been oxidized and the thick reddish-brown fumes of nitric oxide have been expelled. At this point, white fumes will appear and a rather vigorous reaction will occur that is caused by the last traces of organic material being oxidized and the nitric acid fuming off.

55.4 When this reaction starts, remove the flask from the hot plate, swirl gently for a few seconds, and set it on the shelf in front of the hood until the reaction is complete. Place the flask back on the hot plate and continue the digestion until the HClO_4 refluxes about half way up the side of the Erlenmeyer flask and about 5 mL is left in the flask. The HNO_3 - HClO_4 mixture should be clear and colorless. If it is not, set the flask off the hot plate to cool and then add 3 to 5 mL of HNO_3 (sp gr 1.42). Replace the flask on the hot plate and continue heating until the HClO_4 refluxes half way up the flask. Remove the flask from the hot plate and allow the flask and its contents to cool.

Determination of Barium Sulfate

55.5 Wash the modified funnel top thoroughly with water, collecting the rinsings in the flask. Add 50 mL of water. Swirl the flask to mix the solution thoroughly. Add 2 drops of phenolphthalein indicator solution and neutralize the acid with the NaOH solution to a faint pink. Acidify immediately with HCl (1+1), dropwise, until the solution is just acid to phenolphthalein; then add 2 mL of HCl (1+1).

55.6 Filter through a 12.5-cm fine-porosity paper into a clean 400-mL beaker. Wash the flask thoroughly with water, filtering the washings through the paper. Finally wash the

paper thoroughly with ten portions of hot water. Dilute the filtrate to approximately 200 mL. Place the beaker on the hot plate and heat almost to boiling. Slowly add 10 mL of BaCl_2 solution from a pipet, stirring the solution during the addition. Do not add the BaCl_2 solution rapidly, as from a graduate, since the rapid addition will produce an impure precipitate. Remove the stirring rod from the beaker and wash it with a stream of water from the wash bottle, collecting the washings in the beaker. Cover the beaker with a watch glass and keep the mixture near the boiling temperature for 6 h or overnight. Do not allow the liquid to evaporate to dryness.

55.7 Using suction, decant the supernatant liquid through an extra-fine porosity porcelain filter crucible that has been previously rinsed with acetone, ignited, and weighed to the nearest 0.1 mg. Transfer the precipitate with the aid of a stream of hot water. Always use a stirring rod in this transfer. Scrub the sides and bottom of the beaker with a rubber policeman to remove any adhering precipitate. The crucibles may be used to collect several precipitates one on top of the other. Close control of temperature and time of heating and cooling are necessary. Cleaning with hot water is generally sufficient; drastic attack with cleaning solution should be avoided.

55.8 Wash the precipitate on the filter until free of chlorides by the following test: To 5 mL of wash water, collected in a separate test tube or on a watch glass, add 1 mL of HNO_3 (2+3) and 1 mL of AgNO_3 solution. The appearance of a milky white precipitate indicates the presence of chlorides, and the washing should therefore continue until the test is negative. Do not attempt to get a completely negative test for chloride. Discontinue washing when no more than a faint opalescence is produced in the test.

55.9 Finally pour a few millilitres of pure acetone through the filter and suck it dry. Place the crucible in a larger crucible or in a metal tray with perforated sides and bottom for protection and place it in an oven at 120 to 125°C for 1 h. Do not handle the crucibles with the fingers between ignition and the completion of weighing; use forceps.

55.10 Remove the crucible from the oven and ignite it for 10 min in a muffle furnace at $800 \pm 50^\circ\text{C}$. Cool in a desiccator for 75 ± 15 min and weigh to the nearest 0.0001 g. It is permissible to return the crucible to the oven for at least 15 min before transferring to the desiccator.

55.11 From time to time, and especially when using new reagents, run a blank in duplicate in the reagents. If the weight of the precipitate exceeds 0.0005 g, investigate and eliminate the cause. This is equivalent to an error of 0.002 % on a 10-g sample.

56. Calculation

56.1 Calculate the percentage of sulfur and sulfate as follows:

$$\text{Sulfur, \%} = \{[(C - B) - (E - D)] \times 0.1374/A\} \times 100 \quad (26)$$

$$\text{Sulfate, \%} = \{[(C - B) - (E - D)] \times 0.4115/A\} \times 100 \quad (27)$$

where:

A = weight of sample, g,

B = weight of crucible for sample, g,

C = weight of crucible and BaSO_4 for sample, g,

C - B = weight of BaSO_4 for sample, g,

W_1 = weight of crucible for blank, g,
 W_2 = weight of crucible and BaSO_4 for blank, g, and
 W_3 = weight of BaSO_4 for blank, g.

HEAT STABILITY

Summary of Test Method

The heat stability of a cellulose ester is one indication of its quality. It is measured by heating the sample for a specified time and temperature, observing it for amount and uniformity of color developed, and possibly also measuring loss of viscosity as a result of heating. Suggested times of heating are 8 h at 160°C, 8 h at 180°C, or 2 h at 190°C. The time and temperature of heating, method of grading, and to the extent possible, are matters for agreement between the purchaser and the seller.

Significance and Use

63.1 The heat stability of a cellulose ester is one indication of its quality.

Apparatus

59.1 **Heater Block**—A metal block of suitable size heated electrically and maintained at the specified temperature within $\pm 1^\circ\text{C}$. This is best accomplished by providing continuous heat to hold the temperature a few degrees below the specified temperature, and providing intermittent additional heat thermostatically controlled. Holes shall be drilled in the top of the block to hold test tubes, a thermometer, and a thermometer.

59.2 **Test Tubes**, either 18 by 150-mm or 20 by 150-mm, fitted with corks. The corks shall be fitted with glass tubes the length of the cork and 4 mm in inside diameter or shall have a small V-shaped notch of equivalent cross-section cut in a vertical position.

60. Solvent

60.1 **Methylene Chloride-Methyl Alcohol Mixture**—Mix 9 parts by weight of methylene chloride with 1 part of methyl alcohol.

61. Heat Treatment

61.1 Place the sample, ground to pass a No. 20 (841- μm) sieve, in a clean, dry test tube and pack it firmly and uniformly. Stopper with a cork having a notch or tube as described in 59.2. Heat the tube and contents for 8 h at 180°C or as otherwise specified.

62. Dry Color Evaluation

62.1 Examine the heated sample for uniformity of color and for the presence of charred or decomposed spots. Compare the color of the material at the bottom of the tube with standards prepared as follows: Heat portions of a check batch of similar particle size, representative quality and stability, and accepted by mutual agreement between the purchaser and the seller. Pack portions of this check batch firmly in each of twelve clean, dry test tubes and stopper with corks as described in 59.2. Heat the tubes at 180°C, or as otherwise specified, remove one tube each 2 h, and mark the time of heating in hours on each tube. This set of numbered tubes serves as the color standards. They should be checked

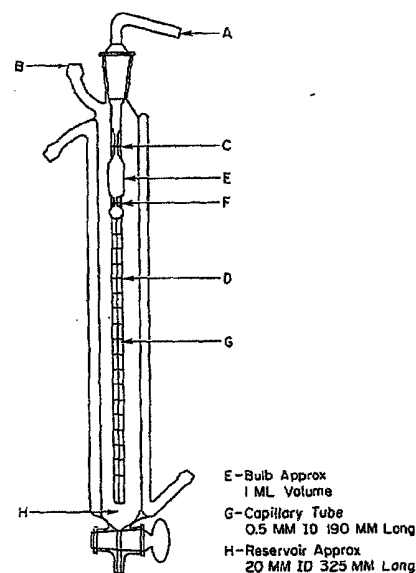


FIG. 3 Wagner Capillary Tube Viscometer¹³

and renewed if necessary every 6 months.

63. Solution Color Using Platinum-Cobalt Standards

63.1 Heat a 1-g sample for the specified time and temperature and, after cooling, examine for charred or decomposed spots. Dissolve the heated sample in 15 mL of the methylene chloride-methyl alcohol mixture. Compare the color of the solution (viewing transversely) with test tubes of platinum-cobalt color standards, prepared as described in Section 80. (It may be necessary to prepare standards having as much as 2000 ppm of platinum for this purpose or to dilute the sample solution before grading.)

64. Solution Color by Spectrophotometer

64.1 The color of the solution prepared as described in Section 63 may also be measured spectrophotometrically. Measure the absorbance at 400 nm against the solvent, using a suitable spectrophotometer with a 1-cm silica cell.

65. Viscosity Change

65.1 Measure the limiting viscosity number of the heated sample and of an unheated sample as described in Sections 67 to 71 of this test method. The percentage loss of viscosity as the result of heating is a measure of heat stability.

66. Precision and Bias

66.1 No statement on bias can be made as no reference material is available as a standard.

¹³ Wagner, R. H., and Russell, John, "Capillary Tube Viscometer for Routine Measurement of Dilute High Polymer Solutions," *Analytical Chemistry*, Vol 20, 1948, pp. 151-7.

TABLE 1 Solvents for Limiting Viscosity Number Determination

Solvent ^A	Ingredients, % by weight	Value of k for Calculation (71.2)
A	90 % acetone ^B 10 % ethyl alcohol ^C	10
B	acetone ^B	10
C or D	90 % methylene chloride ^D 10 % ethyl alcohol ^C	3
E	98 % acetone ^B 4 % water	10
F	90 % methylene chloride ^D 10 % methanol ^E	3

^A Solvent designations conform to those used in Table 2 for viscosity determinations.

^B Acetone (99.4 ± 0.1 %) containing 0.3 to 0.5 % water and under 0.3 % ethyl alcohol.

^C Ethyl alcohol (95 % by volume). Formula 2B or 3A denatured ethyl alcohol may be used.

^D Methylene chloride having a boiling range of 39.2 to 40.0°C and less than 0.001 % acidity calculated as HCl.

^E Methyl alcohol (sp gr 20/20°C = 0.785 to 0.795).

LIMITING VISCOSITY NUMBER

67. Summary of Test Method

67.1 Limiting viscosity number, expressed in millilitres of solution per gram of solute, is determined by measuring the flow times of a solution of known concentration and also of the solvent used and making a calculation by means of the modified Baker-Philippoff equation.

67.2 Intrinsic viscosity, expressed in decilitres per gram of solute, is determined in the same way by expressing c in grams per 100 mL in 71.2. Limiting viscosity number is thus $100 \times$ intrinsic viscosity.

68. Significance and Use

68.1 Limiting viscosity number can be used to estimate the molecular weight of a cellulose ester by using the Mark-Houwink equation and constants measured for the solvent, temperature, and ester of concern.

69. Apparatus

69.1 *Capillary Viscometer*, such as the Wagner apparatus (see Fig. 3) or an Ostwald-Fenske-Cannon pipet, that will give a flow time for the solvent of not less than 70 s.

69.2 *Water Bath*—A constant-temperature water bath controlled at $25.0 \pm 0.1^\circ\text{C}$ and with a pump for circulating the water through the viscometer jacket or tank.

69.3 *Stop Clock or Watch*, calibrated in $\frac{1}{10}$ s.

70. Procedure

70.1 *Sample Preparation*—Dry about 0.26 g of sample in a weighing bottle at $105 \pm 3^\circ\text{C}$ for 2 h, stopper, and cool in a desiccator. Weigh the bottle containing the sample to the nearest 0.001 g, transfer the sample to a 250-mL flask, and reweigh the bottle. Pipet into the flask 100 mL of solvent at $25 \pm 0.1^\circ\text{C}$. The solvent used should be mutually agreed upon by the purchaser and the seller. Suitable solvents are listed in Table 1. After the sample is completely dissolved, place it in the constant-temperature bath at 25°C along with a portion of the solvent used, and allow sufficient time for both to come to temperature before making the viscosity measurements. During this conditioning period, water at

TABLE 2 Solutions for Viscosity Determination

	Formula					
	A	B	C	D	E	F
	Ingredients, Weight %					
Cellulose ester	20 ^A	20 ^A	20 ^B	15 ^C	20 ^A	10 ^C
Acetone ^D	72	80	...	...	...	...
Acetone, 96 percent	...	...	...	...	80	...
Water, 4 percent	...	...	...	...	...	...
Ethyl alcohol ^E	8	...	8	8.5	...	...
Methyl alcohol ^F	...	...	...	...	...	9
Methylene chloride ^D	...	...	72	76.5	...	81
Typical Solution Densities, g/mL at 25°C						
	0.85	0.86	1.25	1.23	0.86	1.24

^A Suitable for most mixed esters having less than about 40 % acetyl and more than about 8 % propionyl or butyryl.

^B Suitable for most of the commercial cellulose acetate propionates and acetate butyrates.

^C Suitable for most of the commercial cellulose acetate propionates and acetate butyrates. Particularly good for esters containing more than 40 % acetyl.

^D Acetone (99.4 ± 0.1 %) containing 0.3 to 0.5 % water and under 0.3 % ethyl alcohol.

^E Ethyl alcohol (95 % by Vol). Formula 2B, 3A, or 3D denatured ethyl alcohol may be used.

^F Methyl alcohol (sp gr 20/20°C = 0.785 to 0.795).

^D Methylene chloride having a boiling range of 39.2 to 40.0°C and less than 0.001 % acidity calculated as HCl.

25°C should be circulating through the water jacket of the viscometer to allow ample time for the pipet to reach temperature equilibrium.

70.2 *Viscosity Measurements*—Rinse the reservoir and the outside of the capillary tube thoroughly with solvent. Rinse the inside of the capillary tube twice by alternately applying pressure at points B and A (see Fig. 3). Discard the wash portion of the solvent. Pour more solvent into the reservoir and allow several minutes for complete drainage and thermal equilibrium to be obtained. Adjust the outer meniscus to a reference point, D, that will give a flow time between 70 and 100 s. Apply air pressure at B to force the solvent up through the capillary past the upper timing mark, C, on the measuring bulb, E. Record the time in seconds required for the meniscus to fall between the timing marks, C and F. Take a minimum of two readings. Repeat these operations, substituting the solution for the solvent.

71. Calculation

71.1 Calculate the viscosity ratio, η/η_0 as follows:

$$\text{Viscosity ratio} = t_1/t_2 \quad (28)$$

where:

t_1 = efflux time of solution, and

t_2 = efflux time of solvent.

NOTE 13—Strictly, the viscosity ratio is defined as η/η_0 where η and η_0 are the viscosities of the solution and solvent, respectively, and are related to the corresponding efflux times by:

$$\eta = Ct - E\rho/t^2 \quad (29)$$

$$\eta_0 = Ct_0 - E\rho_0/t_0^2 \quad (30)$$

where:

C and E are constants for the particular viscometer used.

The equation in 71.1 follows if the second term in these relations, a kinetic energy correction, is negligible and the respective solvent and solution densities, ρ_0 and ρ , are substantially equal.

71.2 Calculate the limiting viscosity number, $[\eta]$, as follows:

COLOR AND HAZE

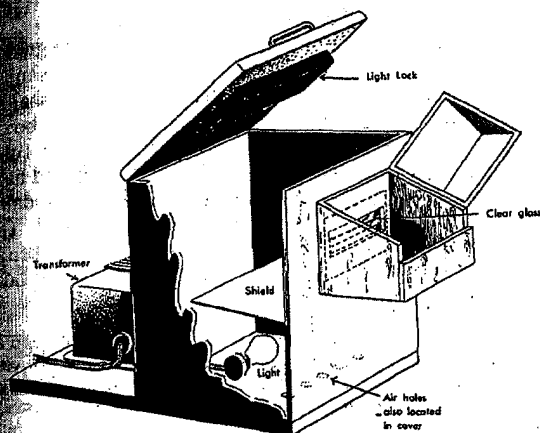


FIG. 4 Color and Haze Apparatus

$$[\eta] = (k/c)[\text{antilog}((\log \eta/\eta_0)/k) - 1] \quad (31)$$

where:

- = values from Table 1 (Note 14), and
- = concentration in g/mL.

NOTE 14—Different values may be used by agreement between the purchaser and the seller.

71.3 Calculate intrinsic viscosity, η , in accordance with 71.2, but express c in grams per 100 mL.

72. Precision and Bias

72.1 No statement on bias can be made as no reference material is available as a standard.

VISCOSITY

73. Significance and Use

73.1 A measurement of viscosity is of great practical utility in determining the proper processing equipment and process concentrations for cellulose esters.

74. Procedure

74.1 *Solution*—Dry the sample for 1 to 2 h at $105 \pm 3^\circ\text{C}$ and cool in a desiccator. Prepare a solution of the dried sample in a solvent and at a concentration mutually agreed upon by the purchaser and the seller. Suitable solutions are listed in Table 2.

74.2 *Viscosity Determination*—Prepare the solution and measure the viscosity in accordance with Test Method D 1343, (see Note 16 in Section 81).

75. Report

75.1 Report the results in poises, unless otherwise specified. The viscosity value shall be prefixed with the letter A, B, C, etc., corresponding to the formula of the solution employed.

76. Precision and Bias

76.1 No statement on bias can be made as no reference material is available as a standard.

77. Summary of Test Method

77.1 Color and haze determinations on cellulose ester solutions are made by comparison with standards. Simultaneous measurement of these properties is desirable because haze reduces the amount of color observed.

78. Significance and Use

78.1 Solution color and haze of a cellulose ester is a measurement of the optical properties of cellulose esters when dissolved in a specific solvent.

79. Apparatus

79.1 *Light Box*—A suitable light box (Fig. 4) is described as follows: The light source consists of a mercury vapor bulb¹⁴ which requires an autotransformer¹⁵ for the current source. The bulb is mounted horizontally across the lower front part of a plywood box 356 mm (14 in.) wide, 430 mm (17 in.) high, and 330 mm (13 in.) deep. This box is lined with a heat resistant board and is painted black inside, except that the inside back surface toward the viewer is white. A bottle holder large enough to hold four bottles is built onto the front of the light box, and a 64 by 150-mm (2½ by 6-in.) horizontal viewing hole is cut through the front of the box. This opening is covered with clear glass, and a 6-mm (¼-in.) strip of black tape is fastened to the glass horizontally to aid in judging haze in the solution. Holes are cut in the bottom and top of the box for cooling by air convection. For continuous use, forced circulation of air would be desirable. A black metal baffle over the bulb prevents direct light on the viewing glass.¹⁶

79.2 *Sample Bottles*—The bottles used for the sample solutions are French square bottles, 470-mL (16-oz), with screw caps. These same bottles may be used for the color and haze standards.¹⁷

NOTE 15—These bottles may also be used for determination of viscosity, as described in 74.2.

79.3 *Cap Liners*—Cap liners shall be of a composition not affected by the solvents used. Liners of fiber board covered with cellophane or aluminum foil are usually satisfactory, but vinyl resin or waxed liners may cause interference with viscosity, color, or haze measurements.

80. Reference Standards

80.1 *Color Standards*—A color standard containing 500 ppm of platinum may be purchased or the solution may also be prepared as follows: Dissolve 1.245 g of potassium platinum chloride (K_2PtCl_6), containing 0.500 g of platinum, and 1.000 g of crystallized cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$),

¹⁴ A General Electric mercury vapor bulb Type H-250, A5-1, or A37/51 (differing only in the shape of the glass globe) has been found satisfactory for this purpose.

¹⁵ A General Electric Autotransformer Model 9T64Y21 has been found satisfactory for this purpose.

¹⁶ A detailed drawing of the color and haze apparatus may be obtained from ASTM Headquarters. Request Adjunct No. 12-408170-00.

¹⁷ Owens-Illinois French square bottles No. A6732 with No. 48-400 caps have been found satisfactory for the sample solutions, viscosity, and for the color and haze standards, No. A6720 bottles with No. 28-400 caps are more convenient.

containing 0.248 g of cobalt, in water, add 100 mL of HCl (sp gr 1.19), and dilute to 1 L with water. Prepare standards containing 50, 60, 70, 80, 90, 100, 125, 150, 175, 200, 250, 300, 350, 400 and 500 ppm of platinum by diluting suitable aliquots of the standard solution to 500 mL with water. Place these standards in the special bottles (see 79.2), taking care to select bottles with good clarity and free of flaws. Label and cap tightly.

80.2 Haze Standards—Prepare haze standards by diluting a stock solution having a turbidity of 1000 ppm. Prepare bottles containing 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 125, 150, 175, 200, and 400 ppm of turbidity, label, and cap tightly.

NOTE 16—The previously recommended stock solution for preparing these standards was made from fuller's earth, water, and hydrochloric acid. This solution is no longer available. A comparable stock solution can be made using diatomaceous earth.¹⁸ To obtain haze levels equivalent to the fuller's earth standard, 1.1 parts of diatomaceous should be used in place of 1.0 parts of fuller's earth in preparing the aqueous suspension. No hydrochloric acid is needed.

81. Procedure

81.1 Prepare the solution to be graded by dissolving the cellulose ester in the specified amount and kind of solvent, in one of the square bottles. See Table 2 for suitable solutions. At least 350 mL are required. Tumble until a uniform solution is obtained. Allow the solution to stand until it is

¹⁸ Celite,[®] available from John-Mansville Filtration and Mineral Division, Ken-Caryl Ranch, Denver, Co. 80217, has been found suitable for this purpose.

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free of bubbles before grading it for color and haze.

81.2 Place the bottle containing the solution to be graded at the front of the shelf on the apparatus and place a similar bottle containing water behind it. Place the freshly shaken haze standard at the front of the shelf beside the bottle containing the solution to be tested and place the color standard behind it. Determine the amount of color and haze in the solution by changing the color and haze standards until as good a match as possible has been obtained. The haze standards settle out quickly so they must be reshaken at short intervals. Report results in parts per million for both color and haze.

NOTE 17—When viscosity, color, and haze determinations, and an observation of general appearance are to be made on a cellulose ester sample, a considerable saving in time can be made by using one solution in a square bottle for all three determinations. Dry the cellulose ester as required for the viscosity determination, prepare the solution carefully, and allow the bottle to stand long enough to form a thick solution before tumbling, to avoid solvent loss around the cap. Use a large enough sample to provide at least 350 mL of solution in the bottle. Measure the viscosity as described in Test Method D 1343.

82. Precision and Bias

82.1 No statement on bias can be made as no reference material is available as a standard.

83. Keywords

83.1 apparent acetyl; ash; cellulose acetate butyrate; cellulose acetate propionate; cellulose esters; color; free acidity; haze; hydroxyl; limiting; partition; sulfate content; viscosity

Standard Specification for Pumice Pigment¹

This standard is issued under the fixed designation D 867; the number immediately following the designation indicates the year of original adoption 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 April 1986.

Scope

- 1.1 This specification covers vesicular pumice suitable for use in the manufacture of organic coatings.
- 1.2 Two types of material are described:
 - 1.2.1 Type A—For traffic paint.
 - 1.2.2 Type B—For nonskid deck paint.

Referenced Documents

2.1 ASTM Standards:

- D 718 Test Methods for Analysis of Aluminum Silicate Pigment²
- D 3360 Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments²

3. Composition and Properties

3.1 The pigment shall be definitely vesicular and shall conform to the following requirements:

Loss on ignition, max, %	5.0
Moisture and other volatile matter, max, %	1.0
Sieve analysis	see Table 1

3.2 The color shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

3.3 When viewed under the microscope, or as photographic mounts, at a magnification of at least 100 diameters, the sample shall exhibit no more nonvesicular particles than

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 D01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vol 06.02.

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a reference sample mutually agreed upon by the purchaser and the seller.

3.4 Pumice for use in paints shall be equal in mechanical analysis to a reference sample mutually agreed upon by the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or after blending in equal quantities, the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with Methods D 718, except that for sieve analysis the appropriate sieves, given in Table 1 of this specification, shall be used.

5.2 Particle Size—Test Method D 3360.

TABLE 1 Sieve Analysis Requirements

Sieve		Percent	
Passing	Retained on	Min	Max
...	No. 50 (300 μ m)	...	0
No. 50 (300 μ m)	No. 100 (150 μ m)	70	...
No. 100 (150 μ m)	...	...	30
Pumice Pigment for Nonskid Deck Paint—Type B			
...	No. 40 (425 μ m)	...	1
No. 40 (425 μ m)	No. 60 (250 μ m)	20	60
No. 60 (250 μ m)	No. 80 (180 μ m)	25	60
No. 100 (150 μ m)	...	...	10



Standard Test Methods of Testing Cellulose Acetate¹

This standard is issued under the fixed designation D 871; the number immediately following the designation indicates the 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 testing cellulose acetate.

1.2 The test procedures appear in the following sections:

	Sections
Ash	8 and 11
Color and Haze	68 to 72
Combined Acetyl or Acetic Acid Content	
Test Method A. Solution Method	17, 19 to 23
Test Method B. Heterogeneous Saponification Method	17, 24 to 26
Free Acidity	12 to 16
Heat Stability	47 to 56
Hydroxyl Content	27 to 33
Intrinsic Viscosity	57 to 62
Moisture Content	4 and 7
Primary Hydroxyl Content	34 to 39
Sulfur or Sulfate Content	40 to 45
Viscosity	63 and 66

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 1193 Specification for Reagent Water²
D 1343 Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method³
D 2929 Test Method for Sulfur Content of Cellulosic Materials by X-Ray Fluorescence³

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.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.

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 D01.36 on Cellulose and Cellulose Derivatives.

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² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ "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."

3.2 Unless otherwise indicated, references to water shall be understood to mean reagent tared, low, wide-form weighing bottle and water, conforming to Specification D 1193.

MOISTURE CONTENT

4. Significance and Use

4.1 Moisture content of the cellulose ester can be used to estimate the dry weight of the cellulose ester. Since cellulose esters are desiccants, their moisture content can vary greatly depending on storage.

5. Procedure

5.1 Transfer about 5 g of the sample to a tared, low, wide-form weighing bottle and weigh to the nearest 0.001 g. Dry in an oven for 2 h at $105 \pm 3^\circ\text{C}$. Remove the bottle from the oven, cover, cool in a desiccator, and weigh.

6. Calculation

6.1 Calculate the percentage of moisture as follows:

$$\text{Moisture, \%} = (A/B) \times 100$$

where:

A = weight loss on heating, g, and

B = sample used, g.

7. Precision and Bias

7.1 No statement on bias can be made as no reference material is available as a standard.

ASH

8. Significance and Use

8.1 Ash content gives an estimate of the inorganic content of cellulose ester samples. The presence of high levels of inorganic content (ash) can be detrimental to the melt stability and optical clarity of a cellulose ester in melt processing or act as a potential source of insolubles when the ester is used in solution.

9. Procedure

9.1 Dry the sample for 2 h at $105 \pm 3^\circ\text{C}$ and weigh 10 to 50 g, to the nearest 0.01 to 0.1 g, depending on its ash content and the accuracy desired. An air-dried sample may be used and calculated to dry weight using the value for moisture determined as in Sections 5 and 6. Burn directly over a flame in a 100-mL tared platinum crucible that has been heated to constant weight and weighed to the nearest 0.1 mg. Add the sample in portions if more than 10 g is taken. The sample should burn gently and the portions should be added as the flame subsides. Continue heating with a burner only as long as the residue burns with a flame.

Transfer the crucible to a muffle furnace and heat at 550 to 600°C for 3 h, or longer if required, to burn all the carbon. Allow the crucible to cool and then transfer it, while still hot, to a desiccator. When the crucible has cooled to room temperature, weigh accurately to the nearest 0.1 mg.

10. Calculation

10.1 Calculate the percentage of ash as follows:

$$\text{Ash, \%} = (A/B) \times 100$$

where:

A = ash, g, and

B = sample used, g.

11. Precision and Bias

11.1 No statement on bias can be made as no reference material is available as a standard.

FREE ACIDITY

12. Significance and Use

12.1 Free Acidity is a measure of unesterified organic acid in the ester. The presence of high levels of free acid is potentially detrimental to the melt processing of the ester and can impact the odor of the ester.

13. Reagents

13.1 *Phenolphthalein Indicator Solution* (1 g/100 mL)—Dissolve 1 g of phenolphthalein in 100 mL of ethyl alcohol (95 %).

13.2 *Sodium Hydroxide, Standard Solution* (0.01 N)—Prepare and standardize a 0.01 N solution of sodium hydroxide (NaOH).

14. Procedure

14.1 Shake 5 g of the sample, ground to pass a No. 20 (850 μ m) sieve and corrected for moisture content if necessary, in a 250-mL Erlenmeyer flask with 150 mL of freshly boiled, cold water. Stopper the flask and allow it to stand for 3 h. Filter off the cellulose acetate and wash it with water. Titrate the combined filtrate and washings with 0.01 N NaOH solution, using phenolphthalein indicator solution.

14.2 Run a blank determination on the water, using the same volume as was used in extracting the sample.

15. Calculation

15.1 Calculate the percentage of acidity as free acetic acid as follows:

$$\text{Free acetic acid, \%} = [(A - B)N \times 0.06 \times 100]/W \quad (1)$$

where:

A = NaOH solution used to titrate the sample, mL,

B = NaOH solution used to titrate the blank, mL,

N = normality of the NaOH solution, and

W = sample used, g.

16. Precision and Bias

16.1 No statement on bias can be made as no reference material is available as a standard.

COMBINED ACETYL OR ACETIC ACID CONTENT

17. Scope

17.1 Two test methods are described for determining the combined acetyl or acetic acid content. The first, described in Sections 19 to 22, is more precise, but less widely applicable, than the method described in Sections 24 to 26.

18. Significance and Use

18.1 Acetyl or acetic acid content is a measure of the amount of acetic acid esterified onto the cellulose backbone of the polymer. The amount of substitution of acetate ester has a very strong effect on the polymer's solubility and physical properties.

Test Method A—Solution Method

19. Apparatus

19.1 *Weighing Bottle*, glass-stoppered, 15-mL capacity, 25-mm diameter by 50-mm high.

19.2 *Tray*, copper or aluminum, approximately 5 $\frac{3}{4}$ in. (136.5 mm) square, containing 25 compartments 1 in. (25.4 mm) square. Each compartment shall have the correct dimensions to contain one weighing bottle. The entire tray shall fit inside a desiccator and should have a basket-type handle to facilitate the introduction and removal of the tray (convenient but not essential).

19.3 *Buret*, automatic zero, 35-mL, 25-mL bulb, stem graduated from 25 to 35 mL in 0.05-mL increments; or pipet, automatic zero, 30-mL, for 1.0 N NaOH solution.

19.4 *Buret*, automatic zero, 15-mL, 10-mL bulb, stem graduated from 10 to 15 mL in 0.05-mL increments, for 1 N H₂SO₄.

19.5 *Buret*, 5-mL, in 0.01 or 0.1-mL divisions, for back titration with 0.1 N NaOH solution.

19.6 *Magnetic Stirrer*, for single flask.

19.7 *Magnetic Stirrer*, capacity twelve or more flasks.

19.8 *Stirring Bars*, stainless steel Type 416, length 50 mm, diameter 5 to 6 mm, or equivalent, dimensions not critical.

20. Reagents

20.1 *Acetone*—Add one 30-mL portion of 1.0 N NaOH solution to a mixture of 150 mL acetone and 100 mL hot water, allow to stand with frequent swirling for 30 min, and titrate with 1.0 N H₂SO₄. Add another 30-mL portion of 1.0 N NaOH solution to 100 mL of hot water, allow to stand for 30 min, and titrate. The difference between the two titrations shall not exceed 0.05 mL.

20.2 *Dimethyl Sulfoxide*.

20.3 *Pyridine*.

20.4 *Sodium Hydroxide Solution* (40 g/L)—Dissolve 40 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

20.5 *Sodium Hydroxide, Standard Solution* (0.1 N)—Prepare and standardize a 0.1 N solution of NaOH.

20.6 *Sulfuric Acid* (1.0 N)—Prepare and standardize a 1.0 N solution of sulfuric acid (H₂SO₄).

20.7 *Phenolphthalein Indicator Solution* (1 g/100 mL)—Dissolve 1 g of phenolphthalein in 100 mL of ethyl alcohol (95 %).

21. Procedure

21.1 Dry 1.9 $\pm$ 0.05 g of the ground well-mixed sample in

a weighing bottle for 2 h at $105 \pm 3^\circ\text{C}$ and weigh the dried sample by difference to the nearest 1 mg into a 500-mL wide-mouth Erlenmeyer flask. Prepare a blank by drying approximately 3.8 g of potassium acid phthalate and weighing it by difference into a flask as described. Carry the blank through the entire procedure.

NOTE 1—Potassium acid phthalate is used so that the concentration of the NaOH in contact with the solvent in the blank will be approximately the same as that in contact with the sample and so that the titration of the blank will be approximately the same as the titration of the sample, thus avoiding errors caused by using a different buret for the titration of the blank and the sample or by refilling the 15-mL buret. If desired, however, the potassium acid phthalate may be omitted.

21.2 If the acetyl content is 32 to 41 % or the acetic acid content is 45 to 57 %, put the sample into solution as follows: Add 150 mL of acetone and 5 to 10 mL of water and swirl to mix. Stopper the flask and allow it to stand with occasional swirling until solution is complete. Solution may be hastened by magnetic stirring or by any suitable mechanical shaking that will provide a gentle rocking type of agitation to avoid splashing the solution on the stopper. It is essential that complete solution be effected. Proceed in accordance with 21.4.

21.3 If the acetyl content is 41 to 44.8 % or the acetic acid content is 57 to 62.5 %, dissolve the sample by either of the following two methods:

21.3.1 Gently rotate the flask by hand to distribute and spread the sample in a thin layer over the bottom of the flask. Add 70 mL of acetone to the flask and swirl gently until the sample particles are completely wetted and evenly dispersed. Stopper the flask and allow it to stand undisturbed for 10 min. Carefully add 30 mL of dimethyl sulfoxide from a graduate to the flask, pouring the solvent down the sides of the flask to wash down any sample particles clinging to the side. Stopper the flask and allow it to stand with occasional swirling until solution is complete. Magnetic stirring or gentle mechanical agitation that will not splash the solution is recommended. When solution appears to be complete, add 50 mL of acetone and swirl or stir for 5 min. Proceed in accordance with 21.4.

21.3.2 Dimethyl sulfoxide is the preferred solvent, but if it is not available, spread the sample in a thin layer over the bottom of the flask, add 15 mL of acetone, swirl to wet the particles with acetone, stopper the flask, and allow the mixture to stand undisturbed for 20 min. Add 75 mL of pyridine without shaking or swirling, and allow to stand for 10 min. Heat the solution just to boiling and swirl or stir for 5 min. Again heat to boiling and swirl or stir for 10 min. Continue to heat and stir until the mixture is homogeneous and all large gel masses are broken down into individual highly swollen particles. When these highly swollen gel particles are well dispersed and are not fused together in large gel masses, no further heating is necessary. Cool the flask, add 30 mL of acetone, and swirl or stir for 5 min. Proceed in accordance with 21.4.

21.4 Add 30 mL of NaOH solution (40 g/L) with constant swirling or stirring to the solution of the sample and also to the blank. Use of a magnetic stirrer is recommended (Note 2). It is absolutely necessary that a finely divided precipitate of regenerated cellulose, free from lumps, be obtained. Stopper the flask and let the mixture stand with occasional

swirling, or stir on the magnetic stirring unit. Allow 30 min for saponification of lower acetyl samples, 2 h for high acetyl samples when dimethyl sulfoxide is the solvent, and 3 h when pyridine is the solvent. At the end of the saponification period, add 100 mL of hot water, washing down the sides of the flask, and stir for 1 or 2 min. Add 4 or 5 drops of phenolphthalein indicator solution and titrate the excess NaOH solution with 1.0 N H_2SO_4 (Note 3). Titrate rapidly with constant swirling or stirring ring until the end point is reached; then add an excess of 0.2 or 0.3 mL of H_2SO_4 . Allow the mixture to stand with occasional stirring or preferably stir on the magnetic stirrer for at least 10 min. Then add 3 drops of phenolphthalein indicator solution to each flask and titrate the small excess of acid with 0.1 N NaOH solution to a persistent phenolphthalein end point. Take extreme care to locate this end point; after the sample is titrated to a faint pink end point, swirl the mixture vigorously or place it for a moment on the magnetic stirrer. If the end point fades because of acid soaking from the cellulose, continue the addition of 0.1 N NaOH solution until a faint persistent end point remains after vigorous swirling or stirring. Titrate the blank in the same manner as the sample.

NOTE 2—While the amount of magnetic stirring is somewhat optional, such stirring during the entire period of the determination is strongly recommended. Solution is more rapid, titrations are more rapid, and the end point can be approached directly and without a back titration.

NOTE 3—It is important to correct all 1.0 N H_2SO_4 buret readings for temperature and buret corrections.

22. Calculation

22.1 Calculate the percentage by weight of acetyl and acetic acid as follows:

$$\begin{aligned} \text{Acetyl or acetic acid, \%} \\ = [(D - C)N_a + (A - B)N_b + P] \times (F/W) \quad (\text{Note 4}) \quad (2) \\ P = (GH \times 1000)/204.2 \end{aligned}$$

where:

A = NaOH solution required for titration of the sample, mL,

B = NaOH solution required for titration of the blank, mL,

N_b = normality of the NaOH solution,

C = H_2SO_4 required for titration of the sample, mL,

D = H_2SO_4 required for titration of the blank, mL,

N_a = normality the H_2SO_4 ,

F = 4.305 for acetyl and 6.005 for acetic acid,

P = milliequivalents of potassium acid phthalate,

G = potassium acid phthalate used, g,

H = purity factor for potassium acid phthalate, and

W = sample used, g.

NOTE 4—When equal volumes of alkali or acid are added to samples and blank, these amounts cancel out. Thus only the amounts of each added in the titration enter into the calculations. Use of potassium acid phthalate in the blank is recommended. When it is not used, the term P drops out of the equation.

23. Precision and Bias

23.1 No statement on bias can be made as no reference material is available as a standard.

Test Method B—Heterogeneous
Saponification Method

Reagents

- 25.1 **Ethyl Alcohol (75 Volume %)**—Mix 790 mL of formula 2B, 3A, or 30 denatured ethyl alcohol and 210 mL of water.
- 25.2 **Hydrochloric Acid (0.5 N)**—Prepare and standardize 0.5 N solution of hydrochloric acid (HCl).
- 25.3 **Sodium Hydroxide, Standard Solution (0.5 N)**—Prepare and standardize a 0.5 N solution of sodium hydroxide (NaOH).

Procedure

- 25.1 Grind the sample in a Wiley mill or other suitable grinder so that 100 % will pass a No. 20 (850- μ m). (Grinding may be omitted if the sample has suitable texture.) Dry about 10 g of the sample in a weighing bottle at $105 \pm 3^\circ\text{C}$ for 2 h, stopper, and cool in a desiccator. (An oven with mechanical circulation is to be preferred over a convection-type oven).
- 25.2 Weigh the bottle containing the sample to the nearest 0.001 g, transfer the sample to a 250-mL Erlenmeyer flask, and weigh the bottle again to the nearest 0.001 g. Handle the bottle with either tongs or a clean dry cloth during these manipulations. Add 40 mL of ethyl alcohol (75 %) to each sample. Include a blank determination with each set of samples and carry the blank determination through the complete procedure, including the back titration.
- 25.3 Heat the flasks, loosely stoppered, for 30 min at 50 to 60°C. Add 40 mL of 0.5 N NaOH solution to each flask and heat again at 50 to 60°C for 15 min. Stopper the flasks tightly and allow to stand at room temperature for about 48 h. If the acetyl content of the sample is over 43 %, or if the sample is hard and horny, allow to stand for about 72 h. At the end of this time back titrate the excess NaOH with 0.5 N HCl, using phenolphthalein as the indicator. Add an excess of about 1 mL of 0.5 N HCl and allow the NaOH to diffuse from the regenerated cellulose for several hours, or, preferably overnight. The disappearance of the pink color indicates the complete neutralization of the NaOH. Titrate the small excess of HCl with 0.5 N NaOH solution to a phenolphthalein end point. Extreme care must be taken to locate this end point. After the sample is titrated to a faint pink end point, stopper the flask and shake vigorously. The end point may fade because of acid diffusing from the cellulose. Continue the addition of 0.5 N NaOH solution and shaking until the faint pink end point persists after vigorous shaking of the flask.

26. Calculation

26.1 Calculate the percentage of combined acetyl or acetic acid as follows:

$$\text{acetyl or acetic acid, \%} = [(D - C)N_a + (A - B)N_b] \times (F/W) \quad (3)$$

where:

- A = NaOH solution required for titration of the sample, mL,
 B = NaOH solution required for titration of the blank, mL,
 N_b = normality of the NaOH solution,
 C = HCl required for titration of the sample, mL,

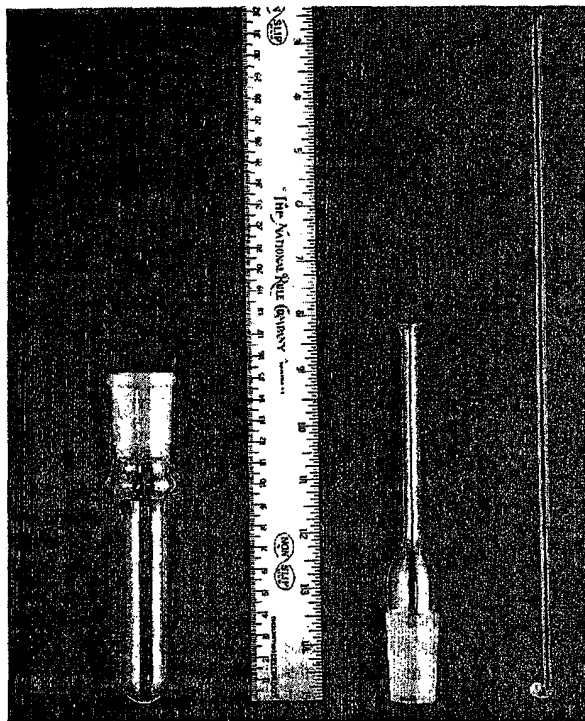


FIG. 1 Special Reflux Tube for Carbanilation

- D = HCl required for titration of the blank, mL,
 N_a = normality of the HCl solution,
 F = 4.305 for acetyl or 6.005 for acetic acid, and
 W = sample used, g.

HYDROXYL CONTENT

27. Scope

27.1 This test method is applicable to pyridine-soluble cellulose esters and is especially useful when the hydroxyl content is low. Samples containing plasticizer may be analyzed directly by this test method because the plasticizer is removed during washing of the carbanilate.

28. Summary of Test Method

29.1 Hydroxyl in cellulose acetate is determined by reaction with phenyl isocyanate in pyridine solution under anhydrous conditions to form the carbanilate derivative. The derivative is then analyzed for its carbanilate content by ultraviolet absorption.

29.2 The acetyl content of cellulose acetates may be calculated provided that the degree of polymerization is not excessively low.

29. Significance and Use

29.1 Hydroxyl content is a measure of the free hydroxyl on the cellulose backbone of the polymer. Hydroxyl content has a strong effect on the polymer's solubility and physical properties. Hydroxyl content also impacts the propensity for this polymer to crosslink with various crosslinking agents.

30. Apparatus

30.1 *Spectrophotometer*,⁵ complete with hydrogen light source and a set of four 1.00-cm quartz cells, or an equally suitable apparatus. The wavelength calibration, as checked against a mercury lamp, shall be within the manufacturer's tolerances. As a further check, measure the absorbance of a potassium chromate (K_2CrO_4) solution prepared as follows: Dissolve 0.0400 g of K_2CrO_4 or 0.0303 g of potassium dichromate $K_2Cr_2O_7$ in 0.05 N potassium hydroxide (KOH) solution and dilute to 1 litre in a volumetric flask with 0.05 N KOH solution. Using the hydrogen lamp, measure the absorbance at 280 nm of a silica cell filled with the K_2CrO_4 solution and also of the same cell filled with water. The absorbance of the solution minus that of the blank shall be 0.723 ± 0.023 .

30.2 *Bottles*, 4-oz, with screw caps, for washing the samples.

30.3 *Special Reflux Tubes* for the carbanilation, constructed as follows (see Fig. 1): Make a test tube approximately 20 by 150 mm from the outer part of a 24/40 standard-taper ground glass joint by closing the open end in a blast lamp. Draw the tubing on the inner joint to a constriction just above the joint. Cut the glass at that point and seal on a short length of 8-mm tubing to provide a bearing for a glass stirrer. Make a stirrer of 4-mm glass rod with a semicircle at right angles to the shaft at the bottom and small enough to fit into the test tube. When properly constructed this unit acts as an air condenser, thus preventing the loss of solvent by evaporation.

30.4 *Pipet*, serological-type, 5-mL capacity, graduated in 0.1-mL divisions.

30.5 *Büchner Funnel*, of a size accommodating 90-mm filter paper.

30.6 *Automatic Shaker*, with speed regulator mechanism.

30.7 *Electric Oven*, maintained at $105 \pm 3^\circ C$.

30.8 *Oil Bath*, equipped with a rack to hold several of the special reflux tubes. This bath shall be kept between 115 and $120^\circ C$.

31. Reagents

31.1 *Acetone*.

31.2 *Ethyl Alcohol*, denatured, Formula 2B, 3A, or 30.

31.3 *Methylene Chloride-Methyl Alcohol Mixture*—Mix 9 parts by weight of methylene-chloride with 1 part of methyl alcohol. This mixture should have an absorbance of less than 0.2 at 280 nm in a 1.00-cm silica cell measured against air. Pure methylene chloride has an absorbance of about 0.05, but the commercial product may have an absorbance as high as 1.00. The methylene chloride and methanol should be selected to have low absorbance; otherwise, they should be redistilled.

31.4 *Phenyl Isocyanate*.⁶

31.5 *Pyridine*, redistilled, low water content, preferably less than 0.05 %.

32. Procedure

32.1 In the following procedure the phenyl isocyanate reagent shall be used under anhydrous conditions. Therefore, the sample, containers, pipet, and all other equipment shall be thoroughly dried.

32.2 Place a 0.5-g sample in a special reflux tube and dry in an electric oven at $105^\circ C$ for 2 h. Remove the tube from the oven, add 5 mL of pyridine, assemble the reflux apparatus complete with glass stirring rod, and place in the 115 to $120^\circ C$ oil bath. Stir occasionally until the sample is completely dissolved. Add 0.5 mL of phenyl isocyanate, stir thoroughly, and reflux in the oil bath for $\frac{1}{2}$ h to complete the reaction. Use 0.1 mL of phenyl isocyanate for each percent of estimated hydroxyl content, but never less than 0.5 mL.

32.3 At the end of the reaction time, remove the sample and dilute it with acetone to the proper viscosity for precipitation. The amount of acetone used to thin the solution is a critical factor in acquiring a good precipitate. Samples having low viscosity require little, if any, dilution. The average sample requires the addition of about an equal volume of acetone. Precipitate the carbanilate by pouring the solution into about 200 mL of ethyl alcohol. The precipitate should be fluffy and white. Sticky precipitates indicate too little dilution. Stir the alcohol vigorously during precipitation. Filter off the precipitate, using paper on a Büchner funnel, with suction applied only as long as is necessary to remove the bulk of the solvent; prolonged suction may cause undesirable clumping together of the precipitate.

32.4 Wash by transferring the precipitate to a 4-oz screw cap bottle containing 75 mL of ethyl alcohol, capping securely, and shaking for $\frac{1}{2}$ h on an automatic shaker at medium speed. Filter the precipitate on the Büchner funnel, pressing out as much liquid as possible with a glass stopper. Repeat the washing and filtering operations twice more. Allow the precipitate to air-dry 1 to 2 h at room temperature with good ventilation or preferably overnight to ensure complete removal of the alcohol. (Samples wet with alcohol may sinter and stick to paper or glass when dried at $105^\circ C$.) Dry the sample at $105^\circ C$ in the oven for 1 h and cool in a desiccator. Small manila envelopes are convenient for drying and cooling the samples.

32.5 Weigh 0.1231 g of the dry precipitate into a 100-mL volumetric flask fitted with a ground-glass stopper. Add 60 to 80 mL of the methylene chloride-methyl alcohol mixture, and shake occasionally until complete solution occurs. Dilute to 100 mL and mix thoroughly. Using the spectrophotometer with a 1-cm silica cell measure the absorbance of the solution at 280 nm against the solvent mixture as a reference.

33. Calculation

33.1 Calculate the percentage of carbanilate, c , for a sample weight of 0.1231 g as follows:⁷

$$\text{Carbanilate, \%} = A \times 17.1 \quad (4)$$

where:

A = absorbance.

33.2 Calculate the percentage of hydroxyl as follows:

⁵ A Beckman Model DU Spectrophotometer has been found satisfactory for this purpose.

⁶ Phenyl isocyanate available as Eastman No. 553 has been found satisfactory for this purpose.

⁷ Malm, C. J., Tanghe, L. J., Laird, B. C., and Smith, G. C., "Determination of Total and Primary Hydroxyl in Cellulose Esters by Ultraviolet Absorption Methods," *Analytical Chemistry*, ANCHA, Vol 26, 1954, p. 189.

$$\text{Hydroxyl, \%} = 14.3c/(100 - c)$$

Calculate the percentage of acetyl as follows:

$$\text{Acetyl, \%} = (4480 - 65.1c)/(100 - c)$$

NOTE 5—The calculation for acetyl content assumes exactly three acetyl groups per anhydroglucose unit and applies to cellulose acetates only.

PRIMARY HYDROXYL CONTENT

Summary of Test Method

The primary hydroxyl content of cellulose acetate is determined by formation of the triphenylmethyl (trityl) ether. Measurement of the trityl group by ultraviolet absorbance.⁷ Trityl chloride reacts preferentially with primary hydroxyls. Since there is also a slight reaction with secondary hydroxyls, standardized reaction conditions are important.⁸

Apparatus

35.1 See Section 30.

Reagents

36.1 Acetone.

36.2 Ethyl Alcohol, denatured, Formula 2B, 3A, or 30.

36.3 Methylene Chloride-Methyl Alcohol Mixture—Mix 9 parts by weight of methylene chloride with 1 part of methyl alcohol. This mixture should have an absorbance of less than 0.02 at 259 nm in a 1-cm silica cell measured against air; otherwise the solvents should be redistilled.

36.4 Pyridine, redistilled to a water content less than 0.05 %. The water content may be reduced further by storing over a suitable drying agent, such as a molecular sieve.⁹

36.5 Trityl Chloride (Chlorotriphenylmethane or Triphenylmethyl Chloride), reagent grade.

Procedure

37.1 The reagents shall be used under anhydrous conditions. It is imperative that the sample and all equipment be thoroughly dry.

37.2 Place a 0.5-g sample in the test tube of the special reflux apparatus and dry for 2 h at $105 \pm 3^\circ\text{C}$. Add 5 mL of pyridine, insert the top of the reflux apparatus and the stirrer and heat with stirring in a 115 to 120°C oil bath. After the sample has dissolved, add 0.5 g of trityl chloride. If the total hydroxyl content exceeds 3 %, use an additional 0.075 g of trityl chloride for each additional 1 % hydroxyl. Stir the mixture thoroughly and reflux in the oil bath for exactly 2 h at 115 to 120°C . Remove the tube and cool.

37.3 Dilute the sample with acetone to the proper viscosity for precipitation. The amount of acetone used to thin the solution is a critical factor in obtaining a good precipitate. Samples having low viscosity require little, if any, dilution. The average sample requires the addition of about an equal volume of acetone. Precipitate the trityl derivative by pouring the solution into about 200 mL of ethyl alcohol with vigorous stirring. The precipitate should be fluffy and

white. Sticky precipitates indicate too little dilution. Separate the precipitate by filtering through paper on a Büchner funnel, with suction applied only as long as necessary to remove the bulk of the solvent; prolonged suction may evaporate the alcohol and cause the precipitate to partially redissolve in the remaining pyridine.

37.4 Wash the precipitate by transferring it to a 4-oz screw cap bottle containing 75 mL of ethyl alcohol, capping securely, and shaking for $\frac{1}{2}$ h on a shaker at medium speed. Again collect the precipitate on a Büchner funnel, pressing out as much liquid as possible with a glass stopper. Repeat this washing and filtering operation twice more, or until the absorbance of the filtrate at 259 nm is about the same as that of an alcohol blank. Allow the precipitate to air-dry on the filter paper for $\frac{1}{2}$ h at room temperature with good ventilation, or preferably overnight, to remove most of the alcohol. (Samples wet with alcohol may sinter or stick to paper or glass when dried at 105°C .) Transfer the sample to a manila envelope, dry it for 1 h at 105°C , and cool in a desiccator.

37.5 Weigh 0.1231 g of the dry trityl ether derivative into a 100-mL volumetric flask fitted with a ground-glass stopper, and dissolve in the methylene chloride-methyl alcohol mixture. Dilute to 100 mL and mix thoroughly. Measure the absorbance of this solution in a 1-cm silica cell using a spectrophotometer at 259 nm against the solvent as a reference.

38. Calculation

38.1 Calculate the trityl content, t , for this concentration of 0.1 g/100 g and with a correction of 0.015 for the absorbance of the cellulose acetate as follows:⁷

$$\text{Trityl, \%} = 25.25 (A - 0.015) \quad (5)$$

where:

A = absorbance.

38.2 Calculate the weight percentage of primary hydroxyl in cellulose acetate as follows:

$$\text{Primary hydroxyl, \%} = 7.02t/(100.4 - t) \quad (6)$$

38.3 Calculate the percentage primary hydroxyl of the total hydroxyl as follows:

$$\text{Primary hydroxyl of total hydroxyl, \%} = (B/C) \times 100 \quad (7)$$

where:

B = value of primary hydroxyl as determined in 38.2, and
 C = value of total hydroxyl as determined in 33.2.

39. Precision and Bias

39.1 No statement on bias can be made as no reference material is available as a standard.

SULFUR OR SULFATE CONTENT

40. Summary of Test Method

40.1 The sulfur or sulfate content of cellulose acetate is measured by oxidizing the sample in a nitric acid-perchloric acid mixture and determining gravimetrically as barium sulfate. To determine combined sulfur the sample must first be reprecipitated into dilute acid to remove noncombined sulfur compounds.

40.2 The sulfur or sulfate content may also be determined by Test Method D 2929. In this case the X-ray method shall be calibrated against the chemical method following in

⁸ Malm, C. J., Tanghe, L. J., and Laird, B. C., "Primary Hydroxyl Groups in Hydrolyzed Cellulose Acetate," *Journal of the American Chemical Society*, JACSA, Vol 72, 1950, p. 2674.

⁹ Molecular Sieve Type 4A as manufactured by the Linde Air Products Co., has been found satisfactory for this purpose.

Sections 42 to 45, and the sample shall be treated in accordance with Section 44 if combined sulfur is to be determined.

41. Significance and Use

41.1 Sulfur and sulfate content indicates the amount of sulfur in the cellulose ester either as inorganic salts (usually sulfates) or as organic sulfate (usually as sulfate ester combined to the cellulose backbone). The presence of high levels of sulfur and sulfate can be detrimental to the melt stability of the ester.

42. Apparatus

42.1 *Funnel*, modified by cutting the stem off at the apex of the funnel and fire polishing.

42.2 *Crucibles*,¹⁰ 30-mL, extra-fine porosity.

42.3 *Oven*, controlled at 120 to 125°C.

42.4 *Muffle Furnace*, controlled at $800 \pm 50^\circ\text{C}$.

43. Reagents

43.1 *Acetone*.

43.2 *Acetic Acid* (1+49)—Mix 1 volume of glacial acetic acid with 49 volumes of water.

43.3 *Barium Chloride Solution* (100 g/L)—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

43.4 *Hydrochloric Acid* (1+1)—Mix 1 volume of concentrated hydrochloric acid (HCl , sp gr 1.19) with 1 volume of water.

43.5 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

43.6 *Nitric Acid* (2+3)—Mix 2 volumes of concentrated HNO_3 (sp gr 1.42) with 3 volumes of water.

43.7 *Nitric Acid-Perchloric Acid Mixture*—Mix 5 volumes of concentrated HNO_3 with 1 volume of concentrated perchloric acid (HClO_4 , 70 %).

43.8 *Phenolphthalein Indicator Solution* (1 g/100 mL)—Dissolve 1 g of phenolphthalein in 100 mL of ethyl alcohol (95 %).

43.9 *Silver Nitrate Solution* (50 g/L)—Dissolve 50 g of silver nitrate (AgNO_3) in water and dilute to 1 L.

43.10 *Sodium Carbonate* (Na_2CO_3).

43.11 *Sodium Hydroxide Solution* (400 g/L)—Dissolve 400 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

44. Procedure

44.1 *Treatment Prior to Analysis*—Remove uncombined sulfur as follows (Note 6): Dissolve 25 g of sample in approximately 300 mL of acetone, depending on the viscosity. If the sample is of too high acetyl content to be directly soluble in acetone, cool in a dry ice cabinet overnight; then allow to come to room temperature while tumbling or stirring. Filter the solution, if necessary, through felt or a coarse sintered-glass crucible. Precipitate with rapid stirring into a beaker or pail containing 2 to 3 L of acetic acid (1+49). Filter through a cloth bag or a Büchner funnel and give two 15-min washes with water using mechanical agita-

tion. A little Na_2CO_3 may be added to the last wash to stabilize samples of high sulfur content. Filter and dry overnight at 60°C .

NOTE 6—To analyze for total sulfur content omit this treatment.

44.2 Decomposition:

44.2.1 Weigh 10 ± 0.1 g of cellulose acetate and transfer to a clean, wide-mouth, 500-mL Erlenmeyer flask. Add 50 mL of the $\text{HNO}_3\text{-HClO}_4$ mixture to the flask, and swirl the flask gently to wet the sample thoroughly. Place the modified funnel in the mouth of the flask and heat the flask carefully on a hot plate in a fume hood.

NOTE 7: Precaution—Use the utmost care in handling the $\text{HNO}_3\text{-HClO}_4$ mixture. If a spill occurs, wash down with plenty of water. Wear safety glasses or a face shield.

44.2.2 After the mixture becomes hot and less viscous, increase the heat of the hot plate. Continue the digestion until all the sample has been oxidized and the thick reddish-brown fumes of nitrogen dioxide (NO_2) have been expelled. At this point, white fumes will appear and a rather vigorous reaction will occur that is caused by the last traces of organic material being oxidized and the HNO_3 fuming off.

44.2.3 When this reaction starts, remove the flask from the hot plate, swirl gently for a few seconds, and set it on the shelf in front of the hood until the reaction is complete. Place the flask back on the hot plate and continue the digestion until the HClO_4 refluxes about half way up the side of the Erlenmeyer flask and about 5 mL is left in the flask. The $\text{HNO}_3\text{-HClO}_4$ mixture should be clear and colorless. If it is not, set the flask off the hot plate to cool and then add 3 to 5 mL of HNO_3 (sp gr 1.42). Replace the flask on the hot plate and continue heating until the HClO_4 refluxes half way up the flask. Remove the flask from the hot plate and allow the flask and its contents to cool.

44.3 Determination of Barium Sulfate:

44.3.1 Wash the modified funnel top thoroughly with water, collecting the rinsings in the flask. Add 50 mL of water. Swirl the flask to mix the solution thoroughly. Add 2 drops of phenolphthalein indicator solution and neutralize the acid with the NaOH solution to a faint pink. Acidify immediately with HCl (1+1), dropwise, until the solution is just acid to phenolphthalein; then add 2 mL of HCl (1+1).

44.3.2 Filter through a 12.5-cm fine-porosity paper into a clean 400-mL beaker. Wash the flask thoroughly with water, filtering the washings through the paper. Finally wash the paper thoroughly with ten portions of hot water. Dilute the filtrate to approximately 200 mL. Place the beaker on the hot plate and heat almost to boiling. Slowly add 10 mL of BaCl_2 solution, from a pipet, stirring the solution during the addition. Do not add the BaCl_2 solution rapidly, as from a graduate, since the rapid addition will produce an impure precipitate. Remove the stirring rod from the beaker and wash it with a stream of water from the wash bottle, collecting the washings in the beaker. Cover the beaker with a watch glass and keep the mixture near the boiling temperature of 6 h or overnight. Do not allow the liquid to evaporate to dryness.

44.3.3 Using suction, decant the supernatant liquid through an extra-fine porosity porcelain filter crucible that has been previously rinsed with acetone, ignited, and weighed to the nearest 0.1 mg. Transfer the precipitate with

¹⁰ Selas No. 3001 crucible has been found satisfactory for this purpose.

aid of a stream of hot water. Always use a stirring rod in transfer. Scrub the sides and bottom of the beaker with a rubber policeman to remove any adhering precipitate. The crucibles may be used to collect several precipitates one on top of the other. Close control of temperature and time of heating and cooling are necessary. Cleaning with hot water is generally sufficient; drastic attack with cleaning solution should be avoided.

44.3.4 Wash the precipitate on the filter until free of chlorides by the following test: To 5 mL of wash water, collected in a separate test tube or on a watch glass, add 1 mL of HNO_3 (2+3) and 1 mL of AgNO_3 solution. The appearance of a milky white precipitate indicates the presence of chlorides, and the washing should therefore continue until the test is negative. Do not attempt to get a completely negative test for chloride. Discontinue washing when no more than a faint opalescence is produced in the test.

44.3.5 Finally pour a few millilitres of pure acetone through the filter and suck it dry. Place the crucible in a larger crucible or in a metal tray with perforated sides and bottom for protection and place it in an oven at 120°C for 1 h. Do not handle the crucibles with the fingers between ignition and the completion of weighing; use forceps.

44.3.6 Remove the crucible from the oven and ignite it for 10 min in a muffle furnace at $800 \pm 50^\circ\text{C}$. Cool in a desiccator for 75 ± 15 min and weigh to the nearest 0.0001 g. It is permissible to return the crucible to the oven for at least 5 min before transferring to the desiccator.

44.3.7 From time to time, and especially when using new reagents, run a blank in duplicate in the reagents. If the weight of the precipitate exceeds 0.0005 g, investigate and eliminate the cause. This is equivalent to an error of 0.002 % on a 10-g sample.

45. Calculation

45.1 Calculate the percentage of sulfur and sulfate as follows:

$$\text{Sulfur, \%} = [(C - B) - (E - D)] \times 0.1374 \times 100 / A \quad (8)$$

$$\text{Sulfate, \%} = [(C - B) - (E - D)] \times 0.4115 \times 100 / A \quad (9)$$

where:

- A = weight of sample, g,
- B = weight of crucible for sample, g,
- C = weight of crucible and BaSO_4 for sample, g,
- C - B = weight of BaSO_4 for sample,
- D = weight of crucible for blank, g,
- E = weight of crucible and BaSO_4 for blank, g, and
- E - D = weight of BaSO_4 for blank, g.

46. Precision and Bias

46.1 No statement on bias can be made as no reference material is available as a standard.

HEAT STABILITY

47. Summary of Test Method

47.1 The heat stability of cellulose acetate is one indication of its quality. It is measured by heating the sample for a specified time and temperature, observing it for amount and uniformity of color developed, and possibly also measuring the loss of viscosity as a result of heating. Suggested times of heating are 8 h at 180°C or 2 h at 190°C . The time and

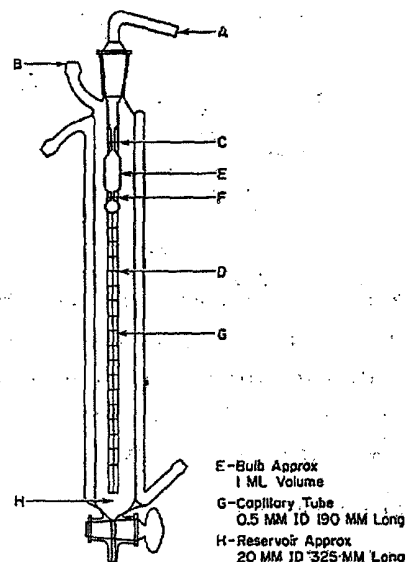


FIG. 2 Wagner Capillary Tube Viscometer¹¹

temperature of heating, method of grading, and limits are matters for agreement between purchaser and the supplier.

48. Significance and Use

48.1 The heat stability of a cellulose ester is one indication of its quality.

49. Apparatus

49.1 **Heater Block**—A metal block of suitable size is heated electrically and maintained at the specified temperature within $\pm 1^\circ\text{C}$. This is best accomplished by providing continuous heat to hold the temperature a few degrees below the specified temperature, and providing intermittent additional heat thermostatically controlled. Holes are drilled in the top of the block to hold test tubes, a thermoregulator, and a thermometer. The block should be insulated.

49.2 **Test Tubes**, either 18 by 150 mm or 20 by 150 mm, fitted with corks. The corks shall be fitted with glass tubes the length of the cork and 4 mm in inside diameter or shall have a small V-shaped notch of equivalent cross-section cut in a vertical position.

50. Solvent

50.1 **Methylene Chloride-Methanol Mixture**—Mix 9 parts by weight of methylene chloride with 1 part of methyl alcohol.

51. Heat Treatment

51.1 Place the sample, ground to pass a No. 20 (850- μm) sieve, in a clean dry test tube and pack it firmly and uniformly. Stopper with a cork having a notch or tube as described in 49.2. Heat the tube and contents for 8 h at

¹¹ Wagner, R. H., and Russell, John, "Capillary Tube Viscometer for Routine Measurement of Dilute High Polymer Solutions," *Analytical Chemistry*, ANCHA, Vol 20, 1948, pp. 151-157.

TABLE 1 Solvents for Intrinsic Viscosity Determination

Solvent ^A	Ingredients, weight %	Value of <i>k</i> for Calculation (see 61.2)
A	90 % acetone ^B 10 % ethyl alcohol ^C	10
B	acetone ^B	10
C or D	90 % methylene chloride ^D 10 % ethyl alcohol ^C	3
E	96 % acetone ^B 4 % water	10
F	90 % methylene chloride ^D 10 % methanol ^E	3

^A Solvent designations conform to those used in Table 2 for viscosity determinations.

^B Acetone (99.4 ± 0.1 %) containing 0.3 to 0.5 % water and under 0.3 % ethyl alcohol.

^C Ethyl alcohol (95 volume %). Formula 2B or 3A denatured ethyl alcohol may be used.

^D Methylene chloride having a boiling range of 39.2 to 40.0 °C and less than 0.001 % acidity calculated as HCl.

^E Methyl alcohol (sp gr 20/20°C = 0.785 to 0.795).

180°C or as otherwise specified.

52. Dry Color Evaluation

52.1 Examine the heated sample for uniformity of color and for the presence of charred or decomposed spots. Compare the color of the material at the bottom of the tube with standards prepared as follows: Heat portions of a check batch of similar particle size, representative quality and stability, and accepted by mutual agreement between the purchaser and the seller. Pack portions of this check batch firmly in each of twelve clean, dry test tubes and stopper with corks as described in 49.2. Heat the tubes at 180°C, or as otherwise specified, remove one tube each 2 h, and mark the time of heating in hours on each tube. This set of numbered tubes serves as the color standards. They should be checked and renewed if necessary every 6 months.

53. Solution Color Using Platinum - Cobalt Standards

53.1 Heat a 1-g sample for the specified time and temperature and, after cooling, examine for charred or decomposed spots. Dissolve the heated sample in 15 mL of the methylene chloride-methanol mixture. Compare the color of the solution (viewing transversely) with test tubes of platinum-cobalt color standards, prepared as described in Section 70. (It may be necessary to prepare standards having as much as 2000 ppm of platinum for this purpose or to dilute the sample solution before grading.)

54. Solution Color by Spectrophotometer

54.1 The color of the solution prepared as described in Section 53 may also be measured spectrophotometrically. Measure the absorbance at 400 nm against the solvent, using a suitable spectrophotometer with a 1-cm silica cell.

55. Viscosity Change

55.1 Measure the intrinsic viscosity of the heated sample and of an unheated sample as described in Sections 57 to 61 of this test method. The percentage loss of viscosity as the result of heating is a measure of heat stability.

56. Precision and Bias

56.1 No statement on bias can be made as no reference

TABLE 2 Solutions for Viscosity Determination

	Formula					
	A	B	C	D	E	F
	Ingredients, weight %					
Cellulose acetate	20 ^A	20 ^A	20 ^B	15 ^C	20 ^A	10 ^C
Acetone ^D	72	80	...	...	...	...
Acetone, 96 %	...	...	...	...	...	...
Water, 4 %	...	...	...	...	...	...
Ethyl alcohol ^C	8	...	8	8.5	...	...
Methyl alcohol ^F	...	...	...	...	...	9
Methylene chloride ^D	...	...	72	76.5	...	81
Typical Solution Densities, g/mL at 25 °C						
	0.84	0.86	1.25	1.23	0.86	1.24

^A Acetyl content 40.5 %, max.

^B Acetyl content 40.5 to 42.7 %.

^C Acetyl content 42.7 to 44.8 %.

^D Acetone (99.4 ± 0.1 %) containing 0.3 to 0.5 % water and under 0.3 % ethyl alcohol.

^E Ethyl alcohol (95 volume %). Formula 2B or 3A denatured ethyl alcohol may be used.

^F Methyl alcohol (sp gr 20/20 °C = 0.785 to 0.795).

^D Methylene chloride having a boiling range of 39.2 to 40.0°C and less than 0.001 % acidity calculated as HCl.

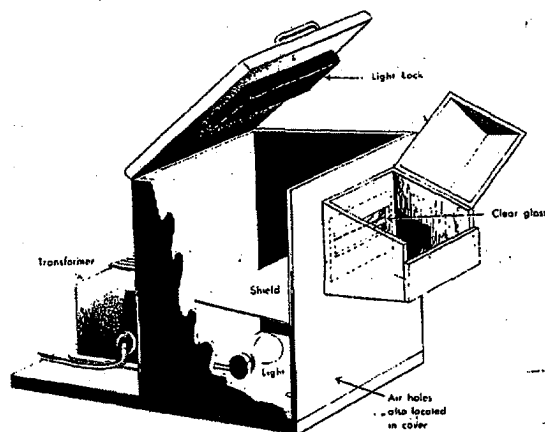


FIG. 3 Color and Haze Apparatus

material is available as a standard.

INTRINSIC VISCOSITY

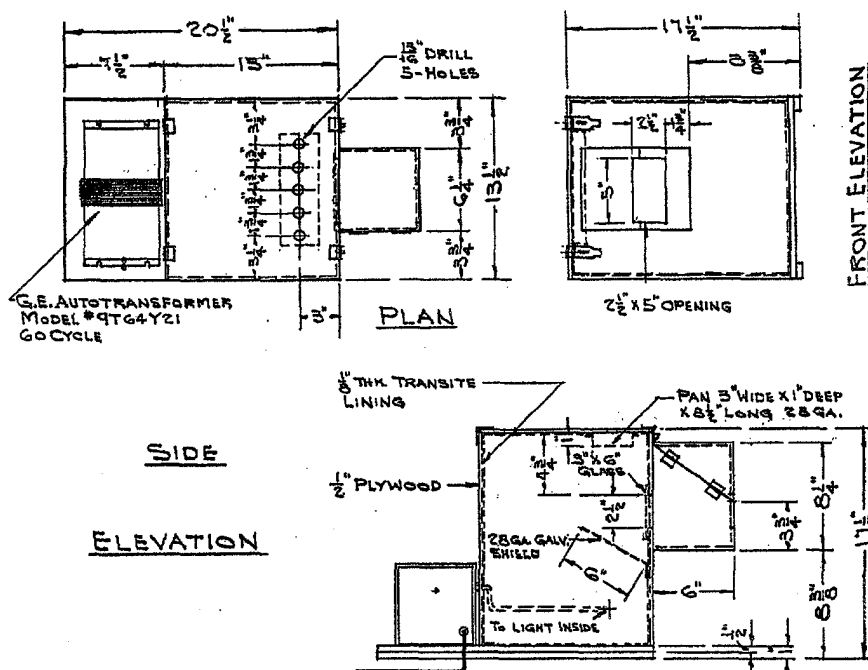
57. Summary of Test Method

57.1 Intrinsic viscosity, expressed in decilitres of solution per gram of solute, is determined by measuring the flow times of a solution of known concentration and also of the solvent used and making a calculation by means of the modified Baker-Philippoff equation.

NOTE 8—By expressing concentration in grams per millilitre rather than grams per decilitre, the result will be limiting viscosity number instead of intrinsic viscosity, and will be 100 times greater.

58. Significance and Use

58.1 Intrinsic viscosity number can be used to estimate the molecular weight of a cellulose ester by using the Mark-Houwink equation and constants measured for the solvent, temperature, and ester of concern.



Metric Equivalents

in.	mm	in.	mm	in.	mm	in.	mm	in.	mm
1/8	3.2	1 3/4	44.4	3 3/4	95.2	6 1/4	158.8	8 1/2	215.9
1/4	12.7	2 1/2	63.5	4 3/4	120.6	7 1/2	190.5	13	330.2
3/8	20.6	3	76.2	5	127.0	8 1/4	209.6	17 1/2	444.5
1	25.4	3 1/4	82.6	6	152.4	8 3/4	212.7	20 1/2	520.7

FIG. 4 Color and Haze Apparatus (Details)

59. Apparatus

59.1 *Capillary Viscometer*, such as the Wagner apparatus (Fig. 2) or an Ostwald-Fenske-Cannon pipet, that will give a flow time for the solvent of not less than 70 s.

59.2 *Water Bath*—A constant-temperature water bath controlled at $25.0 \pm 0.1^\circ\text{C}$ and with a pump for circulating the water through the viscometer jacket or tank.

59.3 *Stop Clock or Watch*, calibrated in tenths of a second.

60. Procedure

60.1 *Sample Preparation*—Dry about 0.26 g of sample in a weighing bottle at $105 \pm 3^\circ\text{C}$ for 2 h, stopper, and cool in a desiccator. Weigh the bottle containing the sample to the nearest 0.001 g, transfer the sample to a 250-mL flask, and reweigh the bottle. Pipet into the flask 100 mL of solvent at $25 \pm 0.1^\circ\text{C}$. The solvent used should be mutually agreed upon by the purchaser and the supplier. Suitable solvents are listed in Table 1. After the sample is completely dissolved, place it in the constant-temperature bath at 25°C along with a portion of the solvent used, and allow sufficient time for both to come to temperature before making the viscosity measurements. During this conditioning period, water at 25°C should be circulating through the water jacket of the viscometer to allow ample time for the pipet to reach temperature equilibrium.

60.2 *Viscosity Measurements*—Rinse the reservoir and the outside of the capillary tube thoroughly with solvent. Rinse the inside of the capillary tube twice by alternately applying pressure at points B and A (Fig. 2). Discard the wash portion of the solvent. Pour more solvent into the reservoir and allow several minutes for complete drainage and thermal equilibrium to be obtained. Adjust the outer meniscus to a reference point, D, that will give a flow time between 70 and 100 s. Apply air pressure at B—to force the solvent up through the capillary past the upper timing mark, C, on the measuring bulb, E. Record the time in seconds required for the meniscus to fall between the timing marks, C and F. Take a minimum of two readings. Repeat these operations, substituting the solution for the solvent.

61. Calculation

61.1 Calculate the relative viscosity, η_{rel} , as follows:

$$\eta_{rel} = t_1/t_2 \quad (10)$$

where:

t_1 = flow time of solution, and

t_2 = flow time of solvent.

61.6 Calculate the intrinsic viscosity, $[\eta]$, as follows:

$$[\eta] = (k/c) [\text{antilog}(\log \eta_{rel}/k) - 1] \quad (11)$$

where:

k = values from Table 1, and
 c = concentration in grams per decilitre (Note 8 in Section 57).

62. Precision and Bias

62.1 No statement on bias can be made as no reference material is available as a standard.

VISCOSITY

63. Significance and Use

63.1 A measurement of viscosity is of great practical utility in determining the proper processing equipment and process concentrations for cellulose esters.

64. Procedure

64.1 *Solution*—Dry the sample for 1 to 2 h at $105 \pm 3^\circ\text{C}$ and cool in a desiccator. Prepare a solution of the dried sample in a solvent and at a concentration mutually agreed upon by the purchaser and the seller. Suitable solutions are listed in Table 2.

64.2 *Viscosity Determination*—Prepare the solution and measure the viscosity in accordance with Test Method D 1343, (Note 10 in Section 71 of these methods).

65. Report

65.1 Report the results in poises, unless otherwise specified. The viscosity values shall be prefixed with the letter A, B, C, etc., corresponding to the formula of the solution employed.

66. Precision and Bias

66.1 No statement on bias can be made as no reference material is available as a standard.

COLOR AND HAZE

67. Summary of Test Method

67.1 Color and haze determinations on cellulose ester solutions are made by comparison with standards. Simultaneous measurement of these properties is desirable because haze reduces the amount of color observed.

68. Significance and Use

68.1 Solution color and haze of a cellulose ester is a measurement of the optical properties of cellulose esters when dissolved in a specific solvent.

69. Apparatus

69.1 *Light Box*—A suitable light box (Figs. 3 and 4) is described as follows. The light source consists of a mercury vapor bulb,¹² which requires an auto transformer¹³ for the current source. The bulb is mounted horizontally across the lower front part of a plywood box 14 in. (350 mm) wide, 17 in. (430 mm) high, and 13 in. (330 mm) deep. This box is lined with a heat resistant board and is painted black inside,

except that the inside back surface toward the viewer is white. A bottle holder large enough to hold four bottles is built onto the front of the light box, and a 2½ by 6-in. (63.5 by 152.4-mm) horizontal viewing hole is cut through the front of the box. This opening is covered with clear glass, and a ¼-in. (6.4-mm) strip of black tape is fastened to the glass horizontally to aid in judging haze in the solution. Holes are cut in the bottom and top of the box for cooling by air convection. For continuous use, forced circulation of air would be desirable. A black metal baffle over the bulb prevents direct light on the viewing glass.

69.2 *Sample Bottles*—The bottles used for the sample solutions are French square bottles, 16-oz, with screw caps.¹⁴ These same bottles may be used for the color and haze standards.¹⁴

NOTE 9—These bottles may also be used for determination of viscosity, as described in 64.2.

69.3 *Cap Liners*—Cap liners shall be of a composition not affected by the solvents used. Liners of fiber board covered with cellophane or aluminum foil are usually satisfactory, but vinyl resin or waxed liners may cause interference with viscosity, color, or haze measurements.

70. Reference Standards

70.1 *Color Standards*—A color standard containing 500 ppm of platinum may be purchased¹⁵ or the solution may also be prepared as follows: Dissolve 1.245 g of potassium platinum chloride (K_2PtCl_6), containing 0.500 g of platinum, and 1.000 g of crystallized cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), containing 0.248 g of cobalt, in water, add 100 mL of HCl (sp gr 1.19), and dilute to 1 L with water. Prepare standards containing 50, 60, 70, 80, 90, 100, 125, 150, 175, 200, 250, 300, 350, 400 and 500 ppm of platinum by diluting suitable aliquots of the standard solution to 500 mL with water. Place these standards in the special bottles (see 69.2), taking care to select bottles with good clarity and free of flaws. Label and cap tightly.

70.2 *Haze Standards*—Prepare haze standards by diluting a stock solution having a turbidity of 1000 ppm. Prepare bottles containing 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 125, 150, 175, 200, and 400 ppm of turbidity, label, and cap tightly.

NOTE 10—The previously recommended stock solution for preparing these standards was made from fuller's earth, water, and hydrochloric acid. This solution is no longer available. A comparable stock solution can be made using a diatomaceous earth.¹⁶ To obtain haze levels equivalent to the fuller's earth standard, 1.1 parts diatomaceous should be used in place of 1.0 parts of fuller's earth in preparing the aqueous suspension. No hydrochloric acid is needed.

71. Procedure

71.1 Prepare the solution to be graded by dissolving the cellulose ester in the specified amount and kind of solvent, in

¹² A General Electric mercury vapor bulb Type H-250, A5-1, or A37-5 (differing only in the shape of the glass globe) has been found satisfactory for this purpose.

¹³ A General Electric Autotransformer Model 9T64Y21 has been found satisfactory for this purpose.

¹⁴ Owens-Illinois French square bottles No. A6732 with No. 48-400 caps have been found satisfactory for the sample solutions, viscosity, and for the color and haze standards, although for the color and haze standards, No. A6720 bottles with No. 28-400 caps are more convenient.

¹⁵ This color standard may be purchased as platinum cobalt chloride (APHA color standard) from the Hartman-Leddon Co., Philadelphia, Pa.

¹⁶ Celite® available from Johns-Manville Filtration and Mineral Division, Ken-Caryl Ranch, Denver, CO. 80217 has been found suitable for this purpose.

of the square bottles. See Table 2 for suitable solutions. At least 350 mL are required. Tumble until a uniform solution is obtained. Allow the solution to stand until it is free of bubbles before grading it for color and haze:

2 Place the bottle containing the solution to be graded in front of the shelf on the apparatus and place a similar bottle containing water behind it. Place the freshly shaken color standard at the front of the shelf beside the bottle containing the solution to be tested and place the color standard behind it. Determine the amount of color and haze in the solution by changing the color and haze standards until as good a match as possible has been obtained. The haze standards settle out quickly so they must be reshaken at short intervals. Report results in parts per million for both color and haze.

NOTE 11—When viscosity, color, and haze determinations, and an observation of general appearance are to be made on a cellulose ester

sample, a considerable saving in time can be made by using one solution in a square bottle for all three determinations. Dry the cellulose ester as required for the viscosity determination, prepare the solution carefully, and allow the bottle to stand long enough to form a thick solution before tumbling, to avoid solvent loss around the cap. Use a large enough sample to provide at least 350 mL of solution in the bottle. Measure the viscosity as described in Test Method D 1343, and then rate the color and haze.

72. Precision and Bias

72.1 No statement on bias can be made as no reference material is available as a standard.

73. Keywords

73.1 apparent acetyl; ash; cellulose acetate; cellulose ester; color; free acidity; haze; hydroxyl; intrinsic viscosity; sulfate content

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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 Mercuric Oxide for Use in Antifouling Paints¹

This standard is issued under the fixed designation D 911; the number immediately following the designation indicates the year of original adoption 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 mercuric oxide for use in antifouling paints.

2. Referenced Documents

2.1 ASTM Standards:

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 284 Test Methods for Chemical Analysis of Mercuric Oxide Pigment³

3. Composition

3.1 The pigment shall consist of finely divided red or yellow mercuric oxide and shall conform to the following

requirements:

Chemically combined mercury, calculated as metallic mercury, min, %	91
Free mercury	0.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve, max, %	10

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods:

5.2 *Chemically Combined and Free Mercury*—Test Methods D 284.

5.3 *Coarse Particles*—Test Methods D 185.

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 D01.31 on Pigment Specifications.

Current edition approved Aug. 19, 1987. Published October 1987. Originally published as D 911 - 47 T. Last previous edition D 911 - 86.

² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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Standard Specification for Cuprous Oxide for Use in Antifouling Paints¹

This standard is issued under the fixed designation D 912; the number immediately following the designation indicates the year of original adoption 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.

Scope

1.1 This specification covers cuprous oxide for use in antifouling paints.

Referenced Documents

- 2.1 *ASTM Standards:*
D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
D 283 Test Methods for Chemical Analysis of Cuprous Oxide and Copper Pigments³

Composition

3.1 The pigment shall consist essentially of finely divided cuprous oxide and shall conform to the following requirements:

Cuprous oxide, min, %	97
Total copper, calculated as Cu, min, %	86
Total reducing power as Cu ₂ O, min, %	97
Metals other than copper, max, %	0.5
Combined chlorides, calculated as Cl, and sulfates, calculated as SO ₄ , max, %	0.5

Acetone-soluble matter, max, %	0.5
Stability: decrease in total reducing power after stability test, max, %	2.0
Coarse particles (total residue retained on a No. 325 (45- μ m) sieve), max, %	0.5
Total nitric acid insoluble residue on a No. 200 (75- μ m) sieve, max, %	0.1

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Coarse Particles*—Test Methods D 185.

5.3 *Cuprous Oxide Content, Total Copper, Reducing Power, Other Metals, Chlorides, Sulfates, Acetone Soluble Matter, and Stability*—Methods D 283.

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 D01.31 on Pigment Specifications.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 912 - 47 T. Last previous edition D 912 - 65 (1975).

² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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Standard Test Methods for Ethylcellulose¹

This standard is issued under the fixed designation D 914; the number immediately following the designation indicates the year of original adoption 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.

NOTE—Editorial changes were made throughout, including the title, in October 1989.

1. Scope

- 1.1 These test methods cover the testing of ethylcellulose.
- 1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 6
Sulfated Ash	7 to 11
Chlorides (as Sodium Chloride)	12 to 16
Alkalinity (as free Sodium Hydroxide)	17 to 19
Ethoxyl Content	20 to 24
Viscosity	25 to 39

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 362 Specification for Industrial Grade Toluene²
 - D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers³
 - D 841 Specification for Nitration Grade Toluene²
 - E 1 Specification for ASTM Thermometers⁴

3. Purity of Reagents and Materials

3.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all of the reagents used shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Where such specifications have not been established, reagents of the best grade available shall be used. References to water shall be understood to mean distilled water.

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 D01.36 on Cellulosics.

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

⁵ "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."

MOISTURE

4. Procedure

4.1 Weigh accurately 2 to 5 g of the sample to the nearest 0.001 g into a tared dish (fitted with a lid) and dry for 2 h in an oven at 100 to 105°C. Remove the dish from the oven, cover with a lid, cool in a desiccator, and weigh.

5. Calculation

5.1 Calculate the percent moisture, M as follows:

$$M = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, g, and
 B = sample used, g.

6. Precision

6.1 Replicates within a given laboratory should agree within $\pm 5\%$ at the 95 % confidence level.

SULFATED ASH

7. Apparatus

- 7.1 Muffle Furnace, maintained at $575 \pm 25^\circ\text{C}$.
- 7.2 Crucibles, either porcelain, Coors No. 1, or platinum.

8. Reagent

8.1 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

9. Procedure

9.1 Ignite a crucible for 10 to 15 min at $575 \pm 25^\circ\text{C}$, cool in a desiccator, and weigh to the nearest 0.001 g.

9.2 Weigh about 5 g of sample to the nearest 0.001 g (previously dried for 3 h at 105°C) into the crucible. Burn off the bulk of the carbonaceous material directly over a flame. After cooling, add 1 mL of H_2SO_4 in such a way as to moisten the entire ash; then cautiously heat with the burner to dense white fumes. Ignite in a muffle furnace at $575 \pm 25^\circ\text{C}$ until all signs of carbon are gone. Cool in a desiccator and reweigh to the nearest 0.001 g.

10. Calculation

10.1 Calculate the percent ash (as sulfate), C , as follows:

$$C = (A/B) \times 100 \quad (2)$$

ash; g, and
sample used, g.

Precision

11 Data obtained from the above procedure indicate laboratory results should agree within $\pm 10\%$ at the 95% confidence level.

CHLORIDES (AS SODIUM CHLORIDE)

Apparatus

12.1 Titration pH Meter.⁶

12.2 *Mercury-Mercurous Sulfate Reference Electrode*⁷—The electrode uses a potassium sulfate electrolyte to avoid chloride contamination from a chloride electrolyte.

12.3 *Silver-Silver Chloride Electrode*⁸—The electrode is coated with silver chloride periodically. Prepare the electrode by polishing with fine steel wool, briefly soaking it in 5% potassium cyanide solution, and rinsing it with water. Coat the electrode with silver chloride by electrodeposition from 0.1 *N* potassium chloride solution using a 3-V dry cell and a platinum wire electrode. Connect the silver electrode to the positive pole of the battery and electrolyze for 20 s; then reverse the connections for 5 s. Repeat these operations twice, and finally, chloridize the silver electrode for 20 s at the positive terminal. Store the silver electrode in 0.1 *N* potassium chloride solution. Rinse the electrode with water and wipe it with a soft tissue before each titration.

12.4 *Salt Bridge for Reference Electrode*—Figure 1 shows one configuration in use. Exact dimensions are not important. The salt bridge is used to keep the reference electrode from plugging with the ethylcellulose slurry.

12.5 Air-Driven Stirrer.

13. Reagents

13.1 *Ethanol* (95 volume %), undenatured or specially denatured conforming to Formula 2B of the U.S. Bureau of Internal Revenue.

13.2 *Ethanol-Distilled Water Solvent Mixture* (80 + 20)—Mix 800 g of 2B ethanol with 200 g of water. Add 7.5 g of Aerosol OT⁹ 100% surface-active agent per 3000 g of ethanol-water mixture.

13.3 *Potassium Nitrate* (KNO_3) Solution (saturated) for salt bridge (Fig. 1).

13.4 *Silver Nitrate, Standard Solution* (0.02 *N*)—Dissolve 3.4 g of silver nitrate (AgNO_3) in water, dilute to 1 L with water in a volumetric flask, and mix. Weigh exactly 0.5845 g of dry, primary standard sodium chloride (NaCl), dissolve in 25 mL of water, and dilute to 1 L with water in a volumetric flask. Add 10 mL of H_2SO_4 (1 + 16) to each aliquot before titrating. Titrate aliquots of this solution potentiometrically

⁶ MacBeth Model T or Leeds and Northrup pH meters have been found satisfactory for this purpose.

⁷ Beckman No. 40455 electrode, manufactured by Beckman Instruments, Inc., 2500 Harbour Blvd., Mail Station E 31D, Fullerton, CA 92634, has been found satisfactory for this purpose.

⁸ Beckman No. 39261 electrode, manufactured by Beckman Instruments, Inc., has been found satisfactory for this purpose.

⁹ Aerosol OT is available from the American Cyanamid Co., Linden, NJ.

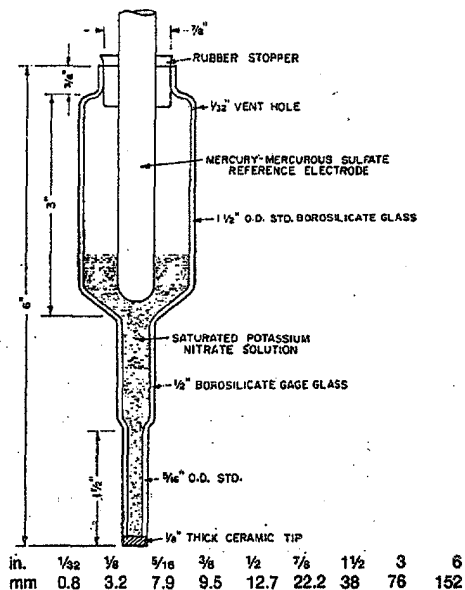


FIG. 1 Salt Bridge and Reference Electrode for Chloride Determination.

with the AgNO_3 solution. Calculate the normality, *N*, of the AgNO_3 solution as follows:

$$N = (A/B) \times 0.01 \quad (3)$$

where:

A = 0.01 *N* NaCl solution added, mL, and
B = AgNO_3 solution required for the titration, mL.

13.5 *Sulfuric Acid* (1 + 16)—Add 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) slowly with stirring into 16 volumes of water.

13.6 *Toluene*, meeting the requirements of Specification D 362.

13.7 *Toluene-Ethanol Solvent Mixture* (90 + 10)—Mix 900 g of toluene with 100 g of ethanol.

14. Procedure

14.1 Weigh accurately 10 g of sample to the nearest 0.001 g (previously dried for 2 h at 100 to 105°C) and transfer to a 600-mL beaker containing 200 mL of the toluene-ethanol solvent mixture. Stir with an air-driven stirrer until solution is complete.

14.2 Add 200 mL of the ethanol-water mixture and agitate for 5 min to form a uniform emulsion. Immerse the electrodes in the emulsion using an air-driven stirrer for mixing. Add 10 mL of H_2SO_4 (1 + 16) and agitate for 3 to 4 min to allow the system to reach equilibrium.

14.3 Titrate slowly with the 0.02 *N* AgNO_3 solution. Make intermittent additions of 0.1 mL. It is advisable to allow longer periods of time between additions of titrant as the end point is approached to avoid passing the equivalence point. Run a blank by the same procedure.

15. Calculation

15.1 Calculate parts per million of chlorides as NaCl , *C*, as follows:

$$C = [(VN \times 0.05845)/W] \times 1\,000\,000 \quad (4)$$

where:

V = AgNO_3 solution, mL,
 N = normality of AgNO_3 solution,
 W = sample used, g, and
 0.05845 = milliequivalent mass of NaCl.

16. Precision

16.1 Data obtained from the above procedure indicate interlaboratory results should agree within $\pm 5.0\%$ at the 95 % confidence level.

ALKALINITY (AS FREE SODIUM HYDROXIDE)

17. Reagents

17.1 *Hydrochloric Acid, Standard (0.1 N)*—Prepare and standardize a 0.1 N solution of hydrochloric acid (HCl).

17.2 *Phenolphthalein Indicator Solution (10 g/L)*—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %).

17.3 *Strontium Chloride Solution (100 g/L)*—Dissolve 100 g of strontium chloride (SrCl_2) in water and dilute to 1 L.

18. Procedure

18.1 Weigh about 10 g of the sample to the nearest 0.001 g (previously dried for 2 h at 100 to 105°C) into a 500-mL Erlenmeyer flask. Add 150 mL of water and heat at the boiling point for 5 min. Add, while stirring, 5 mL of SrCl_2 solution and allow the solution to cool.

18.2 Add 2 drops of phenolphthalein indicator solution and titrate to the first faint pink color with 0.1 N HCl.

19. Calculation

19.1 Calculate the percent of alkalinity, D , as NaOH as follows:

$$D = [(AB \times 0.040)/C] \times 100 \quad (5)$$

where:

A = HCl required for titration of the sample, mL,
 B = normality of the HCl; and
 C = sample used, g.

ETHOXYL CONTENT

20. Apparatus

20.1 *Distillation Apparatus*, as illustrated in Fig. 2, consisting of a boiling flask with a side arm for admission of carbon dioxide (CO_2) or nitrogen, an air condenser with a trap, and a receiver.

20.2 *Oil Bath*, equipped with a heating device, preferably electrical, so that the bath can be maintained at 145 to 150°C.

21. Reagents

21.1 *Bromine Solution*—Dissolve 5 mL of bromine in 145 mL of the potassium acetate ($\text{KC}_2\text{H}_3\text{O}_2$) solution. Prepare the bromine solution fresh daily in a hood to remove bromine vapors.

21.2 *Carbon Dioxide*—Pass the CO_2 through a bubble counter and a dry trap, and then through a pressure regulator consisting of a glass tee whose vertical arm extends almost to

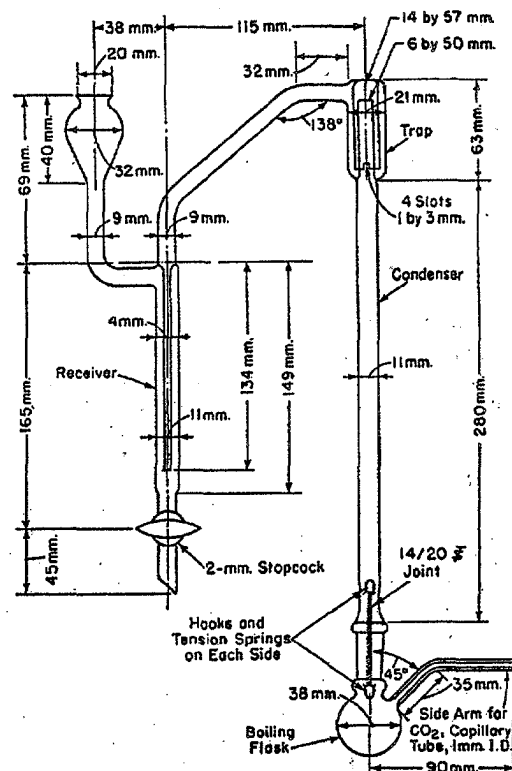


FIG. 2 Distillation Apparatus for Ethoxyl Determination

the bottom of a 10-in. (254-mm) column of water. A screw clamp shall be attached to the thin-walled rubber tubing connecting the horizontal arm of the tee with the boiling flask. This arrangement permits regulation of the flow of gas and allows any excess gas to escape. Nitrogen may be used in place of CO_2 .

21.3 *Formic Acid (90 %)*.

21.4 *Gelatin Capsules*—Gelatin capsules of a suitable size¹⁰ to hold from 50 to 60 mg of the dried sample will be required.

21.5 *Hydriodic Acid (sp gr 1.70)*^{11,12}—Hydriodic acid (HI) forms with water a constant-boiling mixture (boiling point 126 to 127°C) that contains 57 % HI. The concentration of HI in the reagent used should be not less than 56.5 %. The blank determination, which is affected primarily by free iodine in the reagent, should require no more than 0.5 mL of 0.1 N sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) standard solution. If necessary, the acid may be purified by adding to it a small amount of red phosphorus and boiling for 20 to 30 min in a hood while passing a stream of CO_2 into the liquid. Distillation is then carried out behind a safety-glass shield in

¹⁰ Size 0 gelatin capsules available from Parke, Davis and Co. are satisfactory for this purpose.

¹¹ Hydriodic acid, available from Merck and Co., WBC 220, P.O. Box 2000, Rahway, NJ 07065, under the designation "For Methoxyl Determination" has been found satisfactory for ethoxyl determination.

¹² Hydriodic acid suitable for ethoxyl determination may also be prepared by the method of Samsel, E. P., and McHard, J. A., *Industrial and Engineering Chemistry, Analytical Edition*, Vol 14, 1942, p. 750.

hood, using an all-glass apparatus with a slow stream of CO_2 running through the receiver. Under some conditions, the poisonous gas phosphine (PH_3) is formed during distillation, and this may unite with molecular iodine to form phosphorus triiodide (PI_3) which may explode on contact with air. It is, therefore, advisable to keep the current of CO_2 going after the distillation is ended and until the apparatus has cooled; this will prevent air from being sucked into the apparatus. Put the purified HI in small, brown, glass-stoppered bottles, previously swept out with CO_2 , and seal the stoppers with molten paraffin. Store in a dark place. To minimize decomposition of HI due to contact with air, run CO_2 into the bottle while withdrawing portions of the acid for use.

21.6 *Phosphorus Slurry* (0.06 g/100 mL)—Add about 0.06 g of red phosphorus to 100 mL of water. Shake well before using.

21.7 *Potassium Acetate Solution* (100 g/L)—Dissolve 100 g of anhydrous potassium acetate ($\text{KC}_2\text{H}_3\text{O}_2$) crystal in 1 L of a solution containing 900 mL of glacial acetic acid and 100 mL of acetic anhydride.

21.8 *Potassium Iodide* (KI).

21.9 *Sodium Acetate Solution* (220 g/L)—Dissolve 220 g of anhydrous sodium acetate in water and dilute to 1 L.

21.10 *Sodium Thiosulfate, Standard Solution* (0.1 N)—Dissolve 25 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 200 mL of water and dilute to 1 L. Use freshly boiled and cooled water. It is preferable to allow the solution to stand for a few days before standardization. Standardize the solution against 0.1000 N potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) solution prepared by dissolving exactly 4.9037 g of $\text{K}_2\text{Cr}_2\text{O}_7$ (National Institute of Standards and Technology Standard Sample No. 136) in water and diluting to 1 L in a volumetric flask. By means of a buret, measure accurately 35 to 45 mL of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution into a 250-mL Erlenmeyer flask. Add 2 g of KI and 50 mL of sulfuric acid (H_2SO_4 , 1 + 9) and allow to stand for about 5 min. Titrate the liberated iodine with the $\text{Na}_2\text{S}_2\text{O}_3$ solution, using starch indicator solution near the end point. At the end point, the blue color of the starch indicator will be destroyed, leaving the pale green color of the chromate ion. The normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution should be checked at least once a week. Calculate the normality, N , of the $\text{Na}_2\text{S}_2\text{O}_3$ solution as follows:

$$N = (A/B) \times 0.1 \quad (6)$$

where:

A = 0.1000 N $\text{K}_2\text{Cr}_2\text{O}_7$ solution added, mL, and

B = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for the titration, mL.

As an alternative procedure, the $\text{Na}_2\text{S}_2\text{O}_3$ solution may be standardized against 0.1 N iodine that has been standardized in turn against arsenic trioxide (As_2O_3) (National Institute of Standards and Technology Standard Sample No. 83) or potassium iodate (KIO_3).

21.11 *Starch Indicator Solution*.

21.12 *Sulfuric Acid* (1 + 9)—slowly add with stirring 1 volume of concentrated H_2SO_4 (sp gr 1.84) to 9 volumes of water.

22. Procedure

22.1 Dry the sample at 105°C for at least 30 min. Through the condenser, add to the trap in the distillation apparatus

(Fig. 2) enough of the phosphorus slurry to make the trap about half full. Add 19 to 20 mL of the bromine solution to the receiver. Accurately weigh from 50 to 60 mg of the dry sample into a gelatin capsule and drop it into the boiling flask. (The weighing should be done as rapidly as possible without sacrificing accuracy because dry ethyl-cellulose picks up moisture rapidly.)

22.2 Add a few small glass beads or chips of clay plate and then 6 mL of HI. Attach the boiling flask at once to the condenser, using a few drops of HI to moisten the ground-glass joint, and then connect the side arm of the flask to the source of CO_2 . Pass a current of CO_2 into the apparatus at the rate of about 2 bubbles per second. Immerse the flask in the oil bath, maintained at 150°C, and heat for 40 min.

22.3 Add 10 mL of sodium acetate solution to a 500-mL Erlenmeyer flask and wash into it the contents of the receiver; dilute to 125 mL with water. Add formic acid dropwise, with swirling, until the brown color of bromine is discharged, and then add about 6 drops more. A total of 12 to 15 drops is usually required. After about 3 min, add 3 g of KI and 15 mL of H_2SO_4 (1 + 9) and titrate immediately with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution to a light straw color. Add a little starch solution and continue the titration to the disappearance of the blue color.

22.4 *Blank*—Make a blank determination, using the same amounts of reagents and the same procedures as for the specimen. (Usually, about 0.1 mL of 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution is required.)

23. Calculation

23.1 Calculate the percent ethoxyl, E , as follows:

$$E = [(A - B)N \times 0.00751]/W \times 100 \quad (7)$$

where:

A = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the sample, mL,

B = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the blank, mL,

N = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, and

W = sample used, g.

24. Precision

24.1 Replicates within a given laboratory should agree within ± 1.0 % at the 95 % confidence level.

VISCOSITY

25. Scope

25.1 Ethylcellulose viscosity is arbitrarily measured on a 5 % solution in a specific solvent system. The viscosity normally ranges from 7 to 200 cP at 25°C.

25.2 Two test methods for measuring ethylcellulose viscosity are given below. One test method specifies use of the Hercules Horizontal Capillary Viscometer and a solvent system of 80 + 20 toluene-ethanol while the other specifies the Dow Modified Ubbelohde viscometer and solvent systems of 80 + 20 toluene-ethanol for product with an ethoxyl content above 47 % and 60 + 40 toluene-ethanol for ethoxyl content below 47 %.

25.3 The two test methods given below do not give the same resultant viscosity. Therefore, they should be used only in a relative sense and not interchangeably.

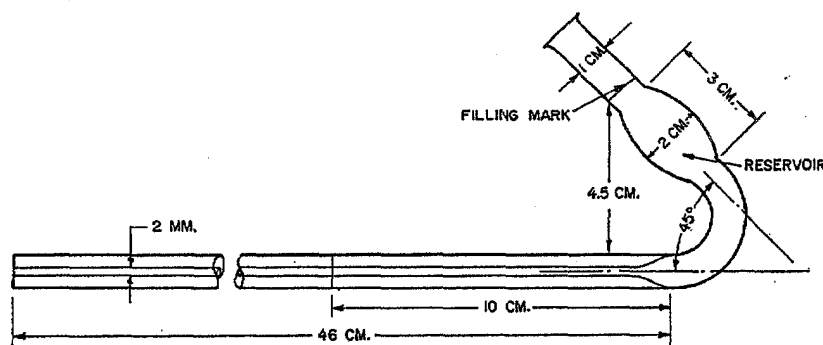


FIG. 3 Hercules Horizontal Capillary Viscometer

25.4 Neither test method is intended to be a referee method.

HERCULE HORIZONTAL CAPILLARY VISCOSITY

26. Apparatus

26.1 *Hercules Horizontal Capillary Viscometer*¹³ (Fig. 3)—The tube is surrounded by a glass tube that acts as a water jacket and is connected with it by rubber stoppers at both ends which have proper holes to provide for water inlet and outlet and for insertion of a thermometer. For convenient use, it is desirable to cut the openings in the stopper at the end next to the reservoir bulb. One of the thermostated water lines should extend inside the jacket, nearly to the opposite end, to provide good circulation. The tube and its jacket are attached to a wood or metal baseboard large enough to support its entire length. The board is in turn hinged at one end to a larger board and is provided with a movable-support arm to hold it at a 45° angle with the base. The bottom board should have adjustable legs so that it can be leveled. The board that supports the capillary tube is equipped with a spirit level. Care must be taken in mounting the tube to see that the capillary is exactly parallel to the board holding the spirit level. The water running through the jacket should come from a constant-temperature bath maintained at 25.0 ± 0.1°C.

26.2 *Shaker, Bottle*.¹⁴

26.3 *Timer*, calculated in 0.1-s units.

27. Reagents

27.1 *Standard Calibrating Liquid*.¹⁵ 100 cP and 25 C.

27.2 *Ethanol* (95 volume %)—Undenatured or specially denatured conforming to Formula 2B of the U.S. Bureau of Internal Revenue.

27.3 *Mixed Solvent* (80 + 20 toluene ethanol by mass)—Prepare by weighing proportional amounts of toluene and ethanol described in 27.1 and 27.2. Mix thoroughly.

27.4 *Toluene*, meeting the requirements of Specification D 362.

¹³ Detailed drawings of the complete installation can be obtained from Hercules Incorporated, Wilmington, DE as Drawings No. 2173U and 2174U.

¹⁴ Size No. 2, International Equipment Co., Boston, MA or equivalent.

¹⁵ Viscometer calibrating oil S-60, available from Cannon Instrument Co., P. O. Box 16, State College, PA 16801, has been found satisfactory for this method.

28. Calibration

28.1 Make a mark on the capillary tube about 10 cm from the point where the capillary is attached to the reservoir. Mount the tube in a water jacket, adjust the temperature, elevate the board holding the tube to the 45° position, and fill the reservoir to the etched mark with the standard liquid. Lower the board holding the tube to the baseboard and level the instrument while holding one finger over the end of the capillary tube. Remove the finger when ready and the liquid will start to flow through the capillary. Start the timer when the liquid reaches the etched mark. Determine the place where the second mark is to be placed by noting the distance the liquid has traveled when the elapsed time, in seconds, is equal to the viscosity of the oil in centipoises. It is advisable to use a movable mark, such as a small-rubber band, until the exact position of the mark has been rechecked quite carefully; then an etched line can be made.

29. Procedure

29.1 Dry a portion of the sample at 100 to 105°C for 1 h. Weigh 5 g of the sample to the nearest 0.01 g into an 8-oz (250-mL) widemouth bottle. Add 95.00 g of the mixed solvent, cover the neck of the bottle with a sheet of cellophane, and screw on the cap.

29.2 *Agitate* on a bottle shaker until the solution is complete by visual inspection. After the solution is complete, place the bottle of the solution in a constant-temperature bath maintained at 25 ± 0.1°C for 1 h.

29.3 Remove the bottle of solution from the bath and check to be sure it is free of air bubbles. Fill the viscometer reservoir to the etched mark, while vertical, with the solution to be tested. Place a finger over the end of the capillary, release the brace, lower the tube, and level.

29.4 Release the finger and determine the time for the liquid to flow from the first mark to the second.

30. Calculation

30.1 Calculate the viscosity as follows:

$$N = td/D \quad (8)$$

where:

N = viscosity, cP,

t = time of flow for the sample, s,

d = density of the sample solution at 25°C (0.859), and

D = density of the oil used for calibration of the viscometer.

TABLE 1. Solutions for Viscosity Determination

Dow Viscometer Designation	Capillary Bore Diameter, mm	Approximate Viscosity, cP at 25 C	Approximate D 446 Viscon Ubbelohde Viscometers	Cannon Standard Viscometer Calibrating Liquid
P	1.10	...	2	S-60
Q	1.49	10	2C	S-200
R	1.77	20	2B	S-200
S	2.51	80	3C	S-600
T	3.15	150	3B	S-600
U	4.40	...	4X	S-8000

31. Report

31.1 Report the viscosity in centipoises, the solution concentration, and the test method (Hercules Horizontal Capillary).

32. Precision

32.1 Duplicate determinations should agree within 5 % of the value obtained.

DOW MODIFIED UBBELOHDE VISCOSITY

33. Apparatus

33.1 *Constant-Temperature Bath*, capable of maintaining a temperature of $25 \pm 0.1^\circ\text{C}$.

33.2 Shaker, Bottle.¹⁴

33.3 *Timer*, calibrated in 0.1-s units.

33.4 *Viscometers, modified Ubbelohde (Fig. 4).*

33.5 **Thermometer**—ASTM Kinematic Viscosity Thermometer, having a range from 74.5 to 79.5 F and conforming to the requirements for Thermometer 45F as prescribed in Specification E 1.

34. Reagents

34.1 *Ethanol* (95 volume %), undenatured or specially denatured conforming to Formula 2B of the U.S. Bureau of Internal Revenue.

34.2 *Toluene*, meeting the requirements of Specification D 841.

34.3 *Standard Calibrating Liquids*¹⁵ (see 35.2).

34.4 *Mixed Solvents* (80 + 20 and 60 + 40 toluene-ethanol by mass)—Mix proportional amounts of toluene and ethyl alcohol described in 34.1 and 34.2. Mix thoroughly.

35. Calibration

35.1 Select viscosity-calibrating liquids that will yield a minimum efflux time of 200 s in the viscometers to be standardized at $25 \pm 0.1^\circ\text{C}$. See Specification D 476.

35.2 Calibrate one viscometer of each capillary size to be used in the viscosity test work using the calibrating liquid that is indicated in Table 1.

35.3 Charge the Ubbelohde viscometer selected with the appropriate viscometer calibrating liquid by pouring the liquid into the large tube to a level in line with the bottom of

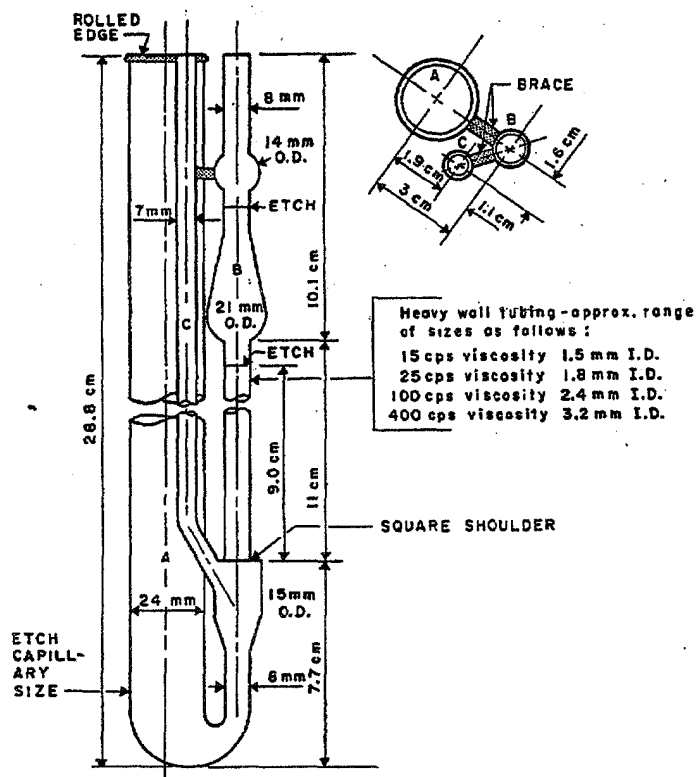


FIG. 4 Dow Modified Ubbelohde Viscometer

the vent-tube entrance. Charge the viscometer in such a manner so that the U-tube at the bottom fills completely without trapping air.

35.4 Place the viscometer in a constant-temperature water bath at $25 \pm 0.1^\circ\text{C}$, immersing the viscometer to cover the functioning areas. Allow the liquid to reach the required temperature. Determine the temperature by placing the thermometer into the liquid. Remove the thermometer. Place a suction bulb over the end of the calibrated flow tube and your finger over the end of the vent tube; then apply suction on the calibrated tube until the liquid level is drawn half way up into the upper bulb of the calibrated tube. Release the suction and finger from the viscometer.

35.5 Time the flow in seconds between the upper and lower calibration lines to the nearest 0.10 s with a timer. If the time in seconds is less than 200, select a viscometer with a smaller capillary and repeat the operation.

35.6 Without recharging the viscometer, make check determinations by repeating the procedures five times. Determine the efflux time by averaging the five values. The range of the individual values should not exceed 0.5 s.

35.7 Calculate the standard viscometer constant, F , for ethylcellulose solutions as follows:

$$F = (V \times d_e)/(d_0/t) \quad (9)$$

where:

V = absolute viscosity of the calibrating liquid at 25°C ,
 d_0 = density of the calibrating liquid at 25°C ,
 d_e = density of the sample solution at 25°C (use 0.861 for 80 + 20 and 0.845 for 60 + 40 toluene-ethanol), and
 t = flow time, s.

36. Procedure

36.1 Weigh 57 ± 0.02 g of the solvent solution into an 8-oz (250-mL) jar. Weigh 3.0 ± 0.01 g of dry sample and add to the solvent in the jar. Close the jar with a lined screw cap and shake to wet and disperse the sample. Continue to shake

for about 15 min on the shaker, or until the sample is completely dissolved.

36.2 Select a Ubbelohde viscometer based on the approximate viscosity range (see 35.2). Add sample solution to the large tube to a level in line with the bottom of the vent tube entrance.

36.3 Place the viscometer in the $25 \pm 0.1^\circ\text{C}$ constant-temperature bath. (Immerse the viscometer to cover the functioning areas.)

36.4 Place a thermometer in the viscometer (see 35.4). After the liquid has reached the required temperature, remove the thermometer. Charge the viscometer by placing a suction bulb over the end of the calibrated flow tube and then, with a finger over the opening of the vent tube, apply suction on the calibrated tube until the liquid level is drawn half-way up into the upper half of the calibrated tube. Release suction and finger from the tubes.

36.5 Time the flow between the upper and lower calibration lines to the nearest 0.1 s.

36.6 Repeat the above procedure and average the two efflux times. If the time range exceeds 1 s, repeat the operation a third time and average the two values that deviate the least.

37. Calculation

37.1 Calculate viscosity, V_1 in centipoises as follows:

$$V_1 = F \times T \quad (10)$$

where:

F = viscometer constant (see 35.7); and
 T = efflux time, s, from 36.6.

38. Report

38.1 Report viscosity in centipoises, solution concentration, and the test method (Dow Modified Ubbelohde).

39. Precision

39.1 Data obtained by this procedure indicate a precision of $\pm 5\%$.

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Standard Specification for Aluminum Powder and Paste Pigments for Paints¹

This standard is issued under the fixed designation D 962; the number immediately following the designation indicates the year of original adoption 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.

^ε Note—The title of this specification was editorially changed in April 1986.

Scope

1.1 This specification covers four types and three classes of aluminum pigments for use in paints.

Referenced Documents

2.1 ASTM Standards:

- D 480 Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes²
- E 34 Test Methods for Chemical Analysis of Aluminum and Aluminum Base Alloys³

3. Classification

3.1 Type I—Leafing Aluminum Pigment Powder:

3.1.1 *Class A, Fine*—Maximum of 0.1 % retained on a No. 325 (45-μm) sieve.

3.1.2 *Class B, Medium*—Maximum of 1.5 % retained on a No. 325 (45-μm) sieve.

3.1.3 *Class C, Coarse*—Maximum of 20 % retained on a No. 325 (45-μm) sieve.

3.2 Type II—Leafing Aluminum Pigment Paste:

3.2.1 *Class A, Fine*—Maximum of 0.1 % retained on a No. 325 (45-μm) sieve.

3.2.2 *Class B, Medium*—Maximum of 1.0 % retained on a No. 325 (45-μm) sieve.

3.2.3 *Class C, Coarse*—Maximum of 15 % retained on a No. 325 (45-μm) sieve.

3.3 Type III—Nonleafing Aluminum Pigment Powder (Note):

3.3.1 *Class A, Fine*—Maximum of 1.5 % retained on a No. 325 (45-μm) sieve.

3.3.2 *Class B, Medium*—Maximum of 6.0 % retained on a No. 325 (45-μm) sieve.

3.3.3 *Class C, Coarse*—Maximum of 20 % retained on a No. 325 (45-μm) sieve.

3.4 Type IV—Nonleafing Aluminum Pigment Paste (Note):

3.4.1 *Class A, Fine*—Maximum of 0.1 % retained on a No. 325 (45-μm) sieve.

3.4.2 *Class B, Medium*—Maximum of 1.0 % retained on a No. 325 (45-μm) sieve.

3.4.3 *Class C, Coarse*—Maximum of 11.0 % retained on a No. 325 (45-μm) sieve.

NOTE—The particle size characteristics of the Type 3 and Type 4 pigments of the same class do not correspond with each other; in the case of Types 1 and 2 they do.

4. Composition and Properties

4.1 *Aluminum Pigment Powder (Type 1, Leafing and Type 3, Nonleafing)*—The aluminum pigment powder shall consist of commercially pure aluminum in the form of fine, polished flakes, and a suitable fatty lubricant. It shall contain no filler or extender pigments.

4.2 *Aluminum Pigment Paste (Type 2, Leafing and Type 4, Nonleafing)*—The aluminum pigment paste shall consist of commercially pure aluminum in the form of fine, polished flakes, and a suitable fatty lubricant combined with a volatile thinner. It shall contain no filler or extender pigments.

4.3 The aluminum pigments, powder, and paste of both leafing and nonleafing types shall conform to the requirements given in Table 1.

4.4 The paint made from the powder or paste shall be free-flowing and shall give a continuous coating at least equal in smoothness, luster, and general appearance to that of a reference sample mutually agreed upon by the purchaser and the seller.

4.5 The aluminum pigments, powder and paste of both leafing and nonleafing types, stored in unopened original packages within 6 months after shipment shall pass all tests applicable to the respective type and class as described in Test Methods D 480.

5. Test Methods and Sampling

5.1 Sampling and tests shall be conducted in accordance with Test Methods D 480. Since several of the methods are empirical and the results are affected by the method, the specified procedures should be closely followed.

6. Packaging

6.1 Aluminum paste should be packaged in a polyvent container that can be safely vented in case of pressure build-up.

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 D01.31 on Pigment Specifications.

Current edition approved Oct. 30, 1981. Published December 1981. Originally published as D 962 - 48 T. Last previous edition D 962 - 66 (1973).

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

³ *Annual Book of ASTM Standards*, Vol 03.05.

TABLE 1 Requirements for Aluminum Pigments

	Leafing		Nonleafing	
	Type 1 (Powder)	Type 2 (Paste)	Type 3 (Powder)	Type 4 (Paste)
Nonvolatile matter at 105 to 110°C, min, %	99	65	99	65
Easily extracted fatty and oily matter (polishing lubricant), max, % ^A	4.0	3.0	4.0	3.0
Total impurities other than fatty and oily matter, max, % ^A	1.0	0.7	1.0	1.25
Coarse particles, max, %:				
Class A (total residue retained on a No. 325 (45-μm) sieve)	0.1	0.1	1.5	0.1
Class B (total residue retained on a No. 325 (45-μm) sieve)	1.5	1.0	6.0	1.0
Class C (total residue retained on:				
No. 100 (150-μm) sieve	0.5	0.5	0.5	0.5
No. 325 (45-μm) sieve)	20.0	15.0	20.0	11.0
Leafing, min, %:				
Class A	50	55	absent	absent
Class B	50	50	absent	absent
Class C	50	50	absent	absent

^A Nonleafing pigments may contain metal compounds as dispersants. If required, a complete analysis may be made in accordance with Test Method E 34.

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Standard Specification for Copper Phthalocyanine Blue Pigment¹

This standard is issued under the fixed designation D 963; the number immediately following the designation indicates the year of original adoption 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 April 1986.

Scope

1.1 This specification covers copper phthalocyanine blue pigment to be purchased in dry powder form for use in paints, printing ink, and related products. Several types are available, intended for different end uses. The specific end use will determine which of the tests in Section 5 are applicable.

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 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out²
- D 362 Specification for Industrial Grade Toluene³
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 476 Specification for Titanium Dioxide Pigments²
- D 600 Specification for Liquid Paint Driers³
- D 1135 Test Methods for Chemical Analysis of Blue Pigments²
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 *Pigment*—The pigment shall consist of the product known commercially as copper phthalocyanine blue, with or without other ingredients incorporated during manufacture to improve or alter the working properties of the pigment, but free of any other coloring matter, either organic or inorganic. The pigment shall conform to the following requirements:

Moisture and other volatile matter, max, %
Other coloring matter

3.0
none

3.2 *Mass Color and Character of Tint*—The mass color and character of the tint formed by mixture with a white pigment shall be the same as, and the strength not less than, that of a reference sample mutually agreed upon by the purchaser and the seller.

3.3 *Oil Absorption*—The oil absorption shall be between 90 and 110 % of that of a reference sample mutually agreed upon by the purchaser and the seller.

3.4 *Reaction in Identification Tests*—The pigment shall show the same reaction in identification tests (see 5.5 through 5.9) as a reference sample mutually agreed upon by the purchaser and the seller.

3.5 *End Product Dispersion Stability*—When specified, end product dispersion stability, determined as described in 5.10 or by using a mutually agreed upon test, shall be the same as or better than that of a reference pigment sample mutually agreed upon by the purchaser and the seller.

NOTE 1—End product dispersion stability of a phthalocyanine pigment is frequently referred to as flocculation-resistance. It is judged as the difference in depth of tint between a sprayed and a poured or brushed coating. The behavior of the pigment can be markedly affected by the formulation used, the type of vehicle, thinner, etc. It is important that the purchaser and seller define the type of composition to be used in carrying out the test if the one described in 5.10 is not acceptable.

3.6 *End Product Storage Stability*—When specified, end product storage stability, determined as described in 5.11, shall be the same as or better than that of a reference pigment sample mutually agreed upon by the purchaser and the seller.

NOTE 2—Storage stability of a phthalocyanine pigment is frequently referred to as crystal stability, since it may be a measure of the tendency of the phthalocyanine to grow in particle size, that is, to grow crystals and thus lose strength. It is also related to a change in dispersion stability as defined in Note 1.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 1000 lb (500 kg), except that for shipments of less than 1000 lb, two samples shall be taken. At the option of the purchaser, each sample may be tested or samples from the same production may be blended in equal quantities to form a composite sample.

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 D01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vol 06.03.

TABLE 1 Ingredients for Dispersion Stability Test

Ingredient	Amount	Specification
Porcelain balls, 1/2-in. (12.7-mm) dia	1350 g	...
Toluene (industrial grade)	380 mL	D 362 ^A
Titanium dioxide (rutile type)	750 g	D 476 ^B
Soya-modified phthalic alkyd solution, ⁴ 50 % solids	275 g	...

^A Specification D 362.^B Specification D 476.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.2 *Moisture and Other Volatile Matter*—Method A of Test Methods D 280 or D 1208.

5.3 *Mass Color and Tinting Strength*—Test Method D 387.

5.4 *Oil Absorption*—Test Method D 281.

5.5 *Identification*—Procedure section (of Identification) of Test Methods D 1135.

5.6 *Basic Dye Derivatives*—Test Methods D 1135.

5.7 *Other Organic Coloring Matter*—Test Methods D 1135.

5.8 *Ultramarine Blue*—Test Methods D 1135.

5.9 *Iron Blue*—Test Methods D 1135.

5.10 *End Product Dispersion Stability*—The dispersion stability shall be determined with a paint, enamel, or lacquer agreed upon, using the pigment sample under test and a reference pigment sample. Unless the purchaser and seller agree otherwise, the following procedure shall be used. It has the advantage of combining the dispersion stability and storage stability (see 5.11) tests.

5.10.1 *Tinting White*—Charge a 1-gal (3.8-L) porcelain ball mill with the ingredients given in Table 1. Rotate the mill at 70 to 75 r/min for 24 h; then add 1900 g of alkyd resin solution,⁴ 90 mL of toluene, and 15 g of liquid drier containing lead and cobalt and conforming to Specification D 600. Rotate the mill for 1 h and pour the mixture through a paint strainer and store in tightly closed containers.

5.10.2 *Mill Base*—In an 8-oz flat, cream jar,⁵ charge 315 g of 1/8-in. (3.2-mm) diameter steel shot; 63 mL of toluene, 31 g of alkyl resin solution,⁴ and 12.5 g of copper phthalocyanine blue. Rotate for 24 h, at 60 to 80 r/min. Add 100 g of alkyd resin and 10 mL of toluene, and rotate for 30 min. Pour the mixture through a paint strainer and store in a tightly closed container until used.

5.10.3 *Tint*—Mix 32 g of mill base (see 5.10.2), 93 g of tinting white (see 5.10.1) and 40 mL of toluene. Shake vigorously for 30 min. Spray at once to good covering on

⁴ AROPLAZ 7310-X-50, available from Spencer Kellogg Div. of Cargill Inc., P.O. Box 807, Buffalo, NY 14240, a short oil alkyd soya type, is suitable for this purpose.

⁵ Cream jars available from Zuckerman Honickman, Inc., 36th and Reed Sts., Philadelphia, PA, have been found satisfactory for this purpose.

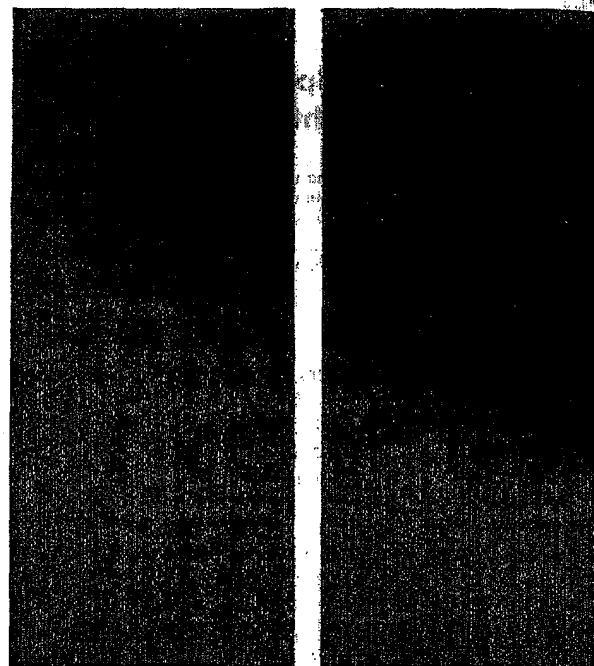


FIG. 1 Illustration of Relative Flocculation Tendency

glazed cardboard or primed metal panels.

5.10.4 *Procedure*—Allow the freshly sprayed panel to dry until the film becomes tacky (15 to 30 min); then with the finger gently rub a portion of the panel. This area will usually show an increase in tint depth over the sprayed portion (Fig. 1). Allow the remaining portion of tint to stand for 30 min, then hand-mix or shake it briefly and pour over a part of the previously sprayed panel. Air dry the panels for 2 h at room temperature and force-dry for 1 h at 105°C. The poured portion of the film is generally equal to or lighter in tint than the sprayed portion. The difference between the strength of the sprayed portion and that of the rubbed and poured areas shall be no more than that of a previously agreed upon standard sample.

5.11 *End Product Storage Stability*—Unless the purchaser and the seller have agreed otherwise, the following procedure shall be used. Tint a portion of the mill base prepared as described in 5.10.2 and spray the panel at once. Store the remainder at 50 to 60°C for a period of six weeks in a tightly closed container (Note 3). Then cool to room temperature, shake well, and prepare a tint as before. Any tendency toward crystallization or flocculation will result in a loss of tinting strength. The behavior of a given sample shall be compared to that of a previously agreed upon standard sample.

NOTE 3—Prolonged storage at elevated temperatures will cause some alkyds to gel.

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Standard Specification for Copper Powder for Use in Antifouling Paints¹

This standard is issued under the fixed designation D 964; the number immediately following the designation indicates the year of original adoption 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 copper powder for use in antifouling paints.

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 283 Test Methods for Chemical Analysis of Cuprous Oxide and Copper Pigments³

3. Composition

3.1 The pigment shall consist essentially of finely divided

copper metal and shall conform to the following requirements:

Copper, min, %	99
Coarse particles, max, %	1.0

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods:

5.1.1 *Copper*—Methods D 283.

5.1.2 *Coarse Particles*—Test Methods D 185.

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 D01.31 on Pigment Specifications.

Current edition approved Aug. 31, 1965. Published October 1965. Originally published as D 964 - 48. Last previous edition D 964 - 55.

² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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Standard Test Methods for Para Red and Toluidine Red Pigments¹

This standard is issued under the fixed designation D 970; the number immediately following the designation indicates the year of original adoption 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 7191 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.

^{e1} NOTE—Keywords were added editorially in January 1991.

Scope

1.1 These test methods cover para red and toluidine red pigments (toners) in the dry form commercially known as "pure."

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller³
- D 1193 Specification for Reagent Water⁴
- D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy⁵
- D 3624 Test Method for Low Concentrations of Mercury in Paint by Atomic Absorption Spectroscopy⁵

3. Significance and Use

3.1 These test methods are used to determine the purity and some physical properties of para red and toluidine red pigments. This information is significant to pigment producers and to coatings manufacturers.

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

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.

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² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

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 06.01.

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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water conforming to Type II of Specification D 1193.

4.3 Chloroform.

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

4.5 *Nitric Acid (3+5)*—Mix 3 volumes of concentrated nitric acid (HNO₃, sp gr 1.42) with 5 volumes of water.

4.6 *Potassium Hydroxide, Alcoholic Solution*—Dissolve 10 g of potassium hydroxide (KOH) in 90 mL of ethyl alcohol (95 %).

4.7 *Sodium Hydroxide (100 g/L)*—Dissolve 100 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

5. Solubility in Chloroform

5.1 Place about 0.05 g of the dry pigment in a 50-mL Nessler tube, add 40 mL of chloroform, and warm slightly, stirring with a glass rod. Compare this solution with a similarly prepared solution of a standard sample of known purity. Complete solubility of the pigment in chloroform is indicated if a clear orange red solution is obtained.

NOTE 1—Any turbidity indicating an impurity may be separated conveniently by extracting 5 g of the pigment in a Soxhlet extractor, using chloroform as the solvent. The residue may then be examined and estimated.

6. Qualitative Test for Identity and Purity

6.1 To 10 mL of the chloroform solution (5.1) add 2 mL of alcoholic potassium hydroxide (KOH) solution. An intense violet coloration indicates the presence of para red toner, (1-(*p*-nitro-phenylazo)-2-naphthol). A wine red coloration indicates the presence of toluidine red toner (1-(2-nitro-*p*-tolyl-azo)-2-naphthol). It is well to make side-by-side comparisons, using a standard pigment as control.

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 Pharmacopoeia."

NOTE 2—Infrared spectrophotometry may also be employed for this purpose if equipment is available and suitable reference spectra have been agreed upon by purchaser and seller.

7. Ash

7.1 Transfer about 2 g of the sample (weighed to 0.1 mg) to a tared porcelain or platinum crucible and heat at red heat until all organic matter is destroyed. Cool and weigh.

NOTE 3—For examination for low concentrations of lead and mercury, refer to Test Methods D 3335 and D 3624.

7.2 Calculate the percent of ash present as follows:

$$\text{Ash, \%} = (R/S) \times 100$$

where:

R = residue, g, and

S = sample used, g.

8. Resistance to Acids and Alkalis

8.1 Place about 0.5 g of the dry pigment in a 50-mL beaker and add about 40 mL of HNO₃ (3+5). Stir well and allow the pigment to settle. Decant the liquid and filter it. The filtrate should show no more color than a reference pigment mutually agreed upon by the purchaser and the seller. Repeat using HCl (1+1). Repeat using NaOH solution

(100 g/L), omitting the filtration.

9. Moisture and Other Volatile Matter

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

10. Coarse Particles

10.1 Determine the percent of coarse particles in accordance with Test Methods D 185.

11. Mass Color and Character of Tint

11.1 Determine the mass color and character of tint in accordance with Test Method D 387.

12. Precision and Bias

12.1 Data are not available to determine the precision and bias of these test methods. There are no plans at present to obtain such data. The methods have been in use for several years and are considered acceptable.

13. Keywords

13.1 para red pigment, analysis of; pigment; toluidine red pigment, analysis of; toner, para red and toluidine red

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Standard Test Method for Determining Total Nitrogen in Resins and Plastics¹

This standard is issued under the fixed designation D 1013; the number immediately following the designation indicates the year of original adoption 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.

Scope

1.1 This test method covers the determination of total nitrogen in nitrogen-containing plastics, resins, and resin solutions. This test method is not applicable for use on materials containing nitro-groups.

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:*
D1193 Specification for Reagent Water²

3. Significance and Use

3.1 Total nitrogen content gives an indication of the level or purity of a nitrogen-containing material such as an amine resin. It is important for the quality control of amine resins and is often used to determine whether the proper amounts of many types of nitrogen-containing materials are present in formulated products.

4. Apparatus

4.1 *Kjeldahl Flasks*, for digestion and distillation, 800-mL capacity, moderately heavy wall, and made of hard glass.

4.2 *Connecting Bulbs*, of the Davison type, or a bulb equally effective in preventing mechanical carry-over of the contents of the distillation flask to the condenser.

4.3 *Digestion and Distillation Equipment*—A suitable Kjeldahl digestion and distillation apparatus, such as any of the well-known commercial units for multiple work. The units may be heated either electrically or by gas burner.

4.4 *Connecting Tubes* made of moderately heavy-wall glass tubing, 6 to 8 in. (150 to 200 mm) in length, for conducting the distillate from the condenser to the receiver.

4.5 *Weighing Tube (for Liquid Resins)*—Any convenient device for weighing a few grams of sample in a matter such

that no loss of volatile constituents will be sustained during the weighing operation.

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, reference to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

5.3 *Hydrochloric or Sulfuric Acid*, (standard 0.5 N)—Dilute 43 mL of HCl (sp gr 1.19) or 14 mL of H₂SO₄ (sp gr 1.84) to 1 L with water. Standardize against the 0.5 N NaOH solution (3.7), using the same indicator as will be used in titration of the specimen (4.4).

5.4 *Metallic Mercury or Mercuric Oxide*.

5.5 *Methyl Purple Indicator Solution*.

NOTE 1—Methyl purple has recently met with considerable favor, and is to be recommended because of the abruptness of its color change in the presence of ammonium salts. This indicator may be purchased in solution form ready for use.

5.6 *Methyl Red Indicator Solution*—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

5.7 *Potassium Sulfate*.

5.8 *Sodium Hydroxide Solution* (760 g/L)—Dissolve 1000 g of technical grade NaOH in 1 L of water.

5.9 *Sodium Hydroxide, Standard Solution* (0.5 N)—Prepare a 0.5 N NaOH solution free of carbonates, using reagent grade NaOH, and standardize against the National Bureau of Standards standard reference material No. 84 of potassium acid phthalate, using phenolphthalein indicator. Protect the solution against CO₂ absorption.

5.10 *Sulfide or Thiosulfate Solution*—Dissolve 40 g of K₂S or Na₂S, or 80 g of Na₂S₂O₃ · 5H₂O, in water 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 D01.33 on Polymers and Resins.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 1013 - 49. Last previous edition D 1013 - 81 (1987)¹.

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."

5.11 Sulfuric Acid (sp gr 1.84).

6. Procedure

6.1 Transfer a portion of the sample, weighed to 1 mg, to a Kjeldahl flask, using a weighing tube if the material is a liquid. The quantity of sample taken should be an amount that will contain from 150 to 250 mg of nitrogen. Add from 0.5 to 0.75 g of metallic mercury or the equivalent weight of HgO, 10 g of K₂SO₄, and 25 to 35 mL of H₂SO₄ (sp gr 1.84). If the material under test is a urethane resin or polymer the amount of H₂SO₄ (sp gr 1.84) should be increased to 60 mL. If preferred, H₂SO₄ (1+1) may be used for digestion in place of H₂SO₄ (sp gr 1.84) in which case the number of millilitres used should be doubled.

6.2 Mix the contents of the flask thoroughly, place on the digestion rack, and heat slowly at first until frothing subsides. Increase the heat until the acid boils briskly then continue the digestion for 2 h after the solution becomes colorless or nearly so.

6.3 After allowing the flask to cool, add about 500 mL of water and a little granular zinc or a few boiling aids to prevent bumping. Add an excess (25 to 30 mL) of K₂S, Na₂S, or Na₂S₂O₃ solution. If Na₂S₂O₃ solution is used, it should be mixed with the NaOH solution (760 g/L) so that both are added together. Add an excess (80 to 90 mL) of NaOH solution (760 g/L), pouring it slowly down the side of the flask so that it does not mix at once with the acid solution. Immediately connect the flask to the connecting bulb and condenser, and mix the contents of the flask thoroughly.

6.4 Distill the solution into 50 mL of 0.5 N HCl or H₂SO₄, making certain that the connecting tube from the

condenser extends below the surface of the acid in the receiver. Continue the distillation until the ammonia has been collected in the receiver (about 300 mL of distillate).

6.5 Add 5 to 7 drops of methyl red or methyl purple indicator solution and titrate the excess acid with 0.5 N NaOH solution.

6.6 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

7. Calculation

7.1 Calculate the percent nitrogen *A* as follows:

$$A = [(B - V) N \times 0.014] / S \times 100$$

where:

B = NaOH solution required for titration of the blank, mL,

V = NaOH solution required for titration of the specimen, mL,

N = normality of the NaOH solution, and

S = specimen weight used, g.

8. Precision

8.1 On the basis of an interlaboratory test of this test method in which operators in five laboratories analyzed six materials, 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 analyst should be considered suspect if they differ by more than 0.25 % absolute.

8.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by analysts in different laboratories, should be considered suspect if they differ by more than 0.5 % absolute.

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 Chemical Analysis of Blue Pigments¹

This standard is issued under the fixed designation D 1135; the number immediately following the designation indicates the year of original adoption 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 that has been adopted by the Department of Defense.

⁶¹ NOTE—Keywords were added editorially in January 1991.

Scope

1.1 These test methods cover procedures for the chemical analysis of blue pigments known commercially as iron blue, copper phthalocyanine blue, and ultramarine blue.

1.2 The analytical procedures appear in the following order:

	Sections
IRON BLUE PIGMENTS	
Identification	6
Moisture by the Brabender Moisture Tester	7 and 8
Moisture by Toluene Distillation	9
Water-Soluble Matter by Extraction	10
Water-Soluble Salts by Electrical Conductivity	11
Detection of Acid-Insoluble Extenders	12 and 13
Detection of Acid-Soluble Extenders	14 to 17
Detection of Organic Colors and Lakes	18
COPPER PHTHALOCYANINE BLUE PIGMENTS	
Identification	19
Moisture and Other Volatile Matter	20
Detection of Basic Dye Derivatives	21
Detection of Other Organic Coloring Matter	22
Detection of Ultramarine Blue	23
Detection of Iron Blue	24
ULTRAMARINE BLUE	
Identification	25
Moisture and Other Volatile Matter	26
Water-Soluble Matter	27
Detection of Basic Dye Derivatives	28
Detection of Other Organic Coloring Matter	29

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 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²

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 March 27, 1986. Published May 1986. Originally published as D 1135 - 50 T. Last previous edition D 1135 - 81.

² Annual Book of ASTM Standards, Vol 06.02.

D 1193 Specification for Reagent Water³

D 1208 Test Methods for Common Properties of Certain Pigments²

D 2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

3. Significance and Use

3.1 These test methods are suitable for determining if impurities are present and establishing that the required pigments are present. These test methods may be used for manufacturing quality control.

4. Purity of Reagents and Water

4.1 *Purity of Reagents*—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 II of Specification D 1193.

5. Preparation of Sample

5.1 Mix the sample thoroughly and take a representative portion for analysis. Reduce any lumps or coarse particles to a fine powder by grinding.

5.2 Grind extracted pigments to pass a No. 80 (180- μ m) sieve.⁶ Discard any skins that do not pass through the sieve. Mix the finely ground pigment thoroughly.

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

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ "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 Detailed requirements for this sieve are given in Specification E 11.

IRON BLUE
(Prussian Blue, Chinese Blue, Milori Blue)

IDENTIFICATION**6. Procedure**

6.1 To approximately 0.1 g of pigment in a 50-mL beaker, add 15 mL of NaOH solution (50 g/L). Heat to boiling. In a few minutes the blue color should be completely destroyed, giving in its place the characteristic reddish brown precipitate of ferric hydroxide. Add HCl (1+1) until faintly acid to litmus. The iron blue should be reformed, yielding again the characteristic blue color.

NOTE 1—If the NaOH treatment does not completely destroy the blue color, the evidence is strong that a foreign pigment is present. If this occurs, it is best to filter the alkaline solution, weakly acidify the filtrate with HCl (1+1), and add approximately 2 mL of a ferric salt solution (ferric sulfate or ferric alum (20 g/L)). The formation of a blue precipitate established the pigment as consisting, at least in part, of iron blue.

MOISTURE BY THE BRABENDER MOISTURE TESTER**7. Apparatus**

7.1 *Brabender Moisture Tester*—The Brabender moisture tester (Fig. 1) consists of a constant-temperature oven with weighing apparatus attached. Specimens placed in the oven may be weighed without opening the oven door or removing the specimen from the oven, as the balance scale is calibrated to read directly in percent of moisture.

8. Procedure

8.1 Set the temperature control at 160°C and allow the oven to reach this temperature before checking or making any weighing. Check the scale by placing a sample dish containing 9.200 g standard analytical balance weights in the oven. The apparatus should read 8.0 % moisture. If it does



FIG. 1 Brabender Moisture Tester

not, adjust to a reading of 8.0 % by turning either the right hand or left hand foot screw.

8.2 Weight 10.000 g of the sample in the aluminum dish and place in the oven. For a series of specimens, all should be placed in the oven at as nearly the same time as possible. No specimen should occupy the position directly in front of the door. Weigh the specimen at the end of each hour for 5 h. After each weighing, return the specimen to its original place in the oven.

NOTE 2—The dried pigment is very hygroscopic. In order to obtain consistent results, the specimen position must not be changed and the oven door must not be opened.

8.3 *Calculation and Report*—Plot time against percent loss in weight on rectilinear cross-section paper. Extrapolate the linear portion of the curve to zero time. That portion beyond about 2 h will be essentially linear. Report the percent loss in weight at the extrapolated zero time as the percent moisture in the pigment.

NOTE 3—The pigment undergoes a slight loss in weight due to decomposition during the heating. The method of plotting and extrapolation corrects for this loss. An approximate value for moisture content may be obtained by taking the reading at the end of the first hour's heating. An occasional pigment may decompose rapidly at the oven temperature. In such cases, determine moisture by the toluene distillation method (Section 9).

MOISTURE BY TOLUENE DISTILLATION

9. Procedure

9.1 Determine the moisture content in accordance with Sections 7 and 8 of Method D 1208, but using 25 g of pigment and 200 mL of toluene and adjusting the calculation accordingly.

WATER-SOLUBLE MATTER BY EXTRACTION

10. Procedure

10.1 Determine whether or not the pigment is easily wet by water at room temperature by adding a little to some water in a beaker. If it tends to float on top of the water with very little, if any, tendency to sink to the bottom or disperse throughout the solution, even after agitation, it contains a hydrophobic treating agent.

10.2 Weigh to 1 mg about 2.5 g of the pigment into a 250-mL volumetric flask. If the pigment is hydrophobic as tested above, moisten thoroughly with a few mL of ethyl alcohol (Note 4). If the pigment is easily wet with water, no alcohol is necessary. Fill the flask about half full with water and shake to disperse the pigment thoroughly. Fill to the mark and again shake. Allow to remain at room temperature for not less than 15 h, shaking from time to time, preferably with an automatic shaking device.

NOTE 4—Ethyl alcohol denatured with acetone (Formula No. 23A) or denatured with methyl alcohol (Formula No. 3A) has been found suitable.

10.3 Let settle, filter through a dry filter paper, and discard the first 20 mL of the filtrate. Transfer 100 mL of the clear filtrate to a weighed dish, and evaporate to dryness on a steam bath. Dry for 1 h in an oven at $105 \pm 2^\circ\text{C}$, cool, and weigh.

10.4 *Calculation*—Calculate the percent of water-soluble matter as follows:

Water-soluble matter, % = grams of residue $\times$ 100

WATER-SOLUBLE SALTS BY ELECTRICAL CONDUCTIVITY

11. Procedure

11.1 Determine water soluble salt content in accordance with Test Method D 2448.

DETECTION OF ACID-INSOLUBLE EXTENDERS

12. Scope

12.1 Acid-insoluble extenders include barium sulfate, silica, and silicates. Alumina may also be found, in part, with this group.

13. Procedure

13.1 Ignite about 1 g (weighed to 0.1 mg) of the sample at a low temperature, just enough to decompose the last trace of pigment but not high enough to render the iron difficultly soluble in HCl (Note 5). Cool, and add 15 mL of HCl (sp gr 1.19) and a few drops of bromine. Cover with a watch glass and evaporate to a sirup. Add about 15 mL of water, and boil. It may be necessary to add a drop or two of HCl to effect complete solution of the ferric iron residue. Filter and wash with hot water. Save the filtrate for the determination of alumina hydrate (Section 16).

NOTE 5—The ignition can conveniently be carried out in a 250-mL beaker or a porcelain dish over a free flame. Oxidation of the specimen is evidenced by a dull glowing. While being heated, it is advisable to roll the specimen around in the beaker or dish exposing all of the surface to the oxygen of the air. A moderately low flame should be used and the ignition is complete when the specimen ceases to glow and acquires a uniform brown color.

13.2 A residue of less than 3 mg that appears as small black specks can be neglected, since quite frequently a small amount of iron is rendered insoluble or a small amount of blue pigment escapes destruction. Ignite the residue and weigh. If appreciably more than 3 mg are present, extenders should be suspected, and if it is required to know which extenders are present, analyze the residue for silica, barium sulfate, and alumina.

NOTE 6—If alumina is present, it may appear both with the acid-insoluble and acid-soluble extenders. As a rule, most of it will appear with the acid-soluble extenders.

DETECTION OF ACID-SOLUBLE EXTENDERS

14. Scope

14.1 Acid-soluble extenders include the alkaline earth carbonates or sulfates, magnesium carbonate, and alumina hydrate.

15. Reagents

15.1 *Ammonium Oxalate Solution*—Dissolve 40 g of ammonium oxalate monohydrate in warm water and dilute to 1 L.

15.2 *Ammonium Phosphate Solution*—Dissolve 100 g of $(\text{NH}_4)_2\text{HPO}_4$ in water and dilute to 1 L.

16. Procedure for Alumina Hydrate

16.1 To the filtrate from 13.1, add NaOH solution (50 g/L) until just alkaline; then add 5 mL excess. Boil for about 2 min and let stand in a warm place until the hydrous iron

oxide is coagulated. Filter through a rapid filter paper, wash a few times with hot water, and discard the precipitate.

16.2 To the filtrate add 7 mL of HCl (1+1). Add NH_4OH (1+4) until just ammoniacal. Boil about 2 min. If no precipitate is apparent, allow to stand about $\frac{1}{2}$ h. If the solution is still clear, no alumina hydrate is present. A white gelatinous precipitate indicates alumina hydrate. Filter and save the filtrate for the detection of alkaline earth and magnesium salts (Section 17).

16.3 If a rough estimate of the amount of alumina hydrate is desired, the residue may be washed, dried, ignited, and weighed as Al_2O_3 .

17. Procedure for Alkaline Earth and Magnesium Salts

17.1 To the filtrate from Section 16, add HCl (1+1) until faintly acid. Divide the filtrate into two portions.

17.2 To one portion of the filtrate, add 15 mL of $(\text{NH}_4)_2\text{HPO}_4$ solution and neutralize with NH_4OH (sp gr 0.90). Add 10 mL excess NH_4OH . If no precipitate forms immediately, let stand for a short time in a cool place with occasional vigorous stirring. Rub the inside of the beaker from time to time with a glass rod to initiate crystallization. A white microcrystalline precipitate indicates the presence of magnesium salts and possibly some alkaline earth salts as well.

17.3 To the other portion of the filtrate, add 5 mL of

ammonium oxalate solution. Make slightly alkaline with NH_4OH (sp gr 0.90). If no precipitate forms immediately, warm on a hot plate and let stand for a short time. A white microcrystalline precipitate indicates the presence of alkaline earth salts. If it is required to know specifically which acid soluble extenders are present, any of the established tests for these metal salts may be employed.

DETECTION OF ORGANIC COLORS AND LAKES

18. Procedure

18.1 Boil 2 g of the sample for 2 min with 25 mL of water. Let settle and decant the supernatant liquid. Similarly, boil the residue with 25 mL of denatured ethyl alcohol (95 %) and decant as before. Likewise boil with 25 mL of chloroform and again decant. If any one of the above solutions is colored, organic colors are present. If all solutions remain colorless, disregarding a slight yellowish cast, organic colors are presumably absent. The presence of organic colors resistant to the above reagents is unlikely, but may be tested by reference to procedures given in standard reference works.⁷

⁷ Reference may be made to the following: Payne, H. F., "Organic Coatings Technology," Vol II, John Wiley & Sons, Inc., New York, NY, 1961.

COPPER PHTHALOCYANINE BLUE

IDENTIFICATION

19. Procedure

19.1 To about 0.05 g of the sample in a 50-mL beaker, add 30 mL of H_2SO_4 (sp gr 1.84). Stir occasionally for 15 min; the sample should dissolve, forming a dark greenish yellow solution (color best seen on the side of the beaker). Pour the solution into 250 mL of water and stir. The copper phthalocyanine should immediately precipitate as a brilliant blue flocculent mass.

19.2 Filter off the precipitate, washing once or twice with water. Scrape a small amount of the precipitate off the filter, place on a clean platinum wire moistened with HCl, and subject it to the low flame of a bunsen burner. As the precipitate burns, a light blue-green flame should be clearly evident. This indicates organically combined copper.

NOTE 7—Characteristic spectrophotometric absorption spectra in the near infrared range (700 to 900 nm) are exhibited by dilute solutions of copper phthalocyanine blue pigments in H_2SO_4 (sp gr 1.84) (2 to 50 mg/L). The absorption maxima are so sharp and well defined that they may be used for positive qualitative identification of the various phthalocyanine pigments.

MOISTURE AND OTHER VOLATILE MATTER

20. Procedure

20.1 Determine moisture in accordance with Method A of Test Methods D 280.

DETECTION OF BASIC DYE DERIVATIVES

21. Procedure

21.1 Add to 1 g of the sample, 50 mL of a mixture of

equal parts of NH_4OH (sp gr 0.90) and denatured ethyl alcohol (95 %). Warm gently and filter. Neutralize the filtrate with tartaric acid solution (200 g/L) until slightly acid to litmus. If the solution is colorless, discounting a slight yellow tinge, no basic dye is present.

21.2 If the solution is colored beyond a slight yellow tinge, add about 5 mL of 0.1 N TiCl_3 solution (Note 8). If a basic dye is present, the color will lighten significantly. If no basic dye is present, no significant color change will occur.

NOTE 8—Titanium trichloride is marketed as a 16 % solution in HCl. Mix 7 mL of this solution with 90 mL of HCl (1+2) to obtain a reagent approximately 0.1 N. Protect from oxidation.

DETECTION OF OTHER ORGANIC COLORING MATTER

22. Procedure

22.1 Weigh about 0.05 g of the sample into each of two 50-mL beakers. Add 25 mL (at room temperature) of denatured ethyl alcohol (95 %) to one beaker and about 25 mL of acetone to the other. Stir each well for a few minutes, and let stand for about 2 h. Filter through two thicknesses of medium-texture, qualitative filter paper. If neither filtrate possesses more than an extremely slight pink, yellow, or blue cast, organic colors are presumably absent, but may be tested for by procedures given in standard reference works.⁷

DETECTION OF ULTRAMARINE BLUE

23. Procedure

23.1 Warm gently about 1 g of the sample with HCl (1+1). Decomposition of the ultramarine blue takes place with evolution of H_2S . This may be detected by either its

own coloration appearing on a strip of moistened paper.

DETECTION OF IRON BLUE

Procedure
Weighing of the sample in a 100-mL beaker, add 25 mL of solution (50 g/L). Boil for about 1 min. Dilute to

approximately 40 mL and filter.

24.2 Add HCl (1+1) to the filtrate until faintly acid to litmus. Add 2 mL of a ferric iron solution (ferric sulfate or ferric alum (20 g/L)). The development of a blue color reveals the presence of ferrocyanide, and hence iron blue in the original pigment. For amounts around 0.05 % iron blue, the color may take 2 to 3 h to develop.

ULTRAMARINE BLUE

IDENTIFICATION

Procedure
Warm gently approximately 0.1 g pigment with HCl. If the pigment is ultramarine blue, the color will be destroyed with the liberation of sulfur and H₂S, may be detected by its characteristic odor or by a coloration appearing on a strip of moistened lead paper when held above the beaker. Quite frequently this is not necessary, the destruction taking place at temperature.

If any color remains after this treatment, it can be evidence that a foreign pigment is present.

MOISTURE AND OTHER VOLATILE MATTER

Procedure
Determine moisture in accordance with Method A of Methods D 280.

WATER-SOLUBLE MATTER

Procedure
Establish whether or not the pigment is easily wet by adding a little to some water in a beaker. If it tends to float on top with little tendency to sink to the bottom or to pass through the solution it contains a hydrophobic binding agent.

22 Transfer 2.5 g (weighed to 1 mg) of the sample to a 100-mL beaker. If the pigment is hydrophobic as tested in 21, moisten thoroughly with a few millilitres of ethyl alcohol (Note 4). If the pigment is easily wet with water, the

alcohol is not necessary. Add 250 mL of water and boil for 5 min with good agitation.

27.3 Cool and transfer to a 250-mL volumetric flask. Dilute to the mark with wash water from the beaker; complete transfer of the pigment to the flask is not necessary. After thorough shaking, allow to settle somewhat and filter the supernatant liquid through a dry paper, discarding the first 20 mL. Evaporate 100 mL of the clear filtrate to dryness in a weighed dish. Heat for 1 h at 105 ± 2°C, cool, and weigh.

27.4 Calculation—Calculate the percent of water-soluble matter as follows:

$$\text{Water-soluble matter, \%} = \text{grams of residue} \times 100$$

DETECTION OF BASIC DYE DERIVATIVES

28. Procedure

28.1 Determine basic dye derivatives in accordance with Section 21.

DETECTION OF OTHER ORGANIC COLORING MATTER

29. Procedure

29.1 Determine other organic coloring matter in accordance with Section 22.

30. Precision and Bias

30.1 Precision and Bias have not been determined.

31. Keywords

31.1 blue pigment, chemical analysis of; Brabender moisture tester; copper phthalocyanine blue, identification of; moisture, Brabender tester; moisture, toluene distillation; pigment, acid soluble extenders; ultramarine-blue pigment, identification of

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Standard Test Method for Roundness of Glass Spheres¹

This standard is issued under the fixed designation D 1155; the number immediately following the designation indicates the year of original adoption 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 method² covers the determination of the percent of true spheres in glass spheres used for retroreflective marking purposes and industrial uses.

1.2 This test method includes two procedures as follows:

1.2.1 *Procedure A*, in which the selected specimen is split into two size ranges or groups prior to separation into true spheres and irregular particles, and

1.2.2 *Procedure B*, in which the selected specimen is split into five size ranges or groups prior to separation.

1.2.3 In determining compliance with specification requirements, either Procedure A or Procedure B may be used. Where tests indicate failure to meet the specified percent of true spheres and irregular particles, the referee test shall be made in accordance with Procedure B.

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:

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

3. Summary of Test Method

3.1 The glass particles are mechanically separated into true spheres and irregular particles by controlled vibration on a glass plate fixed at a predetermined slope.

4. Significance and Use

4.1 The roundness of glass spheres is one measureable aspect relating to their performance as a retroreflective media. The function of this test method is to measure the percent of true spheres as related to compliance with applicable specifications.

NOTE 1—This method has been used in other industrial areas outside the intended scope of 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 D01.44 on Traffic Coatings.

Current edition approved May 26, 1989. Published July 1989. Originally published as D 1155 - 51 T. Last previous edition D 1155 - 53(1983)⁴.

² For information on the development of this test method, reference may be made to the paper by Keeley, A. E., "Roundness Testing of Glass Spheres," *ASTM Bulletin*, No. 174, May, 1951, p. 72.

³ *Annual Book of ASTM Standards*, Vol 14.02.

5. Apparatus (Fig. 1)

5.1 *Electrical Feeder-Vibrator*, upon which is mounted a smooth glass panel, 6 in. (152.4 mm) wide and 15 in. (381 mm) long.

5.2 *Hinged Base*, supporting the vibrator and panel in such a manner that the angle of slope of the glass panel with the horizontal may be varied and fixed in any predetermined position.

5.3 *Vibrator*—Means of varying the amplitude or strength of the vibrations transmitted to the glass panel, at a fixed frequency of 60 impulses per second.

5.4 *Feeding Device or Pan*, affixed to the glass panel in such a manner that the selected sample of glass may be evenly dropped at a uniform rate upon the glass panel, from various heights above the panel and at various points on the slope.

5.5 *Collecting Pans or Containers*, at either end of the sloping panel, in which to collect the spheres and irregular particles.

6. Selection of Specimen

6.1 Select a 50-g specimen of the glass spheres to be tested for roundness in one of the following ways:

6.1.1 By mechanically splitting a bag or other container of glass spheres, selected at random from the shipment to be tested, or

6.1.2 By grain or seed-rod selection from the container.

7. Procedure A

7.1 Sieve the selected specimen through a 300- μ m (No. 50) sieve (Note 2). Run the spheres retained on the sieve as one group, and run the spheres passing the sieve as a second group.

NOTE 2—Detailed requirements for ASTM sieves are given in Specification E 11.

7.2 Level the glass panel; then raise one end from the horizontal by the distance in inches indicated on the calibration curve in Fig. 2 for the average diameter of spheres in the group. Affix the feed hopper to the side of the panel at the upper one-third point of the slope, so that the spheres may be dropped in a uniform monolayer onto the glass panel from a height of approximately 1/2 in. (13 mm).

7.3 Place the size group to be tested in the feed pan, and start the vibrator. Set the vibrator amplitude control at such a position that irregular particles on the upper half of the panel will move slowly up the slope, while the true spheres roll down. Feed slowly, at such a rate that no "bunching up" or flooding of spheres on the panel occurs.

7.4 When the glass panel is well covered with spheres, stop feeding until separation of true spheres has occurred. Stop the vibrator and, after all true spheres have rolled down the

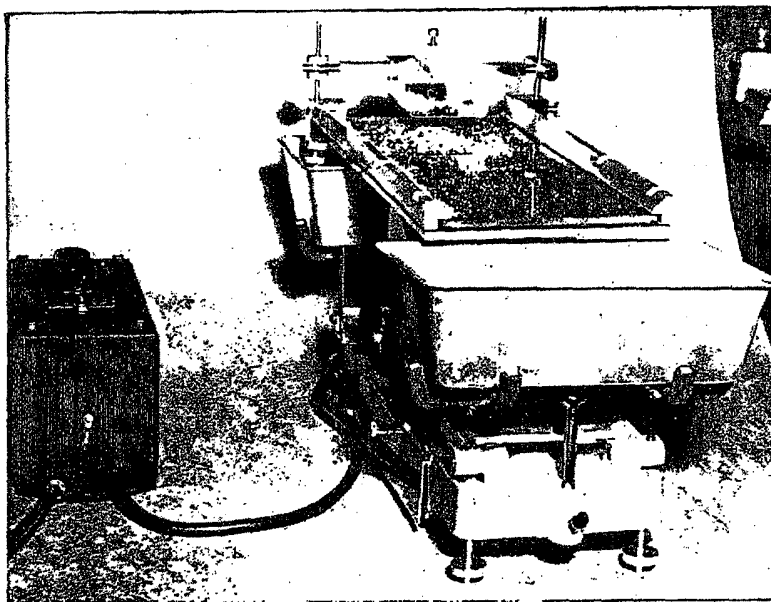


FIG. 1 Apparatus for Roundness Test of Glass Spheres

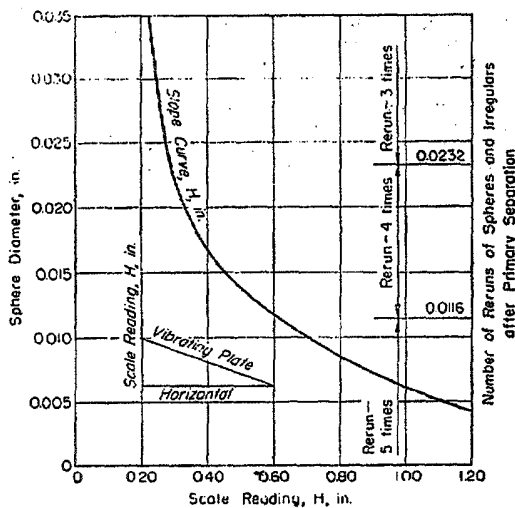


FIG. 2 Calibration Curve for Roundness Tester

slope into the sphere pan, brush or scrape all particles remaining on the panel into the upper pan containing the irregular particles. For purpose of test, all particles not rolling freely down the slope are considered as irregular.

7.5 Repeat the procedure described in 7.3 and 7.4 until the selected size group has been completely separated, removing the true spheres and irregular particles from the collecting pans into appropriate containers.

7.6 Fill the feed pan with the true spheres collected in the primary separation, and repeat the procedure described in 7.3 and 7.4. Next, fill the feed pan with the irregular particles collected in the primary separation, and again repeat the

procedure described in 7.3 and 7.4. Examine the separated spheres and irregular particles under a 20-diameter magnifying glass and repeat the reruns until satisfactory separation is obtained.

7.7 Determine the total weights of the true spheres and of the irregular particles obtained by the above separations, and record.

7.8 Using the second size group obtained in accordance with 7.1, repeat the procedure described in 7.2 to 7.7.

7.9 From the total weight of true spheres obtained from both size groups, calculate the percent of true spheres in the total specimen, using as 100 % the total weight of true spheres plus irregular particles collected in the test—thereby eliminating from the calculation any loss of spheres that may have occurred during handling and testing.

8. Procedure B

8.1 Divide the specimen into five size ranges or groups, as follows:

Passing Sieve	Retained on Sieve
600- μ m (No. 30)	425- μ m
425- μ m (No. 40)	300- μ m
300- μ m (No. 50)	212- μ m
212- μ m (No. 70)	...

8.2 Level the glass panel; then raise one end from the horizontal by the distance in inches indicated on the calibration curve in Fig. 2 for the minimum diameter of spheres in the group. Affix the feed hopper over the center line of the panel, at the upper one-third point of the slope, with the feed end up-slope and approximately $\frac{3}{8}$ in. (10 mm) from the glass panel.

8.3 Place the size group to be tested in the feed pan, and start the vibrator. Set the vibrator amplitude control at such a position that irregular particles on the upper half of the panel will move slowly up the slope, while the true spheres

roll down. Feed slowly, at such a rate that no "bunching up" or flooding of spheres on the panel occurs.

8.4 When the glass panel is well covered with spheres, stop feeding until separation of true spheres has occurred. Stop the vibrator and, after all true spheres have rolled down the slope into the sphere pan, brush or scrape all particles remaining on the panel into the upper pan containing the irregular particles. For purposes of this test, all particles not rolling freely down the slope are considered as irregular.

8.5 Repeat the procedure described in 8.3 and 8.4 until the selected size group has been completely separated, removing the true spheres and irregular particles from the collecting pans into appropriate containers.

8.6 Fill the feed pan with the true spheres collected in the primary separation, and repeat the procedure described in 8.3 and 8.4. Next, fill the feed pan with the irregular particles collected in the primary separation and again repeat the procedure described in 8.3 and 8.4. Examine the separated spheres and irregular particles under a 20-diameter magnifying glass and repeat the reruns until satisfactory separation is obtained.

8.7 The procedure described in 8.3, 8.4, and 8.5 comprises the primary separation and that in 8.6 is one complete rerun. Make a primary separation for each of the five size groups listed in 8.1 and then make the appropriate number of reruns for each size group, as follows:

Spheres Retained on Sieve	Reruns
425- μ m (No. 40)	4
300- μ m (No. 50)	4
212- μ m (No. 70)	5
Spheres Passing Sieve	
212- μ m (No. 70)	5

8.8 From the total weight of true spheres obtained from separations from all five size groups, calculate the percent of true spheres in the total specimen, using as 100 % the total weight of true spheres plus irregular particles collected in the complete test—thereby eliminating from the calculation any loss of spheres that may have occurred during handling and testing.

9. Report

9.1 Report the following information:

9.1.1 The weight percent of true spheres in the total specimen and

9.1.2 Whether Procedure A or Procedure B was used.

10. Precision and Bias

10.1 A round-robin study is currently underway in order to generate a precision statement.

11. Keywords

11.2 roundness; glass spheres

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Standard Test Method for Solvent Tolerance of Amine Resins¹

This standard is issued under the fixed designation D 1198; the number immediately following the designation indicates the year of original adoption 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 quantity of hydrocarbon solvent that an amine resin will tolerate at 77°F (25°C).

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 362 Specification for Industrial Grade Toluene²
- D 891 Test Methods for Specific Gravity of Liquid Industrial Chemicals³

3. Summary of Test Method

3.1 A standard solvent prepared from three relatively pure hydrocarbons is employed to titrate a specimen of the resin to a defined degree of turbidity and the solvent tolerance is calculated from the volume of solvent added.

4. Significance and Use

4.1 This test method gives an indication of the solubility of an amine resin. This property is important in determining the utility of the resin in new paint formulations and in ensuring its compatibility with existing paint formulations. This test method is thus useful for evaluation and quality control of amine resins.

5. Apparatus

- 5.1 *Erlenmeyer Flask*, wide-mouth, 250-mL capacity.
- 5.2 *Buret*, 50-mL capacity, graduated in 0.1-mL divisions.
- 5.3 *Print Specimen*—A sheet of paper having on it printing in a black ink from 10-point, No. 31 old style type, including a double quotation mark (lower case letters approximately 1/16 in. (1.5 mm) high) with normal spacing, upper and lower case with no italicized or bold letters.

6. Reagents

- 6.1 *Decahydronaphthalene*, having a boiling range be-

tween 188 and 195°C and a density, at 25°C, between 0.885 and 0.890 g/mL.⁴

- 6.2 *Isooctane* (2,2,4-trimethylpentane), having a purity of at least 99 mol %.

- 6.3 *Toluene*, conforming to Specification D 362.

- 6.4 *Solvent Mixture*:

6.4.1 Blend the above three hydrocarbons in the proportions indicated below and determine the density at 25°C in accordance with Test Methods D 891.

Parts by Weight	
Isooctane	84
Decahydronaphthalene	8
Toluene	8

6.4.2 Standardize the solvent by titration against 20 ± 0.1 g of mineral-spirits-standardized kauri-butanol solution.⁵ The volume of solvent necessary to reach the end point described in 7.3 should be between 31 and 32 mL. If the volume of solvent is not between 31 and 32 mL make an adjustment by varying the concentration of decahydronaphthalene. If the titrant volume is low add more decahydronaphthalene, if high add 84 + 8 isooctane-toluene mixture. Determine the density of the final mixture at 25°C.

NOTE 1—Although similar in certain respects to commercial products identified as mineral spirits, the solvent possesses an advantage for this determination in that it is a mixture of known constant composition.

7. Procedure

7.1 Adjust the temperature of the solvent and the sample to 77 ± 1°F (25 ± 0.5°C), and conduct the determination at this temperature.

7.2 Weigh the Erlenmeyer flask on a suitable balance to 10 mg. Transfer approximately 10 g of resin to the flask and again weigh to 10 mg to obtain the weight of the specimen.

7.3 Fill the buret with the solvent, and titrate the specimen while swirling the flask constantly. Add the solvent rapidly, but at a rate such that precipitation caused by local excess of solvent is kept to a minimum, until about 90 % of the required amount has been added. As the end point is approached, add the solvent in small increments. The end point is reached when the 10-point print placed beneath the flask becomes illegible. Alternatively the end point may be taken as the point where a double quotation mark can no

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 01.33 on Polymers and Resins.

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² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 15.05.

⁴ The product commercially available as "Decalin" has been found satisfactory for this purpose.

⁵ Mineral-spirits-standardized kauri-butanol solution (Catalog No. 1846), available from Harleco Co., 60th and Woodland Ave., Philadelphia, PA 19143, have been found suitable for this purpose.

longer be resolved. Record the volume of solvent added.

8. Calculation

8.1 Calculate the solvent tolerance T , expressed as grams of solvent tolerated by 100 g of resin, as follows:

$$T = (VD/S) \times 100$$

where:

V = solvent used in the titration, mL,

D = density of solvent, and

S = specimen weight used, g.

NOTE 2—If preferred, instead of determining the density of the solvent the flask may be weighed again after completing the titration, thus obtaining directly the weight of solvent required.

9. Report

9.1 Report the grams of solvent tolerated by 100 g of sample to 1 g. Duplicate runs that agree within 2.7 % relative are acceptable for averaging (95 % confidence level).

10. Precision

10.1 The precision estimates are based on an interlabora-

tory study of this test method on one sample each of a modified urea-formaldehyde resin and a butylated melamine-formaldehyde resin. Six laboratories analyzed each sample in duplicate and repeated the analysis on another day for a total of 48 determinations. The results do not include the component of variation due to difference in solvent since the same solvent was used by all participants. The within-laboratory coefficient of variation was found to be 0.65 % with 11 degrees of freedom and the between-laboratory coefficient of variation was found to be 1.67 % with 5 degrees of freedom. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence limit:

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 2.9 % 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 6.1 % relative.

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Standard Specification for Calcium Carbonate Pigments¹

This standard is issued under the fixed designation D 1199; the number immediately following the designation indicates the year of original adoption 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 specification covers two types of high-content calcium carbonate pigments, as follows:

1.1.1 *Type PC*—Calcium carbonate precipitate, prepared either by complete solution or by carbonation of lime.

1.1.2 *Type GC*—Ground mineral product.

1.2 Six grades of pigments, based on particle size (see 3.3) are covered.

Referenced Documents

2.1 ASTM Standards:

C 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime²

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out³

D 718 Test Methods for Analysis of Aluminum Silicate Pigment³

D 1366 Practice for Reporting Particle Size Characteristics of Pigments³

D 3360 Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments³

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁴

3. Composition and Properties

3.1 The pigment may be prepared by chemical precipitation or by the fine grinding of natural calcium carbonate containing minerals. If additional agents are used or any surface treatment is given, their purpose shall be indicated; acceptance shall be as agreed upon by the purchaser and the seller.

3.2 *Composition*—The pigment shall conform to the requirements for composition prescribed in Table 1.

3.3 *Fineness*—The pigment shall conform to the following general requirements for fineness for the grade specified:

3.3.1 *Grade I (Fine Paint Grade)*—This grade possesses substantial amounts of material in the fine sizes, and is in general essentially below 15 to 20 μm maximum size. Coarse particles retained on the No. 325 (45- μm) sieve shall be less than 0.05 %. The maximum Specific Surface Diameter (SSD) shall be 2.5 μm .

3.3.2 *Grade II (Coarse Paint Grade)*—This grade is characterized by substantial amounts in the 5- to 45- μm range, and is lower in pigment value than Grade I. Coarse particles retained on the No. 325 (45- μm) sieve shall be less than 0.5 %. The maximum SSD shall be 6 μm .

3.3.3 *Grade III (Filler Grade)*—This grade is characterized by substantial amounts in the 10- to 45- μm range but with the coarse particles retained on No. 325 (45- μm) sieve less than 25 % and a maximum SSD of 9 μm .

3.3.4 *Grade IV (Putty Powder Grade)*—This grade possesses less fines, and have substantial amounts of coarse particles. The coarse particles, however, shall not exceed 30 % retained on the No. 200 (75- μm) sieve. The maximum SSD shall be 12 μm .

3.3.5 *Grade V (Superfine Grade)*—This grade is a superfine ground natural limestone and is characterized by major amounts less than 5 μm and a weight median particle size in the range of 1 μm . The SSD is finer than 1 μm .

3.3.5.1 Particle size methods for Grade V that are applicable include transmission electron microscopy, scanning electron microscopy, and the Sedigraph.⁵ Specific Surface Diameter can be determined by BET nitrogen absorption. The method of measurement produces different values, therefore, the method of measurement shall be agreed upon by the purchaser and the seller.

3.3.6 *Grade VI (Ultrafine Grade)*—This grade is an ultrafine precipitated calcium carbonate and is characterized by major amounts less than 2 μm and a median particle diameter determined by electron microscopy in the range of 0.05 μm .

3.3.6.1 Particle size methods for Grade VI that are applicable include transmission electron microscopy and scanning electron microscopy. The Sedigraph will give weight median particle size values approximately 10 times greater (~0.5 μm) than by microscopy. Since the method of measurement produces different values, the method of measurement shall be agreed upon by the purchaser and the seller.

3.3.7 When closer control within a grade is required, the

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 D01.31 on Pigment Specifications.

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² Annual Book of ASTM Standards, Vol 04.01.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

⁵ Sedigraph, manufactured by Micromeritics, 5680 Goshen Springs Rd., Norcross, GA 30093, has been found suitable for this purpose.

fineness requirements shall be as agreed upon by the purchaser and the seller.

3.4 Dry Brightness or Dispersed Color—The dry brightness or dispersed color shall be equal, within agreed upon tolerances, to that of a reference sample agreed upon by the purchaser and the seller.

3.5 Oil Absorption—Oil absorption values shall be as agreed upon by the purchaser and the seller.

4. Sampling

4.1 Two samples, each more than 1 lb (0.45 kg) shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or after blending, in equal quantities, the samples from the same production unit forming a composite sample. Before testing, each of the samples shall be split, and one half of each may be sealed for referee testing.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be agreed upon by the purchaser and the seller.

5.2 Calcium and Magnesium Reported as Carbonate—Methods C 25.

TABLE 1 Composition of Calcium Carbonate Pigments

	Type PC	Type GC
Moisture and other volatile matter, max, %	0.7	0.2
Calcium reported as carbonate, moisture-free, min, %	96.5 ^A	...
Total calcium and magnesium reported as carbonates, moisture-free, min, %	...	94
Magnesium as carbonate, max, %	...	...

^A Does not apply to specialty calcium carbonate.

5.3 Moisture and Other Volatile Matter—Method A of Test Methods D 280.

5.4 Oil Absorption—Test Method D 281.

5.5 Coarse Particles—Method D 718.

5.6 Dispersed Color—Method D 718, substituting the reference and test samples of calcium carbonate pigment for the standard extender pigment and sample respectively.

5.7 Specific Surface Diameter—Practice D 1366.⁶

5.8 Dry Brightness—The test sample and reference sample shall be prepared into suitable smooth, dry, packed surfaces in accordance with accepted practice and tested for reflectance using the green filter in accordance with Test Method E 97.

5.9 Particle Size—Test Method D 3360 is applicable only to Grades I, II, III, and IV.

NOTE 2—See 3.3.5 for other acceptable methods.

6. Keywords

6.1 calcium carbonate

⁶ The Fisher Sub-Sieve Sizer has been found satisfactory for use with this practice.

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Standard Test Methods for Common Properties of Certain Pigments¹

This standard is issued under the fixed designation D 1208; the number immediately following the designation indicates the year of original adoption 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 4462, 5111, 5241, 5261 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.

Scope

1.1 These test methods cover procedures for determining certain properties of pigments. The procedures appear in the following order:

DRY PIGMENTS	Sections
Loss on Ignition and Ash	4
Matter Soluble in Water	5
Hydrogen Ion Concentration (pH Value)	6
Alkalinity or Acidity by Titration	7 and 8
Water Content (Distillation Method)	9 and 10
PIGMENT PASTES IN OIL	
Water Content (Distillation Method)	9 and 10
Pigment Content of Paste in Oil	11
Total Volatile Matter in Paste in Oil	12

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 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation²
D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
D 1135 Test Methods for Chemical Analysis of Blue Pigments³
E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁴

3. Significance and Use

3.1 This collection of test methods is used by pigment producers and paint manufacturers for process control, for product acceptance, and for research and development.

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.

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² *Annual Book of ASTM Standards*, Vols 05.01, 06.01, and 06.03.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ *Annual Book of ASTM Standards*, Vol 15.05.

LOSS ON IGNITION AND ASH

4. Procedure

4.1 Dry about 5 g of the sample at $105 \pm 2^\circ\text{C}$ for 2 h. Transfer about 1 g of the dried pigment, weighed to 0.1 mg, to a previously ignited, weighed porcelain crucible, and ignite at 900 to 1000°C for 20 min. Cool in a desiccator and weigh. Heat again for 10 min at 900 to 1000°C to check the loss in weight.

4.2 *Calculation*—Calculate the percent of loss on ignition, L , and of ash, A , as follows:

$$L = \frac{L_w}{S_1} \times 100$$

$$\left(A = \frac{W_a}{S_1} \times 100 \right)$$

where:

L_w = loss in weight on ignition, g,

A = ash, %,

W_a = weight of ash, g, and

S_1 = specimen weight, g.

MATTER SOLUBLE IN WATER

5. Procedure (Note 1)

5.1 Weigh about 10 g of the sample to 1 mg, and place in a 400-mL beaker. Add 100 mL of water (Note 2), boil for 5 min cool, and transfer quantitatively to a 250-mL volumetric flask. Dilute with water to 250 mL, mix, and allow to settle. Filter the supernatant liquid through a dry paper (Note 3) and discard the first 25 mL. Evaporate 100 mL of the clear filtrate to dryness in a weighed flatbottom dish, preferably in an oven at $105 \pm 2^\circ\text{C}$. Cool and weigh.

NOTE 1—This test method is not suitable for use with iron blue pigment. Use the conductivity method given in Test Methods D 1135.

NOTE 2—If the pigment is found to be strongly water-repellent, wet the sample with a small amount of alcohol or carry out a preliminary washing with chloroform.

NOTE 3—Water-dispersible pigments must be filtered by suitable means or the method may not be applicable. An asbestos-fiber filter paper pad is useful.

5.2 *Calculation*—Calculate the percent of matter soluble in water, M , as follows:

$$M = \frac{R_1 \times 2.5}{S_2} \times 100$$

where:

R_1 = residue weight, g, and

S_2 = specimen weight, g.

HYDROGEN ION CONCENTRATION (pH VALUE)

6. Procedure

6.1 Determine hydrogen ion concentration in accordance with either of the following methods:

6.1.1 *Electrometric Method (Preferred Procedure)*—Weigh about 5 g of the sample to 10 mg, place in a 150-mL beaker, and add 50 g of warm distilled water, freshly boiled to remove carbon dioxide. Mix well by means of a glass rod and cool to 25°C. Measure the pH of the mixture in accordance with Test Method E 70.

6.1.2 *Colorimetric Method*—Weigh about 10 g of the sample to the nearest 10 mg, place in a 250-mL glass-stoppered Erlenmeyer flask, and add 150 g of warm distilled water, freshly boiled to remove carbon dioxide. Stopper the flask and shake vigorously for about 1 min. Cool to 25°C. Let stand 1 h to allow the pigment to settle. Decant the clear liquid and determine its pH at once by means of a suitable indicator, using a block comparator or similar colorimetric method.

NOTE 4—A block comparator or equivalent device is necessary in order to compensate for the color of the extract.

6.2 *Report*—In reporting test results, state the particular method used.

ALKALINITY OR ACIDITY BY TITRATION

7. Reagents

7.1 *Methyl Orange Indicator Solution*—Dissolve 1 g of methyl orange in 1 L of reagent water.

7.2 *Sodium Hydroxide, Standard Solution (0.02 N)*.

7.3 *Sulfuric Acid, Standard Solution (0.02 N)*.

8. Procedure

8.1 Shake 20 g of the dry pigment with 200 mL of distilled water for 5 min, let settle, and filter through a dry filter paper into a dry beaker, discarding the first 10 mL of the filtrate. Transfer 100 mL of the clear filtrate to a flask, add 3 drops of methyl orange indicator solution, and titrate to the end point with 0.02 N sulfuric acid (H_2SO_4), if alkaline, or 0.02 N sodium hydroxide (NaOH) solution, if acid.

8.2 *Calculation*—Calculate the acidity or alkalinity, expressed as milligrams of NaOH equivalent to 1 g of the sample, as follows (1 mL of 0.020 N NaOH solution is equivalent to 0.8 mg of NaOH):

$$\text{Alkalinity or acidity} = \text{mL of NaOH or } H_2SO_4 \times 0.8$$

WATER CONTENT

9. Apparatus

9.1 *Distillation Apparatus*—A water distillation apparatus consisting of a 250-mL roundbottom flask, a straight-tube reflux condenser, and a graduated receiving trap similar to Fig. 1 of Test Method D 95.

9.2 *Heat Source* consisting of a gas burner and oil bath, or an electric heater of the enclosed element type.

10. Procedure (Notes 5 and 6)

10.1 Place 50 g of the sample in the flask and add 100 mL of toluene or of petroleum solvent having a boiling point between 110 and 120°C. Mix well. Add more solvent, if necessary, to cover the pigment.

10.2 Rinse the inner tube of the condenser with a little toluene or petroleum solvent just before starting the distillation, so as to wet the inner surface completely. Distill at a moderate rate until the volume of condensed water no longer increases (about 3 h). If any water is lodged in the condenser tube at the end of the test, wash it down with solvent or with a brush wetted with solvent.

10.3 *Calculation*—Calculate the weight percent of water, W , as follows (assuming that 1 mL of water weighs 1 g):

$$W = \frac{W_c}{50} \times 100$$

where W_c = water collected, mL.

NOTE 5—This test method is not suitable for pigments that release combined water under the conditions of the test.

NOTE 6—Test methods especially adapted for the determination of moisture in iron blue pigments are given in Test Methods D 1135.

NOTE 7—Test Methods designed for the determination of free water (as opposed to water of crystallization) in dry pigments are given in Test Method D 280.

PIGMENT CONTENT OF PASTE IN OIL

11. Procedure

11.1 Select a portion of the well-mixed sample that is free of skins. Weigh to 10 mg, about 15 g of the sample in a weighed 60-mL centrifuge tube. Add about 30 mL of petroleum ether, mix thoroughly with a glass rod, and wash the rod with more petroleum ether (Note 8). Centrifuge at a moderate speed until well settled. Decant the clear supernatant liquid, and repeat the extraction twice with 40 mL of petroleum ether and once with 40 mL of ethyl ether. After decanting the ether, set the tube on top of a warm oven for 10 min and then in an oven at $105 \pm 2^\circ C$ for 2 h. Cool and weigh.

NOTE 8—Other solvents or solvent mixtures may be used, provided that they leave no residue on evaporation.

11.2 *Calculation*—Calculate the percent pigment content, P , as follows:

$$P = \frac{R_2}{S_3} \times 100$$

where:

R_2 = residue weight in centrifuge tube, g, and

S_3 = specimen weight, g.

TOTAL VOLATILE MATTER IN PASTE IN OIL

12. Procedure

12.1 Weigh to 1 mg a flatbottom dish about 80 mm in diameter containing a piece of stiff wire 100 mm long. Leave the wire in the dish throughout the test. Place about 4 g of the sample, weighed to 1 mg in the dish. Spread the specimen over the bottom by means of the wire and heat for $2\frac{1}{2}$ h in an oven at $105 \pm 2^\circ C$. If a skin forms, break it up at intervals during the heating by means of the wire. Cool the dish and contents in a desiccator and weigh. Heat again for 30 min to check the loss in weight.

12.2 *Calculation*—Calculate the percent volatile matter in sample, V , as follows:

$$V = \frac{L}{S_4} \times 100$$

where:

L = loss in weight on heating, g, and

S_4 = specimen weight, g.

PRECISION AND BIAS

13. Precision and Bias

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

13.2 *Bias*—No bias has been determined for this test method.

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Standard Test Method for Sieve Analysis of Glass Spheres¹

This standard is issued under the fixed designation D 1214; the number immediately following the designation indicates the year of original adoption 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 sieve analysis of glass spheres used for retroreflective pavements markings and industrial uses.

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 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis²

D 2013 Method for Preparing Coal Samples for Analysis²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

3. Summary of Test Method

3.1 The spheres are hand-sieved through standard sieves, starting with the sieve with the largest opening specified and progressing successively through the specified sieves in the order of decreasing size of opening, and computing the weight of glass spheres and the percent passing each of the sieves.

4. Significance and Use

4.1 The size or gradation of glass spheres is one measurable aspect of performance as a retroreflective media. The function of this test is to measure the size of glass spheres and to determine compliance with applicable specifications.

NOTE 1—This method has been used in other industrial areas outside the intended scope of this test method.

5. Apparatus

5.1 *Balance*, sensitive to 50 mg.

5.2 *Sieves*, 8 in. (200 mm) in diameter, conforming to Specification E 11, and including such sieves as may be required by the specifications for the glass spheres.

5.3 *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 D01.44 on Traffic Coatings.

Current edition approved May 26, 1989. Published July 1989. Originally published as D 1214 - 52T. Last previous edition D 1214 - 58(1983) ϵ 1.

² *Annual Book of ASTM Standards*, Vol 05.05.

³ *Annual Book of ASTM Standards*, Vols 05.05 and 14.02.

6. Samples

6.1 By quartering or riffle sampling (Note 2), select a representative sample from the material to be tested. Take at least two representative samples of approximately 500 g each from separate packages from each shipment in the ratio of two samples for each 10 000 lb (5000 kg) or fraction thereof. Approximately 50 g of dry glass spheres are required for each test. This specimen is also selected by quartering or riffing.

NOTE 2—The quartering procedure for reducing bulk samples, to obtain representative test samples of suitable size, is described and illustrated in Methods D 346. Various types of riffle samplers are illustrated in Method D 2013.

7. Procedure

7.1 Hand Sieving:

7.1.1 Dry the specimen to substantially constant weight at a temperature of 105 to 110 °C.

7.1.2 Weigh 50 g of the dried glass spheres to the nearest 0.1 g and place on the sieve with the largest opening in the series specified for the test, which shall be thoroughly dry. Hold the sieve, with pan and cover attached, in one hand in a slightly inclined position so that the specimen will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the upstroke. Turn the sieve every 25 strokes about one sixth of a revolution in the same direction. Continue the operation until not more than 0.05 g passes through the sieve in 1 min of continuous sieving. Each time, before weighing the material passing through the sieve, tap the side of the sieve with the brush handle in order to remove any material adhering to the wire cloth.

7.1.3 When the sieving has been finished, remove the cover of the sieve and carefully remove the residue remaining on the sieve to a tared container. Invert the sieve over a piece of glazed white paper and clean the wire cloth by brushing the underside. Add the material thus removed from the wire cloth to the residue removed from the sieve.

7.1.4 Weigh the portion of the specimen retained on the sieve to the nearest 0.1 g. Place the material passing through the largest sieve on the sieve with the next smaller opening for the series selected for the sieve analysis. Continue sieving in a similar manner, using successively each of the selected series of sieves in the order of decreasing size of opening, and recording the weight of that portion of the specimen retained on each sieve.

7.1.5 Washers, slugs, or shot shall not be used on the sieves.

7.2 Machine Sieving:

7.2.1 Mechanical sieving devices may be used but the glass spheres shall not be rejected if they meet the specification requirements when tested by the hand-sieving method

described in 7.1. When mechanical sieving devices are used, their thoroughness of sieving shall be tested by using the same method for comparison.

8. Calculation

8.1 Calculate the weight of material and the percent of the specimen passing each of the sieves.

9. Report

9.1 Report the following information:

9.1.1 Results of the sieve analysis reported as the total percent passing each sieve, expressed to the nearest 0.5 %, and
9.1.2 The method of sieving used.

10. Precision

10.1 A round robin is currently underway in order to generate a precision statement.

11. Keywords

11.1 sieve; glass spheres

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, 1910 Race St., Philadelphia, PA 19103.



Standard Test Methods for Nonvolatile Content of Resin Solutions¹

This standard is issued under the fixed designation D 1259; the number immediately following the designation indicates the year of original adoption 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—Section 12 was added editorially in May 1990.

1. Scope

1.1 These test methods cover the determination of non-volatile content of solutions of resins in volatile organic solvents.

1.2 Two test methods are included as follows:

1.2.1 *Test Method A*—For solutions of non-heat-reactive resins. These solutions contain resins that remain stable and release the solvent under conditions of the test. Examples are ester gums and alkyls.

1.2.2 *Test Method B*—For two types of solutions:

1.2.2.1 Solutions of heat-reactive resins. These solutions contain resins that undergo condensation or other reactions under the influence of heat. Examples include the formaldehyde reaction products of urea, melamine, and phenols.

1.2.2.2 Solutions that release solvent slowly. Examples include epoxy resin solutions.

1.3 Test Methods A and B differ primarily in the drying times and types of oven used.

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. Summary of Test Methods

2.1 In both test methods, a weighed specimen of resin solution is spread under pressure between two weighed sheets of aluminum or tin foil. The coated foil sheets are separated and then dried. The weight of residue is determined and the nonvolatile content is calculated. The test method is unique in that it provides for drying of a very thin film of resin, thus minimizing chances for volatiles to be trapped and held during the heating operation.

2.2 Either a gravity-convection or a forced-ventilation oven and a 30-min heating period at 105°C are used in Test Method A.

2.3 A forced-ventilation oven and a 2-h heating period at 105°C are used in Test Method B.

3. Significance and Use

3.1 The nonvolatile content of resin solutions is useful to

coatings producers and users for the determination of the total solids available for film formation and for the estimation of the volatile organic content.

TEST METHOD A—NON-HEAT-REACTIVE RESIN SOLUTIONS

4. Apparatus

4.1 *Ovens:*

4.1.1 Gravity-convection type, maintained at $105 \pm 2^\circ\text{C}$, with vents open.

4.1.2 Forced-ventilation type, maintained at $105 \pm 2^\circ\text{C}$. For ovens with adjustable air flow rate, set the control damper at 50 %.

4.2 *Aluminum or Tin Foil*,² from 0.0015 to 0.0020 in. (38 to 50 μm) in thickness. Either one piece 6 by 12 in. (150 by 300 mm), or two 6 by 6-in. (150 by 150-mm) pieces may be used. The foil must be perfectly smooth; if it becomes wrinkled during the initial handling, roll smooth as directed in 5.2.

4.3 *Plate Glass*—Two pieces about $\frac{3}{16}$ in. (5 mm) thick; one piece $5\frac{1}{2}$ by $5\frac{1}{2}$ in. (140 by 140 mm) and one piece 7 by 7 in. (180 by 180 mm).

4.4 *Device for Weighing Specimens*³—Apparatus that will prevent loss of volatile matter during the weighing operation such as any of the following, or equivalent:

4.4.1 *Syringe*, Luer, 2 or 5-mL capacity;

4.4.2 *Weighing Buret*, Smith, 10-mL capacity, or

4.4.3 *Bulb Pipet*, dropping, with 50-mL Erlenmeyer flask.

4.5 *Roller, for Smoothing Foil*—Use a ground and polished cylinder, preferably stainless steel, approximately 7 in. (180 mm) long and 2 in. (50 mm) in diameter.

4.6 *Foil Trays*, two types as follows:

4.6.1 Trays measuring $6\frac{1}{2}$ by 12 in. (165 by 300 mm), for use with 6 by 12-in. foil, constructed from No. 22-gage (0.6-mm) aluminum sheet in accordance with dimensions shown in Fig. 1. Several trays may be stacked in the oven to permit running several specimens simultaneously.

4.6.2 Trays measuring $6\frac{1}{2}$ by $6\frac{1}{2}$ in. (165 by 165 mm),

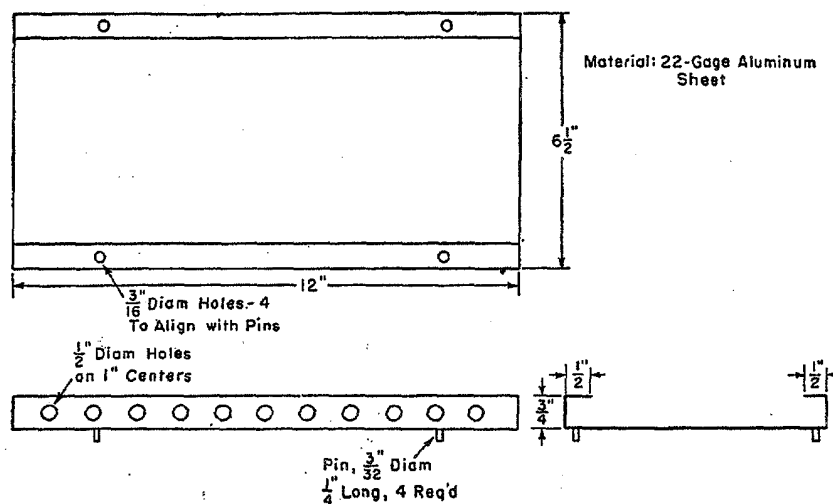
¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved Nov. 29, 1985. Published January 1986. Originally published as D 1259 - 53. Last previous edition D 1259 - 61 (1980)^{e1}.

² Aluminum foil available from Thomas Scientific Co., P.O. Box 99, Swedesboro, NJ 08085; or from Sargent and Welch Scientific Co., 7300 North Linder Ave., Skokie, IL 60077 has been found satisfactory for this purpose.

Tin foil available from J. T. Baker Co., North Broad St., North Philipsburg, NJ 08865 has been found satisfactory for this purpose.

³ A Smith weighing buret is available from Ace Glass Co., 1430 Northwest Blvd., Vineland, NJ 08360. The bulb pipet is available from Thomas Scientific Co. or from Fisher Scientific Co.



Note—Millimetre dimensions appear in section on Apparatus.

FIG. 1 Tray for 6 by 12-in. (150 by 300-mm) Foil

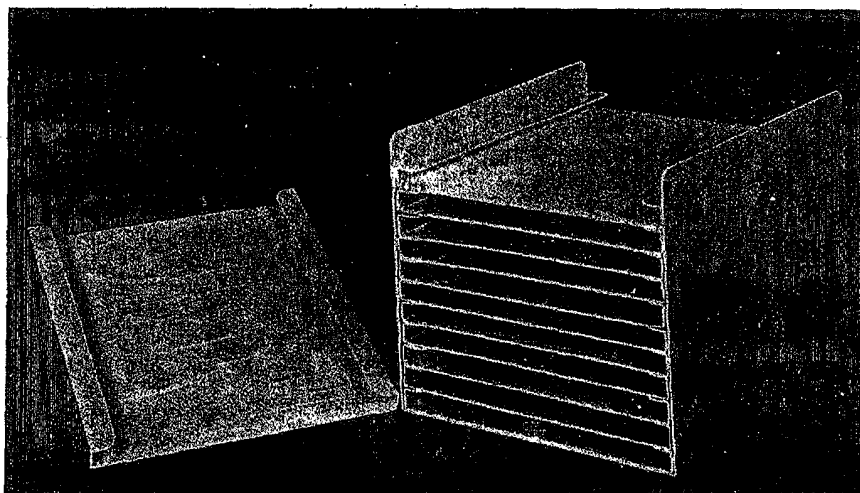


FIG. 2 Trays and Holder for 6 by 6-in. (150 by 150-mm) Foil

for use with 6 by 6-in. foil, with holder, shall be constructed from No. 22-gage aluminum sheet, as shown in Fig. 2.

5. Procedure

5.1 Use the following procedure with the 6 by 12-in. (150 by 300-mm) foil sheets and the 6 1/2 by 12-in. (165 by 300-mm) trays. Alternatively two 6 by 6-in. (150 by 150-mm) foil sheets may be used in a similar manner with the 6 1/2 by 6 1/2-in. (165 by 165-mm) trays. In handling the foil, avoid wrinkling or creasing the sheets until after the specimen has been dried. Sheets may be rolled for convenience of handling and making the initial weighing, but must be kept smooth throughout the pressing and drying operations.

5.2 Weigh the foil to 0.1 mg. Open and place half the foil, with the shiny side up, on the 7 by 7-in. (180 by 180-mm)

glass plate. If necessary, roll smooth with the metal roller. By means of the weighing device, weigh by difference a 0.9 to 1.1-g specimen of the resin solution, to 0.1 mg. Place the specimen on the center of that area of the foil covering the glass plate. Place the other half of the foil on top. Cover the foil with the second glass plate, centering the glass on the foil, and press down sufficiently to cause the specimen to spread uniformly into a thin film, about 3 in. (75 mm) in diameter. The pressure that must be exerted depends on the viscosity of the sample. In case a specimen of low viscosity should extend beyond the edge of the foil, repeat the determination, allowing a few minutes for a portion of the solvent to evaporate from the weighed specimen before covering and pressing it.

5.3 After pressing, open the foil to its full length and place it in the foil tray. Place the tray in either a gravity-convection

or a forced-ventilation oven at $105 \pm 2^\circ\text{C}$ for 30 min.

5.4 Remove the tray from the oven and then carefully remove the foil sheet from the tray. Return the dried film surfaces to the face-to-face position. While the foil is still warm, fold the edges together to enclose completely the dried film. Without undue delay, weigh to 0.1 mg.

6. Calculation

6.1 Calculate the percent nonvolatile content, C , as follows:

$$C = [(A - B) \times 100]/S$$

where:

A = weight of foil plus dried solids, g,

B = weight of foil, g, and

S = weight of sample taken, g.

7. Precision and Bias

7.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

7.1.1 *Repeatability*—The difference between two results, each the mean of duplicate determinations obtained by the same analyst, is normally about 0.2 %, absolute. Two such results should be considered suspect if they differ by more than 0.5 %, absolute.

7.1.2 *Reproducibility*—The difference between two results, each the mean of duplicate determinations, obtained by analysts in different laboratories is normally about 0.4 %, absolute. Two such results should be considered suspect if they differ by more than 1.0 %, absolute.

7.1.3 No bias has been determined for this test method.

TEST METHOD B—HEAT-REACTIVE RESIN SOLUTIONS AND SOLUTIONS THAT RELEASE SOLVENT SLOWLY

8. Apparatus

8.1 *Oven*—Forced-ventilation type, maintained at $105 \pm 2^\circ\text{C}$. For ovens with adjustable air flow rate, set the control damper at 50 %.

8.2 The remainder of the apparatus is identical with that given in 4.2 to 4.6.

9. Procedure

9.1 Weigh the specimen of resin solution and press it

between two sheets of foil as described in 5.1 and 5.2.

9.2 After pressing, open the foil to its full length and place it in the foil tray. Then place the tray in a forced-ventilation oven at $105 \pm 2^\circ\text{C}$ for 2 h. When using $6\frac{1}{2}$ by $6\frac{1}{2}$ -in. trays and holder, place the assembly in the oven with the open ends perpendicular to the direction of air flow.

9.3 Complete the determination as described in 5.4.

10. Calculation

10.1 Calculate the nonvolatile content as described in Section 6.

11. Precision and Bias

11.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level.

11.1.1 *For Heat-Reactive Resin Solutions:*

11.1.1.1 *Repeatability*—The difference between two results, each the mean of duplicate determinations obtained by the same analyst, is normally about 0.3 %, absolute. Two such results should be considered suspect if they differ by more than 0.7 %, absolute.

11.1.1.2 *Reproducibility*—The difference between two results, each the mean of duplicate determinations obtained by analysts in different laboratories, is normally about 0.7 %, absolute. Two such results should be considered suspect if they differ by more than 1.7 %, absolute.

11.1.1.3 No bias has been determined for this test method.

11.1.2 *For Solutions that Release Solvent Slowly:*

11.1.2.1 *Repeatability*—The average difference between two results each the average of duplicate determinations, obtained by the same analyst is normally about 0.1 %, absolute. Two such results should be considered suspect if they differ by more than 0.3 %, absolute.

11.1.2.2 *Reproducibility*—The average difference between two results obtained by analysts in different laboratories will approximate 0.2 %. Two such results should be considered suspect if they differ by more than 0.5 %, absolute.

11.1.2.3 No bias has been determined for this test method.

12. Keywords

12.1 foil method; heat-reactive; non-heat-reactive; non-volatile; resin solution

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Designation: D 1301 - 91

Standard Test Methods for Chemical Analysis of White Lead Pigments¹

This standard is issued under the fixed designation D 1301; the number immediately following the designation indicates the year of original adoption 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 Methods 7041, 7051 of Federal Test 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 procedures for the chemical analysis of basic carbonate white lead and basic sulfate white lead.

NOTE 1—If it is necessary to separate these pigments from others, refer to Methods D 215.

1.2 The analytical procedures appear in the following order:

	Section
Preparation of Sample	6
<i>Basic Carbonate White Lead:</i>	
Small Amounts of Iron	7
Total Lead	8
Moisture and Other Volatile Matter	9
Carbon Dioxide (Evolution Method)	10
Carbon Dioxide and Combined Water (Combustion Method)	11
Lead Carbonate	12
Total Matter Insoluble in Acetic Acid	13
Total Matter Insoluble in Acid Ammonium Acetate	14
Total Impurities Other Than Moisture	15
Coarse Particles	16
<i>Basic Sulfate White Lead:</i>	
Small Amounts of Iron	17
Total Lead	18
Moisture and Other Volatile Matter	19
Total Sulfate	20
Zinc Oxide	21
Basic Lead Oxide	22
Total Impurities	23
Coarse Particles	24

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 25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime²
 D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³

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 Feb. 22, 1991. Published April 1991. Originally published as D 1301 - 53 T. Last previous edition D 1301 - 86.

² *Annual Book of ASTM Standards*, Vol 04.01.

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

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments⁴

D 1193 Specification for Reagent Water⁵

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints⁶

D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints⁶

D 3280 Test Methods for Analysis of White Zinc Pigments⁴

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁷

3. Significance and Use

3.1 These test methods are suitable for determining the level of purity and for determining the levels of various impurities. They may be used to establish compliance with specification requirements.

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 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

4.3 Concentration of Reagents:

4.3.1 *Concentrated Acids and Ammonium Hydroxide*—When acids and ammonium hydroxide are specified by name or chemical formula only, it shall be understood that concentrated reagents of the following specific gravities or concentrations are intended:

Acetic acid, CH ₃ COOH	99.5 %
Hydrochloric acid, HCl	sp gr 1.19
Hydrofluoric acid, HF	48 %
Nitric acid, HNO ₃	sp gr 1.42
Sulfuric acid, H ₂ SO ₄	sp gr 1.84
Ammonium hydroxide, NH ₄ OH	sp gr 0.90

⁴ *Annual Book of ASTM Standards*, Vol 06.02.

⁵ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 06.01.

⁷ *Annual Book of ASTM Standards*, Vol 14.02.

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."

The desired specific gravities or concentrations of all other concentrated acids are stated whenever they are specified. **Warning**—See Section 5.

4.3.2 *Diluted Acids and Ammonium Hydroxide*—Concentrations of diluted acids and ammonium hydroxide, except when standardized, are specified as a ratio stating the number of volumes of concentrated reagent to be diluted with a given number of volumes of water, as in the following example: HCl (1 + 99) means 1 volume of concentrated HCl (sp gr 1.19) diluted with 99 volumes of water.

5. Hazards

5.1 The concentrated acids bases and other reagents used in these test methods can be dangerous. Check their Material Safety Data Sheets, (MSDS) before use.

6. Preparation of Sample

6.1 Grind dry pigments, if lumpy or not finely ground, to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. Mix the sample in all cases thoroughly and comminute before taking specimens for analysis.

6.2 In cases of pastes in oil, extract the oil from the pigment as described in Test Methods D 2371 or D 2372, but without straining.

6.3 Dry pigments separated from paints or pastes in oil in an oven at 203 to 210°F (95 to 98°C) for 2 h, grind to a fine powder, pass through a No. 80 (180-μm) sieve (Note 2) to remove skins, and mix thoroughly. Such pigments, after weighing, should be moistened with a little ethyl alcohol (95 %) before adding reagents for analysis.

NOTE 2—Detailed requirements for this sieve are given in Specification E 11.

6.4 Preserve all samples in stoppered bottles or containers.

BASIC CARBONATE WHITE LEAD

7. Small Amounts of Iron

7.1 Reagents:

7.1.1 *Ammonium Hydroxide* (sp gr 0.90). **Warning**—See 5.1.

7.1.2 *Hydrofluoric Acid* (48 %). **Warning**—See 5.1.

7.1.3 *Nitric Acid* (sp gr 1.42). **Warning**—See 5.1.

7.1.4 *Sulfuric Acid* (sp gr 1.84). **Warning**—See 5.1.

7.2 Procedure:

7.2.1 Weigh to 10 mg about 1 g of specimen into a 400-mL beaker. Treat the sample with 10 mL of HNO₃ (1 + 1) and dilute to about 200 mL with water. If insoluble matter remains following treatment with HNO₃ and dilution, filter and wash the residue with hot water until lead free. Evaporate the filtrate and washings to about 200 mL. Add 20 mL of H₂SO₄ (1 + 1) to precipitate the bulk of the lead (it is unnecessary to evaporate down). Cool, filter, and wash with diluted H₂SO₄ (1 + 99). Save the precipitate for determination of total lead (Section 8).

7.2.2 Ignite the HNO₃-insoluble matter and treat with HF and H₂SO₄. Bring into solution, filter (any precipitate is probably BaSO₄), and add to the PbSO₄ filtrate.

7.2.3 Colorimetrically determine iron in the combined

filtrates by the thiocyanate method,⁹ using the same amounts of reagents in preparing the reference standards. If copper is present in the filtrate, as shown by the characteristic blue-green or yellow color, remove it by precipitating the iron with NH₄OH, filtering, washing, redissolving the Fe(OH)₃ in 10 mL of HNO₃ (1 + 1), and diluting to about 200 mL before proceeding with the thiocyanate method.

8. Total Lead

8.1 Apparatus:

8.1.1 *Gooch Crucible*, prepared prior to use.

8.2 Reagents:

8.2.1 *Acetic Acid* (glacial)—**Warning**—See 5.1.

8.2.2 *Ammonium Hydroxide* (sp gr 0.90)—**Warning**—See 5.1.

8.2.3 *Ethyl Alcohol* (95 volume %)—**Warning**—See 5.1.

8.2.4 *Potassium Dichromate Solution* (100 g K₂Cr₂O₇/L)—**Warning**—See 5.1.

8.3 Procedure:

8.3.1 Ignite the PbSO₄ precipitate and filter paper from 7.2.1 at or below 1020°F (550°C), and transfer the residue to a 400-mL beaker. (If preferred, a new 1-g specimen of pigment may be weighed to 10 mg into a 400-mL beaker. Proceed to 8.3.2.)

8.3.2 Moisten with water and add 5 mL of glacial acetic acid. Warm to dissolve the material and dilute to about 200 mL with water. Neutralize the solution with NH₄OH and then make slightly acid with acetic acid, adding about 3 mL excess. Filter off any insoluble residue and wash thoroughly with hot water.

8.3.3 Unite the filtrate and washings, heat to boiling, and add 15 mL of K₂Cr₂O₇ solution. Stir and heat until the yellow precipitate assumes an orange color. Let settle and filter on a weighed Gooch crucible. Wash by decantation with hot water until the washings are colorless. Finally transfer all the precipitate from the beaker to the crucible and wash with ethyl alcohol (95 %). Dry at 220 ± 4°F (105 ± 2°C) for 1 h. Cool in a desiccator and weigh as PbCrO₄.

8.4 Calculation:

$$\text{PbO, \%} = (P \times 0.691/S) \times 100$$

where:

P = PbCrO₄ precipitate, g,

S = specimen, g, and

0.691 = PbO/PbCrO₄ = 223.19/323.18.

9. Moisture and Other Volatile Matter

9.1 *Procedure*—Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

10. Carbon Dioxide (Evolution Method)

10.1 *Apparatus*—Knorr type of CO₂ evolution apparatus with dropping funnel, condenser, and suitable purifying train.

NOTE 3—A description of a suitable purifying train, is found in the Carbon Dioxide Standard Method section of Methods C 25.

10.2 Reagent:

⁹ Described in Scott, *Standard Methods of Chemical Analysis*, Fifth Edition, D. Van Nostrand Co., New York, NY, 1939, p. 486.

10.2.1 Nitric Acid (1 + 19).

10.3 Procedure—Transfer about 2 g of the sample, weighed to 10 mg, to a clean, dry evolution flask. Connect the evolution flask to the absorption train, which previously has been flushed free of any CO₂, and add 100 mL of HNO₃ (1 + 19) through a separatory funnel. When all of the HNO₃ has been introduced into the flask, close the stopcock from the separatory funnel. Heat the solution in the flask to gentle rolling and boil for 5 min. Turn off the heat and aspirate CO₂ free air through the system for 20 min. Remove the absorbing tube from the system, seal, cool in a desiccator, and weigh. The increase in weight is CO₂.

10.4 Calculation—Calculate the percent of carbon dioxide as follows:

$$\text{CO}_2, \% = (C_1/S_1) \times 100$$

where:

C_1 = CO₂, g, and

S_1 = specimen, g.

11. Carbon Dioxide and Combined Water (Combustion Method)

11.1 Apparatus—Combustion Train, consisting of the following parts connected in the order specified; tank of purified compressed nitrogen, purifying jars including a CO₂ absorption jar, drying tube, combustion tube, tube furnace provided with suitable controls to maintain the temperature from 840 to 1020°F (450 to 550°C), absorption bulb for water, and an absorption bulb for CO₂.

11.2 Procedure:

11.2.1 Heat the furnace, without the combustion tube, from 840 to 1022°F (450 to 550°C). Connect the combustion tube beside the furnace, connect the absorption tubes to the nitrogen supply, and pass a slow stream of nitrogen (about 30 mL/min) through them, to clear out any residual moisture and CO₂. Accurately weigh the absorption bulbs and reconnect them in the train. Transfer 1 g of the specimen, weighed to 10 mg, to a combustion boat that has been previously ignited and cooled.

11.2.2 With the nitrogen still flowing, disconnect the train and place the boat containing the specimen in the middle of the tube with the aid of a hooked wire. Flush the combustion tube thoroughly with nitrogen and reconnect with the train. Place the tube in the furnace.

11.2.3 Continue the combustion for 30 min, or until the water that condenses in the inlet arm of the first absorption bulb has been completely swept into the bulb. Disconnect the absorption bulbs from the combustion tube, after closing all stopcocks, place in a desiccator to cool, and then weigh.

11.3 Calculation:

$$\text{Carbon dioxide, \%} = 100 \times C_1$$

$$\text{Combined water, \%} = 100 W_1 - M$$

$$\text{Combined water as Pb(OH)}_2, \% = (100 W_1 - M) \times 13.39$$

where:

C_1 = CO₂, g,

W_1 = total water, g,

M = free moisture, %, and

13.39 = Pb(OH)₂/H₂O = 241.20/18.015.

12. Lead Carbonate

12.1 Calculation—Calculate the percent of PbCO₃ from the CO₂ content, as follows:

$$\text{PbCO}_3, \% = C_1 \times 6.071/S_2 \times 100$$

where:

C_1 = CO₂ (10.4 or 11.3), g,

S_2 = specimen weight used in the CO₂ determination, g, and

$$6.071 = \text{PbCO}_3/\text{CO}_2 = 267.20/44.01.$$

13. Total Matter Insoluble in Acetic Acid

13.1 Apparatus—Gooch Crucible, prepared and weighed prior to use.

13.2 Reagent—Acetic Acid (3 + 2).

13.3 Procedure—Transfer 10 g of the sample, weighed to 10 mg, to a 250-mL beaker and add 40 mL of acetic acid (2 + 3). Heat until solution is complete and filter through a previously prepared and weighed Gooch crucible. Wash thoroughly with hot water; dry at 220 ± 4°F (105 ± 2°C) for 1 h, cool, and weigh.

13.4 Calculation—Calculate the percent of total matter insoluble in acetic acid as:

$$(R/S_3) \times 100$$

where:

R = residue, g, and

S_3 = specimen, g.

14. Total Matter Insoluble in Acid Ammonium Acetate

14.1 Apparatus—Gooch Crucible, prepared and weighed prior to use.

14.2 Reagent—Acid Ammonium Acetate Solution—Mix 150 mL of acetic acid (3 + 2) with 100 mL of water and then with 95 mL of NH₄OH (sp gr 0.90).

14.3 Procedure—Transfer about 10 g of the sample, weighed to 10 mg, to a 250-mL beaker. Add 40 mL of acid ammonium acetate solution and heat until solution is complete. Filter through a previously prepared and weighed Gooch crucible and wash thoroughly with hot water. Dry at 105 to 110°C for 1 h, cool, and weigh.

14.4 Calculation—Calculate the percent of total matter insoluble in acid ammonium acetate as:

$$(R/S_4) \times 100$$

where:

R_1 = residue, g, and

S_4 = specimen, g.

15. Total Impurities Other Than Moisture

15.1 Calculations:

15.1.1 Calculate the percent of total impurities other than moisture as:

$$100 - (L + C + H + M)$$

where:

L = PbO % (8.4),

C = CO₂ % (10.4 or 11.3),

H = combined water, % (11.3), and

M = free moisture, % (9.1).

15.1.2 In the case of extracted pigments where direct determination of combined water cannot be made conveniently, calculate the impurities other than moisture as: 100

$(L + 1.205 D + M)$ where L , D , are as defined in 15.1.1 and $1.025 = 2 \text{ CO}_2 + \text{H}_2\text{O} / 2 \text{ CO}_2 = 106.035/88.02$.

16. Coarse Particles

16.1 *Procedure*—Determine coarse particles in accordance with Test Methods D 185.

BASIC SULFATE WHITE LEAD

17. Small Amounts of Iron

17.1 *Procedure*—Determine small amounts of iron in accordance with Section 7.

18. Total Lead

18.1 *Procedure*—Determine total lead in accordance with Section 8.

19. Moisture and Other Volatile Matter

19.1 *Procedure*—Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

20. Total Sulfate

20.1 *Apparatus*—Gooch Crucible, ignited and weighed prior to use.

20.2 Reagents:

20.2.1 *Barium Chloride Solution* (100 g BaCl_2/L)—Dissolve 117 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in water and dilute to 1 L.

20.2.2 *Bromine Water* (saturated).

20.2.3 *Hydrochloric Acid* (1+1).

20.2.4 *Sodium Chloride*.

20.2.5 *Sodium Carbonate Solution* (saturated).

20.3 Procedure:

20.3.1 Transfer 0.625 g of the sample to a 400-mL beaker. Add 2 g of NaCl , 3 to 4 mL of bromine water, and 25 mL of HCl (1+1), and heat over a low flame until solution is complete. Dilute to 75 mL with water and bring to boiling to expel the bromine. Cool somewhat, but not enough for the PbCl_2 to separate, and then cautiously add, by means of a pipet, Na_2CO_3 solution until decidedly alkaline. Bring to boiling and transfer to a 250-mL volumetric flask. Cool to room temperature, dilute to the mark, and mix. Filter through a dry paper, discarding the first 15 to 20 mL of filtrate. Measure exactly 200 mL in a volumetric flask and transfer to a 600-mL beaker. The test solution will now be equivalent to 0.5 g of the original specimen.

20.3.2 Carefully add HCl (sp gr 1.19) from a pipet to the alkaline solution until the solution is neutral, and add 0.4 mL excess for each 100 mL of solution. Bring to boiling to expel the CO_2 and then add to the boiling solution, drop by drop, 20 to 25 mL of a BaCl_2 solution. Allow to stand in a

warm place for at least 2 h. Filter on a previously ignited and weighed Gooch crucible or a fine-textured filter paper and wash with hot water. Dry and ignite. Cool, and weigh as BaSO_4 .

20.4 Calculations:

$$\text{Total sulfate as } \text{SO}_3, \% = 68.6 \times P$$

$$\text{Total sulfate as } \text{PbSO}_4, \% = 259.8 \times P$$

where:

P = BaSO_4 precipitate, g.

$$68.6 = \text{SO}_3/\text{BaSO}_4 \times 100/0.5 = 80.06/233.40 \times 200$$

$$259.8 = \text{PbSO}_4/\text{BaSO}_4 \times 100/0.5 = 303.25/233.40$$

21. Zinc Oxide

21.1 *Procedure*—Determine zinc oxide in accordance with Methods D 3280.

22. Basic Lead Oxide

22.1 *Calculation*—Calculate the percent of basic lead oxide as follows:

$$\text{Basic PbO}, \% = L = S_{u_1}(0.736)$$

where:

L = total lead as PbO , % (Section 18),

S_{u_1} = total sulfate as PbSO_4 , % (20.4), and

$$0.736 = \text{PbO}/\text{PbSO}_4 = 233.19/303.25$$

23. Total Impurities

23.1 *Calculation*—Calculate the percent of total impurities as follows:

$$\text{Total impurities}, \% = 100 - (L + S_{u_2} + Z)$$

where:

L = total lead as PbO , % (Section 18),

S_{u_2} = total sulfate as SO_3 , % (20.4), and

Z = zinc oxide, % (Section 21).

24. Coarse Particles

24.1 *Procedure*—Determine coarse particles in accordance with Test Methods D 185.

PRECISION

25. Precision

25.1 Data are not available to determine the precision of these test methods. There are no plans at present to obtain such data. The test methods have been in use for many years and are considered acceptable.

26. Keywords

26.1 ammonium acetate soluble, pigment; basic carbonate, white lead; basic lead oxide pigment; basic sulfate white lead; carbon dioxide, pigment; chemical analysis, white lead pigment; lead carbonate pigment

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Standard Test Method for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric)¹

This standard is issued under the fixed designation D 1306; the number immediately following the designation indicates the year of original adoption 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 7022 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.

Scope

1.1 This test method covers the gravimetric determination of phthalic anhydride in alkyd resins and esters that contain dibasic acids such as maleic, fumaric, adipic, and sebacic, which would interfere if Test Method D 563 was 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 563 Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions²
- D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 The specimen is saponified with alcoholic potassium hydroxide and benzene to precipitate quantitatively the potassium salt of phthalic acid as $C_6H_4(COOH)_2 \cdot C_6H_5OH$. Interfering substances are eliminated by dissolving the precipitate in water, adjusting the pH of the solution to 2.5 with nitric acid, and filtering. Phthalic acid is then precipitated as nonstoichiometric lead phthalate and calculated to phthalic anhydride, using a factor obtained when compositions of known purity were analyzed similarly.

4. Significance and Use

4.1 The phthalic anhydride content of alkyd resins controls the properties of the final film.

5. Apparatus

5.1 *Flask and Condenser*—A 500-mL Erlenmeyer flask fitted with an air-cooled glass reflux condenser 30 in. (760 mm) in length. The connection between the flask and condenser shall be a standard-taper 24/40 ground-glass joint.

5.2 *Water Bath*.

5.3 *Guard Tube*, filled with soda lime.

5.4 *Fritted-Glass Filter Crucible*, medium porosity, of 30-mL capacity.

5.5 *Filter Flasks*, suction-type.

5.6 *Crucible Holder*.

5.7 *Oven*, of gravity convection type.

5.8 *Desiccator*, containing concentrated H_2SO_4 (sp. gr 1.84) as the desiccant.

5.9 *Flash Filtrator*.

5.10 *pH Test Assembly*.

5.11 *Volumetric Flask*, 100-mL.

5.12 *Erlenmeyer Flask*, 250-mL, wide-mouth, with glass stopper not smaller than a No. 27.

5.13 *Delivery Pipet*, 2-mL.

6. Reagents

6.1 *Purity of Reagent*—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 as defined by Type II of Specification D 1193.

6.3 *Acetic Acid, Glacial*.

6.4 *Alcohol-Benzene Wash Solution*—Mix 1 volume of absolute ethyl alcohol (Note 1) with 3 volumes of benzene.

NOTE 1—The alcohol may be denatured Formula 2-B, but must be anhydrous.

6.5 *Benzene* (anhydrous).

6.6 *Ether* (anhydrous).

6.7 *Lead Acetate Solution*—Dissolve 25 g of lead acetate trihydrate in glacial acetic acid and dilute to 100-mL volume with acetic acid.

6.8 *Methanol* (anhydrous).

6.9 *Nitric Acid* (1+3)—Mix 1 volume of concentrated nitric acid (HNO_3) (sp gr 1.42) with 3 volumes of water.

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.

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² Annual Book of ASTM Standards, Vol 06.02.

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

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."

6.10 *Potassium Hydroxide Alcoholic, Solution* (66 g/L)—Dissolve 66 g of potassium hydroxide (KOH) in 1 L of absolute ethyl alcohol (Note 1). Allow the solution to stand overnight protected against carbon dioxide (CO₂) absorption. Filter just before use.

7. Procedure

7.1 Weigh by difference, from a closed container into the 500-mL Erlenmeyer flask, a sample of resin or resin solution sufficient to yield from 0.8 to 1.2 g of potassium alcohol phthalate. Add 150 mL of benzene, warming slightly on the steam bath if necessary, to effect solution. Add 60 mL of alcoholic KOH solution, and attach the condenser. Place the flask in a water bath to a depth approximately equal to that of the contents of the flask. Warm the bath, maintaining a temperature of 40°C for 1 h, then gradually raise the temperature until the alcoholic solution boils gently. Reflux for 1½ h.

7.2 Remove the flask from the bath and wash down the inside of the condenser with a few millilitres of alcohol-benzene wash solution. Remove the condenser, cap the flask with the soda-lime guard tube, and cool by means of running water or an ice bath.

7.3 When cool, filter immediately and as rapidly as possible, through a fritted-glass crucible that previously has been tared, using the alcohol-benzene wash solution for transferring the precipitate and washing the reaction flask. Wash the precipitate with successive portions of alcohol-benzene wash solution until a few millilitres of washings collected in a second suction flask are no longer alkaline to phenolphthalein. (Normally about 75 mL of wash solution are sufficient.) Do not allow air to be drawn through the crystals as they are hygroscopic. Finally, pour 25 mL of ether into the crucible and draw through the precipitate with the aid of suction.

7.4 Wipe the outer surface of the crucible with a clean cloth and place in a gravity convection oven at 60°C for 1 h. Cool to room temperature in a desiccator, and weigh.

NOTE 2—The crucible weighings are for determining aliquot size and need be weighed to the nearest 10 mg only.

7.5 Dissolve the contents of the crucible in 70 mL of water, using a filtrator so as to collect the washings in a

250-mL beaker. Adjust the pH of the specimen to 2.5, using HNO₃ (1+3) and a pH test assembly. After the specimen has stood 30 min or longer, filter through double thicknesses of the finest paper directly into a 100-mL volumetric flask (Note 3). Dilute to volume with water, simultaneously using the water to wash the beaker and filter paper; mix thoroughly.

NOTE 3—If the solution does not cloud when acidified, it may be diluted to volume at once and the filtering omitted.

7.6 Withdraw an aliquot containing not less than 60 nor more than 90 mg of the dissolved salts. Transfer this aliquot into the 250-mL Erlenmeyer flask. Dry the contents of the flask in the oven at 60°C. Add 5 mL of glacial acetic acid, vent the stopper by inserting a paper strip under one side, and heat in the oven at 60°C for 1 h. Add 100 mL of anhydrous methanol and continue heating in the oven with occasional agitation until the material is completely dissolved. To the hot solution, add slowly, by pipet, 2.0 mL of lead acetate solution. Agitate the solution during the addition of this reagent. Return the flask to the 60°C oven for 1 h. Remove and stopper tightly after 30 min. Allow to stand 12 h or longer. Filter the solution through a dry tared fritted-glass crucible of medium porosity, to which additional mats of coarse and fine asbestos have been added. Agitate the solution just before filtering. Wash the flask thoroughly, using anhydrous methanol. Examine the filtrate carefully before discarding and, if cloudy, filter again through the same crucible. Dry the crucible for 1 h at 105°C, cool in a desiccator, and weigh.

8. Calculation

8.1 Calculate the percent of phthalic anhydride *A* in the specimen as follows:

$$A = [(P \times 0.323)/S] \times 100$$

where:

P = lead precipitate, g, and

S = specimen represented in the aliquot used, g.

9. Precision

9.1 Two results obtained by operators should be considered suspect if they differ by more than 1 % absolute.

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Standard Test Methods for Apparent Free Phenols in Synthetic Phenolic Resins or Solutions Used for Coating Purposes¹

This standard is issued under the fixed designation D 1312; the number immediately following the designation indicates the year of original adoption 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, including renumbering Sections 4 through 18 in November 1987.

Scope

1.1 These test methods cover the determination of the amount of apparent free phenol in synthetic phenolic resins and solutions used for coating purposes. The test method for isolation of the free phenol applies to all the commonly used resins except those containing *p*-phenyl-phenol. Test Method A applies to the simpler phenols up to and including the phenols; Test Method B applies to the common alkylated phenols.

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 are given in Sections 11 and 15.

2. Referenced Document

2.1 *ASTM Standard:*
D1193 Specification for Reagent Water²

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.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.

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

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 Varnish and Resins, Including Shellacs.

Current edition approved Sept. 10, 1956. Published November 1956. Originally published as D 1312 - 54. Last previous edition D 1312 - 54 T.

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."

ISOLATION OF FREE PHENOLS

4. Apparatus

- 4.1 *Steam Generator.*
- 4.2 *Distillation Flask*—A 500-mL Kjeldahl flask.
- 4.3 *Condenser.*

5. Reagents

5.1 *Sodium Hydroxide Solution (100 g NaOH/L)*—Dissolve 100 g of NaOH [Caution] in water and dilute to 1 L.

6. Procedure

6.1 Transfer 1 to 2 g of the sample, weighed to the nearest 0.01 g, to a 500-mL Kjeldahl flask. Add 50 mL of water. Lead steam from a steam generator through a T-trap and then through a tube extending to the bottom of the flask. Take off the distillate through a second bent tube that extends just through the stopper of the flask and is attached to the top of an inclined long condenser. Using a 1000-mL volumetric flask as a receiver, collect 900 mL of the distillate. A small flame may be applied to the bottom of the Kjeldahl flask during distillation to maintain the volume of water constant. If the distillate is clear, dilute to 1000 mL; if not, add several millilitres of NaOH solution to dissolve the insoluble phenols. Determine the apparent free phenol in accordance with Method A or B, as required.

NOTE 1—*p*-Phenylphenol is not completely isolated by this procedure.

TEST METHOD A—SIMPLER PHENOLS

7. Summary of Test Method

7.1 Free phenols are isolated by steam distillation, reacted with a measured excess of bromine, and the excess back-titrated with standard sodium thiosulfate solution.

8. Apparatus

- 8.1 *Iodine Flasks*, 500-mL, glass-stoppered.

9. Reagents

9.1 *Bromide-Bromate Solution*—Dissolve 2.784 g of KBrO₃ [Caution] and 10 g of KBr [Caution] in 1 L of water.

9.2 *Potassium Iodide Solution (100 g KI/L)*—Dissolve 100 g of KI [Caution] in water and dilute to 1 L.

9.3 *Sodium Thiosulfate, Standard Solution (0.1 N)*—Dissolve 25 g of Na₂S₂O₃·5H₂O [Caution] in 1 L of water. Standardize against iodine.

9.4 *Starch Indicator Solution*—Dissolve 1 g of soluble starch in 100 mL of boiling water and cool.

10. Procedure

10.1 Pipet a 25-mL (or larger) aliquot of the distillate into a 500-mL iodine flask. Add 25 mL of KBr - KBrO₃ solution, shake, and add 10 mL of HCl (sp gr 1.19). Stopper quickly, shake to mix thoroughly, and fill the gutter with water. Let stand 15 min. (If the bromine color disappears during the shaking, take a fresh aliquot, add double the amount of bromate solution, and proceed as described.) Raise the stopper carefully and add 10 mL of the KI solution. Shake, and wash down the stopper and walls of the flask. Titrate with 0.1 N Na₂S₂O₃ solution using 1 mL of starch indicator at the end.

10.2 *Blank*—Run a blank in exactly the same manner, except to omit the test material.

11. Calculation

11.1 Calculate the percentage of apparent free phenols (Note 2), as follows:

$$\text{Phenols, \%} = [(T_1 - T) N \times 1.567] / W$$

where:

T_1 = Na₂S₂O₃ solution required for titration of the blank, mL,

T = Na₂S₂O₃ solution required for titration of the sample, mL,

N = normality of the Na₂S₂O₃ solution, and

W = sample represented in the aliquot used, g.

NOTE 2—The cresols (CH₃C₆H₄OH) and xylenols (CH₃)₂C₆H₃OH have one or two reactive positions on the ring, depending on the isomer, but their tendency to add more bromine as an addition product that is not later removed by KI renders the exact extent of reaction uncertain. Since those materials are ordinarily used as mixtures in a resin-forming reaction, it is convenient and sufficiently informative to express the amount found as primary phenol.

12. Precision

12.1 The individual test results should not differ from the mean by more than ± 0.5 %.

TEST METHOD B—COMMON ALKYLATED PHENOLS

13. Summary of Test Method

13.1 Free phenols are isolated by steam distillation, reacted with a measured excess of iodine, and the excess back-titrated with standard sodium thiosulfate solution.

14. Apparatus

14.1 *Iodine Flasks*, 500-mL, glass stoppered.

15. Reagents

15.1 *Iodine Solution*—Dissolve 4.2 g of iodine [Caution] in 15 g of a saturated aqueous KI solution and dilute to 1 L.

15.2 *Sodium Bicarbonate Solution* (84 g NaHCO₃/L)—Dissolve 84 g of NaHCO₃ in water and dilute to 1 L.

15.3 *Sodium Hydroxide Solution* (100 g NaOH/L)—Dissolve 100 g of NaOH [Caution] in water and dilute to 1 L.

15.4 *Sodium Thiosulfate, Standard Solution* (0.1 N)—See 7.3.

15.5 *Starch Indicator Solution*—See 7.4.

15.6 *Sulfuric Acid* (1+19)—Add slowly, while stirring, 1 volume of concentrated H₂SO₄ [Caution] (sp gr 1.84) to 19 volumes of water.

16. Procedure

16.1 Add 15 mL of NaOH solution to the steam distillate and dilute to 1 L. Add 100 mL of water to a 500-mL iodine flask. Pipet a 10-mL (or larger) aliquot of distillate into the flask (Note 3) and add 30 mL of the iodine solution. Stopper and shake continuously for 5 min. Add 50 mL of H₂SO₄ (1+19) to the flask and titrate with 0.1 N Na₂S₂O₃ solution to a colorless end point, using 1 mL of starch solution as the indicator.

NOTE 3—If the sample is pure *p*-phenylphenol, add 50 mL of the NaHCO₃ solution to the flask before the sample is added to prevent the formation of color that interferes with the titration end point.

16.2 *Blank*—Run a blank in exactly the same manner, except omit the sample.

17. Calculation

17.1 Calculate the percentage of apparent free alkylphenols (Note 4) as follows:

$$\text{Phenols, \%} = [(B - V) N \times F] / S$$

where:

B = Na₂S₂O₃ solution required for titration of the blank, mL,

V = Na₂S₂O₃ solution required for titration of the sample, mL,

N = normality of the Na₂S₂O₃ solution,

F = conversion factor

= 3.755 for *p*-tertiary-butyl phenol

= 4.106 for *p*-tertiary-amyl phenol

= 4.255 for *p*-phenyl phenol, and

S = sample represented in the aliquot used, g.

NOTE 4—The alkyl phenols form normal di-iodo compounds.

18. Precision

18.1 The individual test results should not differ from the mean by more than ± 0.5 %.

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Standard Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method¹

This standard is issued under the fixed designation D 1343; the number immediately following the designation indicates the year of original adoption 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 4272-1 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} Keywords were added editorially in March 1991.

Scope

1.1 This test method describes the apparatus and general procedure for making ball-drop viscosity measurements on solutions of various cellulose derivatives. Instructions for sample preparation, solution concentration, and other details are discussed in the ASTM methods for the respective cellulose derivatives.

1.2 This test method is applicable to solutions of various cellulose derivatives having viscosities greater than 10 P, by using balls of various diameters and densities. Viscosity results are expressed preferably in poises.

1.3 In commercial practice, viscosities are often expressed in seconds using $\frac{3}{32}$ -in. (2.38-mm) stainless steel balls.² When the viscosity is outside the practical range for these balls (75 to 300 P), the measurement can be made using a calibrated pipet viscometer or a different ball and calculating the observed viscosity to the corresponding time for a $\frac{3}{32}$ -in. ball, even though it is a small fraction of a second.

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:

- D 301 Test Methods for Soluble Cellulose Nitrate³
- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)⁴
- D 817 Test Methods of Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate³
- D 871 Test Methods of Testing Cellulose Acetate³

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 D01.36 on Cellulose and Cellulose Derivatives.

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² When a $\frac{3}{32}$ -in. stainless steel ball is used, the viscosities in seconds should be practically the same as those obtained using the apparatus described in Section 11 of Methods D 871 - 48, and in Section 10 of Specifications D 301 - 50, which last appeared in the 1952 Annual Book of ASTM Standards, Part 4.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 05.01.

3. Summary of Test Method

3.1 A solution of the cellulose derivative is made in a suitable solvent and allowed to equilibrate at a chosen temperature. A stainless steel or aluminum ball is dropped into the solution, and the time required for it to cover a measured distance in its fall is recorded. The viscosity of the solution can then be calculated in poise or recorded in seconds.

4. Significance and Use

4.1 This test provides an easy method of determining the viscosity of cellulose derivatives in a given solvent. The answers are in units commonly used in industrial practice. Such information is needed for cellulose derivatives that are to be extruded, molded, sprayed, or brushed as is or in solution.

5. Apparatus

5.1 Constant-Temperature Water Bath, glass-walled.

5.2 Bottles and Caps:

5.2.1 Bottles, round or square, conforming to the dimensional requirements shown in Table 1, shall be used. Screw caps of metal or phenolic plastic in sizes to fit the bottles and having aluminum foil or cardboard and cellophane liners may be used to close the bottles. Alternatively, rubber stoppers covered with aluminum or tin foil, may also be used as closures. In this latter case, solvent loss during measurement of viscosity can be minimized by removing the stopper, leaving the foil in place, and making a small hole in the center of the foil through which the balls may be dropped.

5.2.2 Timing marks shall be provided around each bottle or on the front and back of the glass-walled constant-temperature water bath, to avoid parallax errors. The lower timing mark shall be approximately 1.25 in. (32 mm) above the base of the bottle, and the upper mark shall be 2.00 ± 0.02 in. (50.8 ± 0.5 mm) above the lower mark. A practical means of marking consists of wrapping a 2-in. strip of transparent sheeting around the water bath at the proper location. The edges of the sheeting may be darkened with crayon. A light located back of the water bath aids in observing the ball during its fall.

5.3 *Balls*—Unless specifically directed otherwise, balls of varying size and density shall be used, depending on the viscosity of the solution. Table 2 gives the useful ranges, approximate apparatus constants, and dimensions of several

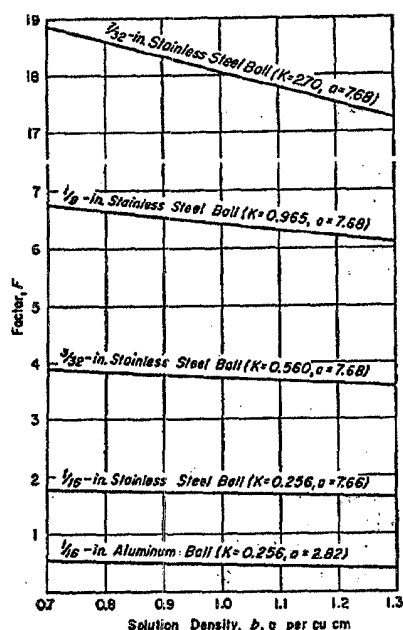


FIG. 1 Factors for Converting Viscosities in Seconds to Poises
 $\eta = F \times t$

TABLE 1 Bottles^A

Bottle	Round	Square
Capacity, oz	16	16
Weight, oz	12	12
Height, in.	6.7	7
Inside diameter, cm	6.4	
Side to side, cm	...	6.0
Corner to corner, cm	...	7.2

^A Approximate sizes. Bottles satisfactory for this purpose may be obtained from the Owens Illinois Glass Co., Ohio Illinois Bldg., Toledo, Ohio, 43601, as follows: round bottle No. C-3145 with cap 83-400, French square bottle A-6732 with cap 48-400.

such balls. The exact diameter, weight, and density shall be determined accurately for each lot of balls used.

5.4 *Graduate*—A 50 or 100-ml graduated cylinder, having a round top opening that can be tightly stoppered, shall be used for determining the density of the solution in grams per millilitre.

5.5 *Stop Watch*—A stop watch reading to 0.2 s.

6. Calibration

6.1 Calculate the apparatus constant, K , using the follow-

ing equation and exact dimensions of the bottle and balls used:

$$K = 2gr^2[1 - 2.104(d/D) + 2.09(d/D)^3]/9L$$

where:

g = acceleration of gravity in cgs units

r = ball radius, cm,

d = ball diameter, cm,

D = bottle diameter, cm (in the case of square bottles the average of the side to side and corner to corner diameters shall be used), and

L = distance of ball drop, cm.

7. Procedure

7.1 *Preparation of Solution*—Dry the sample and prepare a solution as specified for the particular material. Such instructions are given in the viscosity sections of Methods D 301, D 871, and Methods D 817. Weigh into the bottle an appropriate amount of dry sample and specified solvent, accurate to 0.1 g, to make about 350 ml of solution. Close the bottle tightly. Allow to stand a short time for the solvent to penetrate the sample. Then tumble or shake until a uniform solution is obtained. Transfer to the water bath at 25 ± 0.1 C, and allow the solution to come to temperature.

7.2 *Viscosity Determination*—Drop a 3/16-in. (2.38-mm) stainless steel ball through the center of the column of solution and time its fall through the marked 2-in. (50.8-mm) distance, using a stop watch and taking precautions to avoid parallax errors. If the observed time is less than 20 s or greater than 100 s repeat the measurement, unless directed otherwise, using a different ball (see Table 2) which has a time of fall within these limits. If the solution is known to be thixotropic in nature or if the times of fall for successive balls vary significantly, use freshly prepared solutions for duplicate measurements or measurements with balls of other sizes.

7.3 *Determination of Lower Viscosities*—If the viscosity of the solution is too low to measure satisfactorily using one of the balls, use a calibrated pipet as described in Test Method D 445, or other instrument of suitable range. Calculate the result in poises. Convert poises to equivalent ball-drop seconds as shown in 8.2.

7.4 *Density Determination*—Determine the density of the solution in grams per cubic centimetre by measuring the volume at 25 ± 0.1 C of a known weight of the solution contained in a suitable tightly stoppered graduated cylinder.

8. Calculation

8.1 *Ball-Drop Viscosities*—Calculate the viscosity in poises as follows:

$$\eta = K(a - b)t$$

TABLE 2 Balls

Ball	Viscosity Range, P	Typical Data			
		Apparatus Constant, K	Diameter, cm	Weight, g	Density, g/cm ³ , a
1/16-in. (1.59-mm) (aluminum)	10 to 50	0.256	0.1588	0.00691	2.82
1/16-in. (1.59-mm) (stainless steel)	35 to 150	0.256	0.1588	0.01805	7.66
3/16-in. (2.38-mm)	75 to 300	0.560	0.2380	0.0542	7.68
1/8-in. (3.18-mm)	125 to 600	0.965	0.3170	0.1277	7.68
5/16-in. (5.56-mm)	350 to 1800	2.70	0.5556	0.6897	7.68

where:

- η = viscosity at the specified temperature, P,
- K = apparatus constant,⁵
- ρ = ball density⁵, in g/cm³,
- ρ_s = solution density, g/cm³, and
- t = time of fall, s.

In the case of a ball of stated diameter and density, this calculation can be simplified to:⁵

$$\eta = F \times t,$$

where:

$$F = K(a - b).$$

This factor varies with solution density, b. Approximate factors for the various balls can be read from Fig. 1. Exact factors can be calculated from the exact measurements of the viscometer and balls.

8.2 *Poises to Seconds*—Poises may be converted to equivalent ball-drop seconds, t , as follows:

$$t(\text{for } \frac{3}{32}\text{-in. ball}) = \eta/K(a - b)$$

where:

- η = observed viscosity, P,
- K = apparatus constant for the $\frac{3}{32}$ in. stainless steel ball,
- a = ball density for the $\frac{3}{32}$ -in. stainless steel ball, and

⁵ See Table 2 for approximate values.

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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.

b = solution density for the solution being tested.

9. Report

9.1 Results shall be reported in poises, or in seconds, for a $\frac{3}{32}$ -in. stainless steel ball.

10. Precision and Bias

10.1 The within-laboratory precision of the test was determined by submitting 25 pairs of replicate samples to be run by any one of several operators. Each sample in a specific pair was submitted within 3 to 5 days. Each pair consisted of one sample at the 20-second viscosity level and one at the 60-second viscosity level. The data below shows the 95 % confidence limits (two sigma) for the two levels.

Viscosity Level	95 % Confidence Limits
60 sec	± 3.83 sec
20 sec	± 1.00 sec

Data are not available at this time to show interlaboratory precision.

10.2 Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring ball-drop viscosity, no statement on bias can be made.

11. Keywords

11.1 ball drop; cellulose esters; viscosity



Standard Test Methods for Methylcellulose¹

This standard is issued under the fixed designation D 1347; the number immediately following the designation indicates the year of original adoption 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.

^{e2} NOTE—Keywords were added editorially in April 1991:

1. Scope

1.1 These test methods cover the testing of methylcellulose.

1.2 The test methods appear in the following order:

	Sections
Moisture	4 and 5
Ash—as Sulfate	6 to 8
Chlorides—as Sodium Chloride	9 to 11
Alkalinity—as Na ₂ CO ₃	12 to 14
Iron	15 to 19
Heavy Metals	20 to 22
Methoxyl Content	23 to 26
Viscosity:	
Water-Soluble Methylcellulose	27 to 29
Alkali-Soluble Methylcellulose	30 to 31
pH	32
Solids	33 to 34
Density	35 to 39

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. For a specific hazard statement, see Note 1.*

2. Referenced Document

2.1 ASTM Standard:

D 96 Test Methods for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)²

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.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.

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 D01.36 on Cellulosics.

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² Annual Book of ASTM Standards, Vol 05.01.

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."

3.2 Unless otherwise indicated, references to water shall be understood to mean distilled water.

MOISTURE

4. Procedure

4.1 Transfer 2 to 5 g of the sample, weighed to the nearest 0.01 g, to a tared dish (fitted with a lid) and dry it for 3 h in an oven at 105 ± 3°C. Remove the dish from the oven, cover it with a lid, cool in a desiccator, and weigh.

5. Calculation

5.1 Calculate the percent moisture, *M*, as follows:

$$M = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, g, and

B = sample used, g.

ASH—AS SULFATE

6. Reagent

6.1 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

7. Procedure

7.1 Weigh, to the nearest 0.01 g, about 2 g of the sample (previously dried for ½ h at 105°C) and transfer it to a tared platinum crucible. Place it in a muffle furnace at 575 ± 25°C for approximately ½ h, to char the organic material.

7.2 Cool the crucible and add 1 mL of H₂SO₄ so that it completely wets the charred residue. Then cautiously heat it over a small flame to dense white fumes. Place the crucible in a muffle furnace at 575 ± 25°C and leave it there until all signs of carbon are gone (approximately 1 h). Transfer the specimen to a desiccator until cool, then weigh.

8. Calculation

8.1 Calculate the percent of ash, *C*, as follows:

$$C = (A/B) \times 100 \quad (2)$$

where:

A = ash, g, and

B = sample used, g.

CHLORIDES—AS SODIUM CHLORIDE

9. Reagents

9.1 *Ferric Alum Indicator Solution*—Add 100 g of ferric aluminum sulfate (Fe₂(SO₄)₃ · (NH₄)₂SO₄ · 24H₂O) to 250 mL

water. Heat it to boiling and add HNO_3 (sp gr 1.42) slowly until the red color is removed. This will usually require about 15 mL of HNO_3 . Filter the solution and store it in a glass bottle.

9.2 Potassium Thiocyanate, Solution, Standard (0.1 N)—Dissolve 10 g of potassium thioanionate (KCNS) in 1 L of water. By means of a pipet, measure 25 mL of 0.1000 N AgNO_3 solution into a 400-mL beaker. Add 100 mL of water, 10 mL of nitric acid (HNO_3 , sp gr 1.42) and 5 mL of ferric alum indicator solution. Titrate with the KCNS solution, while stirring, until a faint persistent red color is produced. Calculate the normality of the KCNS solution, N , as follows:

$$N = (A/B) \times 0.1 \quad (3)$$

where:

A = 0.100 N AgNO_3 solution added, mL, and

B = KCNS solution required for the titration, mL.

9.3 Silver Nitrate, Solution, Standard (0.1 N)—Grind silver nitrate (AgNO_3) crystals fine enough to pass through a 850- μm (No. 20) sieve, and then dry for 2 h at 110°C. Prepare a 0.1000 N solution by dissolving 16.989 g of dry AgNO_3 in chloride-free water and diluting it to 1 L in a volumetric flask.

10. Procedure

10.1 Weigh, to the nearest 0.01 g, about 1.0 g of the sample (previously dried for ½ h at 100 to 105°C) and transfer to a 500-mL wide-mouth Erlenmeyer flask. Add 250 mL of hot water to the flask and swirl it for a few minutes, then cool to dissolve.

10.2 Add 5 mL of 0.1000 N AgNO_3 solution and 5 mL of ferric alum indicator solution, and then back-titrate with 0.1 N KCNS solution to the first appearance of a faint pink color.

11. Calculation

11.1 Calculate the percent of chlorides, C , as sodium chloride (NaCl) as follows:

$$C = [(AB - CD) \times 0.0585]/E \times 100 \quad (4)$$

where:

A = AgNO_3 solution added, mL,

B = normality of the AgNO_3 solution,

C = KCNS solution required to back-titrate the excess AgNO_3 , mL,

D = normality of the KCNS solution, and

E = sample used, g.

ALKALINITY—AS SODIUM CARBONATE, ANHYDROUS

12. Reagents

12.1 Methyl Purple Indicator Solution.

12.2 Sulfuric Acid, Standard (0.01 N)—Prepare and standardize a 0.01 N solution of sulfuric acid (H_2SO_4).

13. Procedure

13.1 Weigh, to the nearest 0.01 g, about 1.0 g of the sample (previously dried for ½ h at 100 to 105°C) and transfer it to a 500-mL wide-mouth Erlenmeyer flask. Add 250 mL of hot water to the flask and swirl it for a few minutes, then cool to dissolve.

13.2 Add 4 drops of methyl purple indicator to the flask solution and titrate to a blue-gray end point with 0.01 N H_2SO_4 .

14. Calculation

14.1 Calculate the percent of alkalinity, D , as anhydrous sodium carbonate (Na_2CO_3) as follows:

$$D = [(AB \times 0.053)/C] \times 100 \quad (5)$$

where:

A = H_2SO_4 required for titration of the sample, mL,

B = normality of the H_2SO_4 , and

C = sample used, g.

IRON

15. Apparatus

15.1 Photometer—Any photoelectric filter photometer or spectrophotometer suitable for measurements at 430 nm.

15.2 Kjeldahl Flasks, calibrated to contain 30 mL, and made of heat- and chemical-resistant glass.

16. Reagents

16.1 Ammonium Hydroxide (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

16.2 Buffer Solution—Dissolve 20 g of sodium bicarbonate (NaHCO_3) and 10 g of sodium carbonate (Na_2CO_3) in water and dilute to 1 L.

16.3 Disodium-1,2-Dihydroxybenzene-3,5-Disulfonate Solution⁴—Prepare an aqueous solution containing 25 g/L.

16.4 Hydrogen Peroxide (30 %)—Concentrated hydrogen peroxide (H_2O_2).

16.5 Iron, Solution, Standard (0.0001 g Fe/mL)—Dissolve 0.01 g of iron powder containing not less than 99.9 % Fe in hydrochloric acid (HCl , sp gr 1.19). Oxidize the solution with bromine water and expel the excess by boiling. Dilute to 1 L in a volumetric flask.

16.6 Phenolphthalein Indicator Solution.

16.7 Sulfuric Acid (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

16.8 Sulfuric Acid (1 + 4)—Carefully mix 1 volume of H_2SO_4 (sp gr 1.84) with 4 volumes of water, adding the H_2SO_4 gradually while mixing.

17. Preparation of Calibration Curve

17.1 Following the procedure given in Section 18, and using varied amounts of the standard iron solution prepared in accordance with 16.1, prepare a calibration curve showing iron content in parts per million and the corresponding photometer readings.

18. Procedure

18.1 Weigh approximately 2 g of the sample of methylcellulose to the nearest 0.01 g, and transfer by means of a funnel to a Kjeldahl flask. Place the flask at a 20° angle in a furnace at 600°C, or on a microdigestion rack equipped with electric heating elements, and heat until some charring of the methylcellulose has taken place. (Care must be taken not to

⁴ A suitable prepared solution of this reagent, known as Tiron, is available from the La Motte Chemical Products Co., Chestertown, MD.

char too much.) Remove and allow to cool.

18.2 Add 3 mL of concentrated H_2SO_4 to the flask. Place the flask on the digestion rack and digest. Cool, and add H_2O_2 dropwise until the solution is clear. Heat over a Meker burner to a volume of 2 mL. Cool, and wash the sides of the flask with water. Add 3 drops of phenolphthalein indicator solution. Add NH_4OH to a red end point. Wash the neck of the flask. The solution should be clear and not greater than 20 mL in volume.

18.3 Add 2 mL of the color-forming solution described in 16.3 and mix. Dilute to near the mark with buffer solution and mix thoroughly. Adjust the solution to a pH of 7 by adding NH_4OH or H_2SO_4 (1 + 4), and then dilute to the mark. Transfer a small portion to an absorption cell and determine the photometer reading at 430 nm.

19. Calculation

19.1 Read the iron content, in parts per million, directly from the calibration curve (Section 17).

HEAVY METALS

20. Apparatus

20.1 Nessler Tubes, 50-mL.

20.2 Volumetric Flasks, 50-mL.

21. Reagents

21.1 *Acetic Acid* (6 + 94)—Mix 6 volumes of glacial acetic acid with 94 volumes of water.

21.2 *Ammonium Hydroxide* (1 + 5)—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH , sp gr 0.90) with 5 volumes of water.

21.3 *Hydrochloric Acid* (1 + 3)—Mix 1 volume of concentrated hydrochloric acid (HCl , sp gr 1.19) with 3 volumes of water.

21.4 *Hydrogen Sulfide Solution* (Saturated)—Saturate cold water with hydrogen sulfide (H_2S).

21.5 *Lead, Solution, Standard* (1 mL = 0.1 mg Pb)—Dissolve 0.1598 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 100 mL of water to which has been added 1 mL of concentrated nitric acid (HNO_3 , sp gr 1.42). Dilute to 1000 mL with water.

21.6 *Lead, Solution, Standard* (1 mL = 0.01 mg Pb)—Dilute 10.0 mL of $\text{Pb}(\text{NO}_3)_2$ solution (1 mL = 0.1 mg Pb) to 100 mL with water. This solution must be freshly prepared. When 0.1 mL of this standard lead solution is employed to prepare the standard to be compared with a solution of 1 g of the substance being tested, the comparison solution thus prepared contains the equivalent of one part of lead per million parts of the substance tested.

21.7 *Phenolphthalein Indicator Solution*.

22. Procedure

22.1 Add 5 mL of HCl (1 + 3) to the residue in the platinum crucible that was used in the sulfate ash determination (Sections 6 and 7). Digest the residue by slowly boiling for a few minutes over a small flame. Transfer the contents of the crucible to a 50-mL volumetric flask, using about 25 mL of water to rinse the crucible. Neutralize with NH_4OH (1 + 5) to a phenolphthalein end point and dilute to 50 mL.

22.2 Transfer a 25-mL aliquot of the solution to a 50-mL Nessler tube, and add 2 mL of acetic acid (6 + 94) and 10

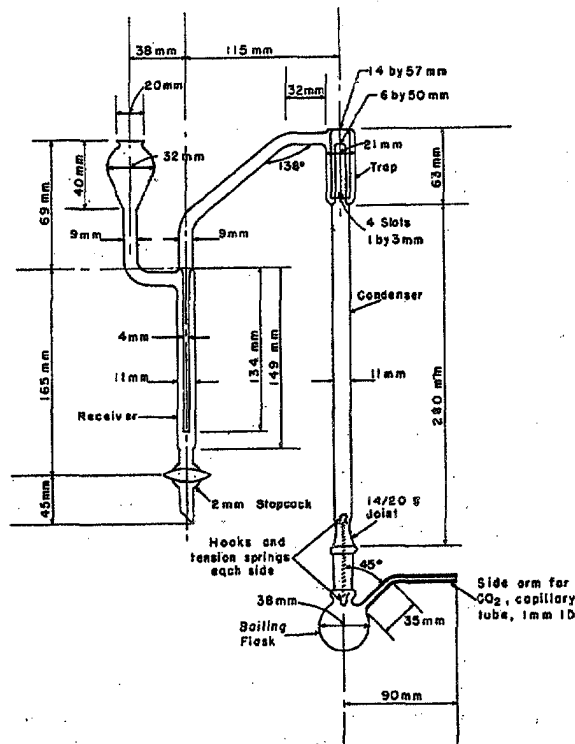


FIG. 1 Distillation Apparatus for Methoxyl Determination

mL of a saturated solution of H_2S . Mix, allow to stand for 10 min, and compare with a standard lead solution to which H_2S has been added.

22.3 Report the lead content in parts per million.

METHOXYL CONTENT

23. Apparatus

23.1 *Distillation Apparatus* (Fig. 1), consisting of a boiling flask with a side arm for admission of carbon dioxide (CO_2) or nitrogen, an air condenser with a trap, and a receiver.

23.2 *Oil Bath*, equipped with a heating device, preferably electrical, so that the bath can be maintained at 145 to 150°C.

24. Reagents and Materials

24.1 *Bromine Solution*—Dissolve 5 mL of bromine in 145 mL of the potassium acetate solution. Prepare the bromine solution fresh daily in a hood to remove bromine vapors.

24.2 *Carbon Dioxide*—This may be obtained by the interaction of marble and HCl (1 + 1) in a Kipp generator or, preferably, from a cylinder of the gas equipped with a suitable needle valve. The CO_2 shall be passed through a bubble counter and a dry trap, and then through a pressure regulator consisting of a glass tee whose vertical arm extends almost to the bottom of a 10-in. (254-mm) column of water. A screw clamp is attached to the thin-walled rubber tubing connecting the horizontal arm of the tee with the boiling

flask. This arrangement permits regulation of the flow of gas and allows any excess gas to escape. Nitrogen may be used in place of CO₂.

24.3 *Formic Acid* (HCOOH, 90 %).

24.4 *Gelatin Capsules*—Gelatin capsules of a suitable size to hold 50 to 60 mg of the dried specimen will be required.

24.5 *Hydriodic Acid* (57 %, sp gr 1.70)—Hydriodic acid (HI) forms with water a constant-boiling mixture (boiling point 126 to 127°C) which contains 57 % HI. The concentration of HI in the reagent used should be not less than 55 %. The blank determination, which is affected primarily by free iodine in the reagent, should require not more than 5 mL of 0.1 *N* sodium thiosulfate (Na₂S₂O₃) solution.⁵ If necessary, the acid may be purified by adding to it a small amount of red phosphorus and boiling for 20 to 30 min in a hood, while passing a stream of CO₂ into the liquid. Distillation shall then be carried out behind a safety glass shield in a hood, using an all-glass apparatus with a slow stream of CO₂ running through the receiver.

NOTE 1—Warning—Under some conditions, the poisonous gas phosphine (PH₃) is formed during distillation and this may unite with molecular iodine to form phosphorus triiodide (PI₃), which may explode on contact with air. It is, therefore, advisable to keep the current of CO₂ going after the distillation is ended and until the apparatus has cooled; this will prevent air from being sucked into the apparatus. Put the purified HI in small, brown, glass-stoppered bottles, previously swept out with CO₂, and seal the stoppers with molten paraffin. Store in a dark place. To minimize decomposition of HI due to contact with air, run CO₂ into the bottle while withdrawing portions of the acid for use.

24.6 *Potassium Acetate Solution*—Dissolve 100 g of anhydrous potassium acetate crystals in 1 L of a solution containing 900 mL of glacial acetic acid and 100 mL of acetic anhydride.

24.7 *Potassium Iodide* (KI).

24.8 *Sodium Acetate Solution* (220 g/L)—Dissolve 220 g of anhydrous sodium acetate in water and dilute to 1 L.

24.9 *Sodium Thiosulfate Solution, Standard* (0.1 *N*)—Dissolve 25 g of sodium thiosulfate (Na₂S₂O₃ · 5H₂O) in 200 mL of water and dilute to 1 L. Use freshly boiled and cooled water. It is preferable to allow the solution to stand for a few days before standardization. Standardize the solution against 0.1000 *N* potassium dichromate (K₂Cr₂O₇) solution prepared by dissolving exactly 4.9037 g of K₂Cr₂O₇ (National Institute of Standards and Technology Standard Sample No. 136) in water and diluting to 1 L in a volumetric flask. By means of a buret, measure accurately 35 to 45 mL of the K₂Cr₂O₇ solution into a 250-mL Erlenmeyer flask. Add 2 g of KI and 50 mL of H₂SO₄ (1+9) and allow to stand for about 5 min. The flask should be stoppered during the standing period to avoid loss of iodine. Titrate the liberated iodine with the Na₂S₂O₃ solution, using starch indicator solution near the end point. At the end point, the blue color of the starch indicator will be destroyed, leaving the pale green color of the chromate ion. The normality of the Na₂S₂O₃ solution should be checked at least once a week.

⁵ Hydriodic acid suitable for methoxyl determination may be prepared by the method of Samsel, E. P., and McHard, J. A., *Industrial and Engineering Chemistry, Analytical Edition*, Vol 14, 1942, p. 750. Hydriodic acid available from Merck & Co., WBC 220, P.O. Box 2000, Rahway, NJ 07065 under the designation "For Methoxyl Determination" has been found satisfactory for this purpose.

Calculate the normality of the Na₂S₂O₃ solution, *N*, as follows:

$$N = (A/B) \times 0.1 \quad (6)$$

where:

A = 0.1000 *N* K₂Cr₂O₇ solution added, mL, and

B = Na₂S₂O₃ solution required for the titration, mL.

As an alternative procedure, the Na₂S₂O₃ solution may be standardized against 0.1 *N* iodine solution that has been standardized in turn against arsenic trioxide (As₂O₃, National Institute of Standards and Technology Standard Sample No. 83) or potassium iodate (KIO₃).

24.10 *Starch Indicator Solution*.

24.11 *Sulfuric Acid* (1 + 9)—Carefully mix 1 volume of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) with 9 volumes of water, adding the H₂SO₄ gradually while mixing.

25. Procedure

25.1 Dry the sample at 105°C for at least 30 min. Through the condenser, add to the trap in the distillation apparatus (Fig. 1) enough distilled water to make the trap about half full. Add 8 to 9 mL of bromine solution to the receiver. Weigh 50 to 60 mg of the dry sample, to the nearest 0.1 mg, into a gelatin capsule and drop it into the boiling flask. (The weighing should be done as rapidly as possible without sacrificing accuracy, since dry methylcellulose picks up moisture rapidly.)

25.2 Add a few small glass beads or chips of clay plate and then 6 mL of HI. Attach the boiling flask at once to the condenser, using a few drops of HI to moisten the ground-glass joint, and then connect the side arm of the flask to the source of CO₂. Pass a current of CO₂ into the apparatus at the rate of about 2 bubbles/s. Immerse the flask in the oil bath, maintained at 150°C, and heat for 50 min.

25.3 Add 10 mL of sodium acetate solution to a 500-mL Erlenmeyer flask and wash into it the contents of the receiver; dilute to 125 mL with water. Add HCOOH dropwise, with swirling, until the brown color of bromine is discharged, and then add about 6 drops more. A total of 12 to 15 drops is usually required. After about 3 min add 3 g of KI and 15 mL of H₂SO₄ (1 + 9) and titrate immediately with 0.1 *N* Na₂S₂O₃ solution to a light straw color. Add a little starch solution and continue the titration to the disappearance of the blue color.

25.4 *Blank*—Make a blank determination, using the same amounts of reagents and the same procedure as for the sample. (Usually, about 0.1 mL of 0.1 *N* Na₂S₂O₃ solution is required.)

26. Calculation

26.1 Calculate the percent of methoxyl, *M*, as follows:

$$M = [(A - B)C \times 0.00517]/D \times 100 \quad (7)$$

where:

A = Na₂S₂O₃ solution required for titration of the sample, mL,

B = Na₂S₂O₃ solution required for titration of the blank, mL,

C = normality of the Na₂S₂O₃ solution, and

D = sample used, g.

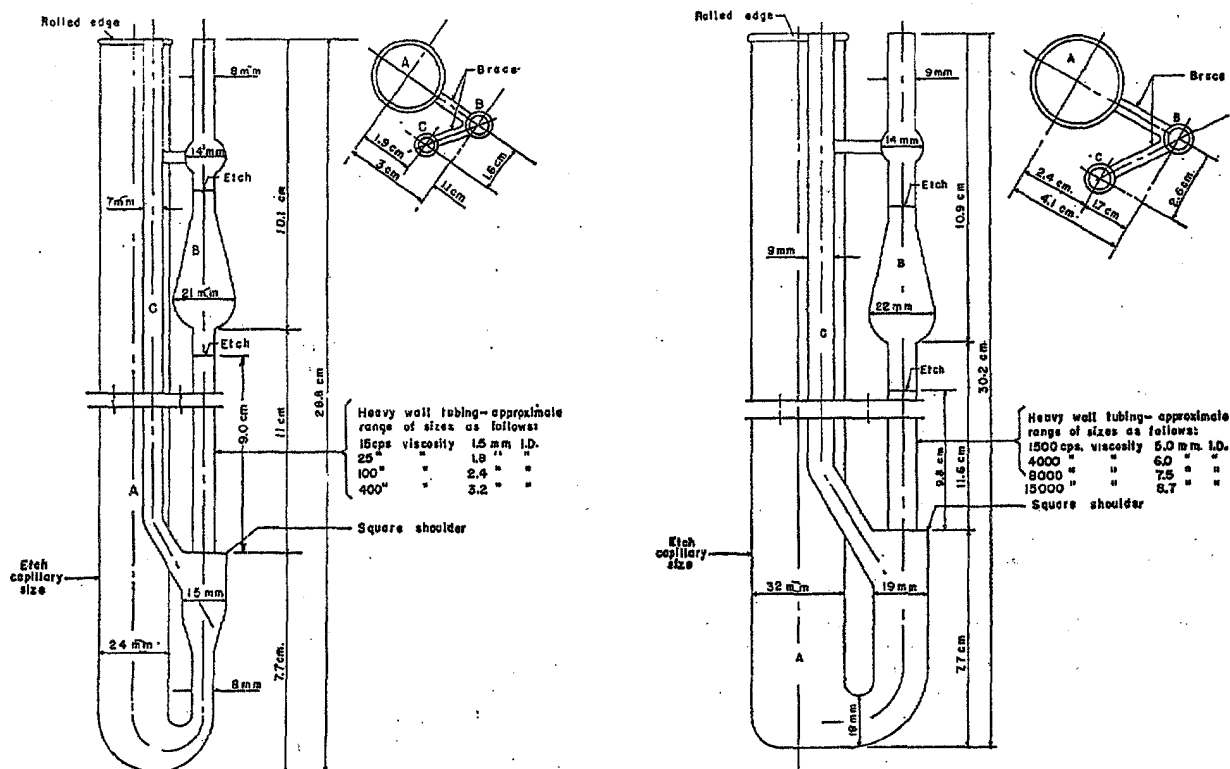


FIG. 2 Methycellulose Viscometers

VISCOSITY OF WATER-SOLUBLE METHYLCELLULOSE

27. Apparatus

27.1 Viscometer, Fig. 2(a) or (b).

NOTE 2—If a viscometer has been repaired, it should be recalibrated before it is used again. Even minor repairs can cause significant changes in the K value.

27.2 Mechanical Stirrer.

28. Procedure

28.1 Determine the moisture content of a portion of the sample. Since cellulose and its water-soluble derivatives are hygroscopic, exposure of the sample to the atmosphere should be kept to a minimum. Changes in moisture content can introduce large errors into the accuracy of the determination and this step should never be omitted if precise results are desired.

28.2 Correcting for the moisture content, weigh out enough of the sample of undried methylcellulose to give 2.000 g of solids, calculated as follows:

$$\text{Weight of specimen, g} = [100 / (100 - \text{moisture content, \%})] \times 2 \quad (8)$$

28.3 Place the sample in an 8-oz (250-cm³) wide-mouth bottle. This weighing step is critical in obtaining good checks and should be done on a good balance sensitive to 1 mg. Weights to the nearest 0.01 g will be sufficiently accurate.

28.4 Add 98.0 g of hot water (85 to 90°C) to the 8-oz

bottle containing the 2-g specimen of methylcellulose.

28.5 Agitate with a mechanical stirrer for 10 min, then place the bottle in an ice bath (0 to 5°C) until solution is complete. Equip the stirrer assembly with a one-hole stopper or bottle cap so that no water vapor is lost during agitation. For precision, it is preferable to deair the solution by some means such as centrifuging.

28.6 When solution is complete, as evidenced by the absence of partially swollen or undispersed particles, determine the viscosity in a methylcellulose viscometer at $20 \pm 0.1^\circ\text{C}$. Observe two precautions at this point: (1) the solution shall be essentially free of air bubbles, and (2) the temperature of the material in the tube shall be checked to make certain that it is actually at the bath temperature.

28.7 The methylcellulose viscosity tube (Fig. 2) consists of three parts: (1) a large filling tube with a reservoir at its lower extremity, A; (2) the orifice tube, B; and (3) an air vent to the reservoir, C. When B is filled, close C to prevent the sucking of air bubbles into the orifice tube.

28.8 Before the specimen is allowed to flow through the orifice for the viscosity determination, open the vent, C, so that the column of solution in B will flow into the reservoir against atmospheric pressure. Failure to open C before running will cause false values in the viscosity results.

29. Calculation

29.1 Calculate the viscosity as follows:

$$V = Kdt$$

(9)

where:

- = viscosity, cP,
- = viscometer constant (Note 2),
- = density of the methylcellulose solution at 20/20°C (Note 3), and
- = time for the solution to pass from the upper to the lower mark of the viscometer, s.

NOTE 3—The viscometer constant is determined by passing a standard oil of known viscosity through the tube and determining the time of flow. The above equation can then be solved for K .

NOTE 4—For routine work, the density of solutions of methylcellulose may be assumed to be 1.00.

VISCOSITY OF ALKALI-SOLUBLE METHYLCELLULOSE

30. Reagents

30.1 *Sodium Hydroxide Solution* (40 g NaOH/L)—Dissolve 40 g of sodium hydroxide (NaOH) in carbon dioxide (CO₂)-free water and dilute to 1 L.

31. Procedure

31.1 Proceed as directed in Sections 28 and 29, except add 98.0 g of the NaOH solution to the sample, instead of adding hot water in accordance with 28.3.

pH

32. Procedure

32.1 Determine the pH of the viscosity solution from Section 28, using any suitable pH meter.

SOLIDS

33. Apparatus

33.1 *Oil Tubes*, graduated, 100-mL, tapered, conforming to the requirements prescribed in 3.2 and Fig. 1 of Test Methods D 96.

33.2 *Centrifuge*, capable of whirling filled centrifuge tubes at a speed that will produce a centrifugal force of 725 times gravity.

34. Procedure

34.1 Add 1.50 g of bone-dry methylcellulose to 148.5 g of 90°C water in a 2¼ by 6-in. (32 by 152-mm) bottle and agitate vigorously for about 15 min, or until the material has become finely divided. Place an ice bath around the bottle and agitate the mixture until the solution is effected. (This

usually requires about 15 min.)

34.2 Place 100 mL of this 1 % solution in an oil tube, cool to 10°C, and centrifuge at 725 times gravity for 5 min. The solution temperature shall be below 20°C when finished. Read the volume percent of solids from the graduations on the tube.

DENSITY

35. Scope

35.1 This test method covers the determination of the bulk density of methylcellulose.

36. Summary of Test Method

36.1 A weighed amount of methylcellulose is transferred to a 250-mL graduated cylinder and the graduate vibrated to settle the powder.

37. Apparatus

37.1 *Vibrator*—A magnetic-type electric vibrator attached to the vertical support rod of a ring stand approximately 1 ft (0.3 m) above the base. A condenser clamp of sufficient size to hold a 250-mL graduated cylinder also shall be attached to the above rod. The base of the stand should be weighted.

38. Procedure

38.1 Place 50.0 g of methylcellulose in a 250-mL graduated cylinder and place the cylinder in the condenser clamp. Turn on the vibrator and allow the cylinder to vibrate for 3 min. Record the level (in millilitres) to which the specimen has compacted.

38.2 Alternatively, the specimen may be compacted manually. Tap it on a hard surface by dropping the cylinder repeatedly from a height of about 1 in. (25 mm), until the volume of the specimen remains constant. In order to prevent cylinder breakage, cover the tapping surface with a ¼ to ½-in. (3 to 6-mm) thick rubber sheet, or use a plastic graduated cylinder.

39. Calculation

39.1 Calculate the density, D , in grams per-millilitre as follows:

$$D = 50/\text{observed reading, mL} \quad (10)$$

40. Keywords

40.1 ball drop; cellulose esters; viscosity

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Standard Test Methods for Moisture in Cellulose¹

This standard is issued under the fixed designation D 1348; the number immediately following the designation indicates the 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 determination of moisture in cellulose using two oven-drying procedures and one Karl Fischer procedure.

1.2 The test procedures appear in the following order:

	Sections
Test Method A—Specimen Weighed in Oven	4 to 10
Test Method B—Specimen Weighed Outside of Oven	11 to 17
Test Method C—Karl Fischer Method	18 to 25

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. Significance and Use

2.1 These test methods determine the amount of moisture contained in a cellulose sample which determines the amount of bone dry cellulose present in a sample. The actual amount of cellulose in a sample is an essential entity when using cellulose as a starting material for the production of cellulose derivatives.

3. Sampling

3.1 Cellulose in a variety of forms is sampled for moisture, and no single set of directions can be given that is applicable to all types of cellulose material. The following general considerations should be borne in mind.

3.1.1 Cellulose, either in compact form, such as wood, sheeted pulp or paper, baled cotton or baled staple rayon, or in loose form such as sawdust or chips, may have an appreciably different moisture content in sections lying relatively close together. In order to secure representative samples, therefore, a bulk sample should be made up of small portions taken from various parts of the lot and having the proper proportion of edge and center material.

3.1.2 Except for those samples taken in an atmosphere with which the sample is in equilibrium, the moisture content of the sample will begin to change immediately after it is removed from its original surroundings. This change can be reduced by taking extra layers of sheeted material and discarding a few layers from the top and bottom before weighing, folding, or rolling the sample to reduce the exposed area, and by placing small samples in cans or bottles and

protecting larger samples by wrapping in rubber sheets, moistureproof cellophane, or other protective wrappings. These means do not provide continuous protection, and the test samples should be weighed as soon as possible.

3.1.3 When possible, bulk samples should be taken. These samples should weigh from 100 to 300 g, the larger samples being taken when the moisture content is low or variable. Following the initial weighing, the bulk samples should be cut up or torn into small pieces and then mixed and allowed to stand overnight or longer in a sealed container to obtain moisture equilibrium before weighing out test specimens; or the sample may be allowed to come to approximate equilibrium with the laboratory air and reweighed to determine moisture change before weighing test specimens. The latter procedure is recommended since it permits routine weighing of samples without the use of special weighing bottles or boxes, and a series of accumulated samples can be weighed simultaneously. Predrying is very desirable on samples with a high moisture content (more than 5 % above equilibrium value). When samples have been predried, calculate the moisture lost, as follows:

$$R = [M - A/A] \times 100 \quad (1)$$

where:

R = moisture, air-dry sample basis, %,
 M = original mass of the sample, g, and
 A = air-dry mass of sample, g.

3.1.4 The apparent moisture subsequently observed when testing a predried sample must be calculated on the basis of the original sample mass in order to get the original moisture content. Calculate the original mass of the air-dried sample as follows:

$$\text{Original sample mass, g} = [A \times (100 + R)]/100 \quad (2)$$

NOTE 1: *Example*—If 1000 g of bulk (wet) sample on exposure to air lose 200 g of water, the apparent moisture content is 20 % and equivalent regain is 25 %. If an 8 g specimen of the air-dry material is taken for drying in the oven, the original mass is $8 \times 1.25 = 10$ g.

TEST METHOD A—SPECIMEN WEIGHED IN OVEN

4. Scope

4.1 This test method for moisture determination is applicable to a variety of cellulose types and can be used in most cases where a sample does not contain nonaqueous material volatile at 105°C. The test method can be used for samples having either high or low moisture content.

5. Summary of Test Method

5.1 The specimen is heated to constant mass at 105°C in a ventilated gravity-convection oven, in a current of dry air, for a period of 2 h. If no dry air is used, the specimen is heated for 4 h.

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 D01.36 on Cellulose.

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Apparatus

6.1 *Oven with Built-In Weighing Equipment*—Such an oven employs a system of tared containers mounted on a table or track that can be rotated to bring specimens to a specific point in the oven, where the specimen and container can be placed on a hook or a tray connected to an outside balance. The oven must be capable of maintaining a constant temperature of $105 \pm 3^\circ\text{C}$, with an average temperature of 105°C ; continuous operation below 105°C is not satisfactory.

NOTE 2—In ovens having heating elements at the bottom only, and no cross circulation, the temperature of the lower shelves should be checked before they are used.

6.2 *Dry Air Stream*—Air, dried by passing through silica gel, aluminum oxide, concentrated sulfuric acid, or other suitable drying agent, should be passed through the oven at a rate sufficient to effect a complete change once every 2 min. If the air is dried by means of concentrated sulfuric acid, adequate traps must be provided. The use of sulfuric acid followed by perchloric drying agents should be avoided.

6.3 *Shallow Glass Weighing Bottles*—Bottles with ground-glass stoppers, measuring 30 mm high, and having a capacity of 30 mL.²

6.4 *Seamless Metal Weighing Boxes*, having a wall height, when open, preferably not over 1 in. (25.4 mm).

7. Procedure

7.1 Remove the basket, shallow pan, or other container supplied for use with the oven (Note 3). Weigh the container and place in it a specimen of 10 to 50 g weighed to the nearest 0.005 g. Designate this mass as M . Place the specimen in a tared container in the oven in such a manner that it can be reweighed without removal from the oven.

NOTE 3—Experience has shown that when yarn and fiber specimens are left in comparatively deep weighing bottles in the oven, relatively discordant results are obtained and the drying period is unnecessarily long. To avoid these difficulties specimens should be dried in containers that give the cellulose free access to the air. For fibrous or bulky materials to be weighed in an oven, use containers such as open wire grills or baskets. For small specimens to be weighed out of the oven, use wire screen baskets that will fit in weighing bottles, but if the specimen is powdery or tends to shed lint or fine short fibers, use a small weighing bottle or metal boxes.

7.2 Dry the specimen for 2 h at $105 \pm 3^\circ\text{C}$; passing a current of dry air into the bottom of the oven during the drying period. (If dry air is not forced through the oven, dry for 4 h.)

7.3 At the end of the specified period, cut off the flow of air and weigh the specimen without removing it from the oven. Continue drying for $\frac{1}{2}$ h longer, with the normal air flow, and reweigh the specimen. Repeat the drying and weighing until the mass loss between successive weighings is not more than 0.005 g, or until the specimen shows a gain in mass. Designate this mass as D .

7.4 Additional specimens should not be placed in the oven until the first specimens have attained constant mass.

8. Calculation

8.1 Calculate moisture, as measured in the oven, as follows:

$$\text{Moisture content, \%} = [(M - D)/M] \times 100 \quad (3)$$

$$\text{Moisture, dry basis (regain), \%} = [(M - D)/D] \times 100 \quad (4)$$

where:

M = original mass of specimen, and

D = mass of oven-dry specimen.

9. Report

9.1 Report the moisture in the cellulose on either or both of the following bases:

9.1.1 On the basis of the original sample, when it is termed "moisture content," "moisture as received," or "moisture as is" basis.

9.1.2 On the basis of the oven-dry cellulose, when it is termed "moisture, dry basis" or "moisture regain."

9.2 In order to avoid confusion always use the appropriate term.

10. Precision and Bias

10.1 Samples having a moisture content in the range of 5 to 15 % should show a standard deviation of 0.07 or less on replicates in a specific laboratory, and a standard deviation of 0.1 or less on replicates between laboratories.

10.2 Reproducible but less accurate values (under unfavorable conditions the absolute error may be as high as 1 %) will be obtained if the current of predried air is omitted. Since the error will be identical for all similar samples in the oven at one time, the results obtained in these cases will be comparable. The magnitude of error will vary directly with the relative humidity of the air entering the oven and with the equilibrium moisture content of samples at low relative humidity. Thus, the error will be higher on regenerated cellulose than on wood pulp or cotton, but it still would not usually be greater than 0.2 %, absolute.

TEST METHOD B—SPECIMEN WEIGHED OUTSIDE OF OVEN

11. Scope

11.1 The scope and application of this test method are the same as those of Test Method A (see Section 4).

12. Summary of Test Method

12.1 See Test Method A (Section 5).

13. Apparatus

13.1 *Oven Without Built-In Weighing Equipment*—Any ventilated oven capable of maintaining a constant temperature of $105 \pm 3^\circ\text{C}$, with an average temperature of 105°C ; continuous operation below 105°C is not satisfactory.

NOTE 4—In ovens having heating elements at the bottom only, and no cross circulation, the temperature of the lower shelves should be checked before they are used.

13.2 *Dry Air Stream*—See 6.2.

13.3 *Shallow Glass Weighing Bottles*—See 6.3.

13.4 *Seamless Metal Weighing Boxes*—See 6.4.

13.5 *Weighing Bottles*, glass with ground-glass stoppers, approximately 40 mm wide and 80 mm high.

13.6 *Wire Baskets*—Fitted baskets for weighing bottles made from approximately 15-mesh stainless steel, Monel, or other suitable screen. The height and depth of the basket will be determined by the weighing bottle used; the basket must

² Weighing bottles similar to Nos. J-555, J-560, or J-574, available from Scientific Glass Apparatus Co., 2007 Kramer Lane, Suite 100, Austin, TX 78759, have been found satisfactory for this purpose.

slide into and out of the bottle without binding. The basket should have a solid bottom, but no top is required.

14. Procedure

14.1 If the sample is free of lint, dust, or short fibers, place approximately 10 g of the sample into a previously dried and desiccated wire basket (Note 2) contained in a dry weighing bottle. Stopper the weighing bottle, and weigh to the nearest 0.001 g. Designate this mass as M . Remove the basket, containing the specimen from the weighing bottle and place the basket, weighing bottle, and stopper in the oven. If the specimen includes powder-like material, transfer the specimen directly into a small weighing bottle or can. Stopper the bottle and weigh to the nearest 0.001 g (M). Remove the stopper from the bottle and place the bottle containing the specimen and the stopper in the oven.

14.2 Dry for 2 h at $105 \pm 3^\circ\text{C}$, passing a current of dry air into the bottom of the oven during the drying period. (If dry air is not forced through the oven, dry for 4 h.)

14.3 At the end of the specified period, quickly place the basket and specimen in the weighing bottle again and stopper it (or stopper directly). Remove the weighing bottle from the oven and place it in a desiccator containing an efficient desiccant, such as anhydrous calcium sulfate.³ Allow it to cool for 1 h, momentarily open the weighing bottle to equalize the pressure, and weigh to the nearest 0.001 g.

14.4 Return the specimen to the oven, exposing it as directed above, and dry for at least 1 h more. Place it in a desiccator to cool and weigh in accordance with 14.3. Repeat the drying and weighing until the mass loss between two successive weighings is not more than 0.005 g (or until the specimen shows a gain in mass). Designate the lowest observed mass as D .

14.5 When constant mass has been obtained, discard the specimen and weigh the weighing bottle (plus basket) or can. Designate this mass as T .

14.6 Do not place additional specimens in the oven until the first specimens have attained constant mass.

15. Calculation

15.1 Calculate moisture, as measured outside of the oven, as follows:

$$\text{Moisture content, \% } [(M - D)/(M - T)] \times 100 \quad (5)$$

$$\text{Moisture, dry basis, (regain), \% } = [(M - D)/(D - T)] \times 100 \quad (6)$$

where:

M = original mass of the specimen (plus basket) and weighing bottle,

D = oven-dry mass of the specimen (plus basket), and

T = mass of the empty weighing bottle (plus basket).

16. Report

16.1 Report the moisture in the cellulose as directed in Section 9.

17. Precision and Bias

17.1 See Section 10.

TEST METHOD C—KARL FISCHER METHOD

18. Scope

18.1 This test method covers the determination of moisture in cellulose by titration with Karl Fischer reagent. The test method is applicable to all types and forms of cellulose. It is especially useful with samples containing nonaqueous material volatile at 110°C , since such substances interfere in the oven-drying methods. Anhydrides, alkalies, and large amounts of aldehydes and ketones interfere.

18.2 The Karl Fischer titration method is especially valuable where only small amounts of samples are available. The procedure lends itself to multiple determinations.

18.3 It is essential that the Karl Fischer reagent, standard water solution, and anhydrous methanol be protected from atmospheric moisture at all times. During the titration a stream of dry air or nitrogen will protect the contents of the titration flask from atmospheric moisture pick-up.

19. Summary of Test Method

19.1 An excess of Karl Fischer reagent is added to the specimen suspended in anhydrous methanol. After shaking for 15 to 20 min to extract the moisture, the excess is back-titrated with standard water solution. It is also permissible to titrate directly to the end point with Karl Fischer reagent. The end point is best detected electrometrically, but, with practice, it may be satisfactorily determined visually.

NOTE 5—To determine the moisture content of certain plastic materials the following solvents or solvent combinations may be used: chloroform, *m*-cresol, pyridine, *o*-dichlorobenzene-methanol, dioxane-methanol; dioxane-pyridine, methylene chloride-methanol (1+1), pyridine-methanol, and toluene-methanol.

20. Apparatus

20.1 *Buret*, automatic; 25 or 50 mL, attached to an amber glass reservoir so that any air entering the system must pass through an efficient absorber containing anhydrous calcium sulfate (Drierite) and soda-asbestos (Ascarite).

20.2 *Pipets*, automatic or transfer, 50- and 100-mL.

20.3 *Flasks*, or bottles, glass-stoppered, 125-mL or 250-mL, for visual end point, or suitable enclosed reaction vessel for electrometrically determined end point.

20.4 *Bottle*, dropping, or equivalent, for weighing water for standardization of reagent.

20.5 *Electrode and Meter System*, with provision for adequate stirring, and flasks or containers adapted to the assembly if the end point is to be determined electrometrically.

20.6 *Filter*, sintered glass,⁴ coarse porosity, 30-mL.

20.7 *Bottle*, weighing, low-form, 45 by 65 mm for drying wet pulp.

21. Reagents

21.1 *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-

³ Indicating Drierite, available as No. 07-578-3 from Fisher Scientific Co., 50 Faden Rd, Springfield, NJ 07081, has been found satisfactory for this purpose.

⁴ Pyrex No. 36060-30C, manufactured by Corning Glass Works, Houghton Park, Corning, NY 14830, or equivalent has been found satisfactory for this purpose.

Society, where such specifications are available.⁵ Other reagents 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.

21.2 *Air or Nitrogen*, under a pressure of 6 to 12 in. of water (1.5 to 3.0 kPa) and dried by passing through concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) followed by indicating grade anhydrous calcium sulfate or other suitable indicating desiccant. If the titration assembly provides adequate protection from atmospheric moisture, this will not be required.

21.3 *Karl Fischer Reagent*—Suitable reagent may be purchased from laboratory supply houses or prepared as follows:

21.3.1 Place 530 ± 5 mL of dry methanol (CH_3OH) in a clean, dry, 9-L chemical-resistant glass carboy. Add 2025 ± 10 mL of pyridine. Add 1270 ± 1 g of iodine crystals and shake the carboy until the iodine is completely dissolved. Place the carboy in a cracked ice bath and allow to stand about 1 h.

21.3.2 Weigh on a platform scale to 25 g (0.05 lb) a cylinder of sulfur dioxide (SO_2) with chemical-resistant glass tubing attached to the outlet with polyethylene tubing. Tilt the cylinder with the outlet down.

21.3.3 Open the cylinder valve and allow the sulfur dioxide to run into the solution in the carboy. Use the chemical-resistant glass tubing, through which the SO_2 flows, to stir the solution. When 955 ± 5 g (2.1 ± 0.05 lb), as determined by difference in scale mass reading, have run in, stopper the bottle tightly.

21.3.4 Add about 4300 mL of methylene chloride (CH_2Cl_2) and mix well. If desired, the methylene chloride may be omitted and a total of 6375 mL of pyridine used. Allow the solution to stand 24 h before use. Protect from contamination by atmospheric moisture by storing in an all-glass apparatus. Standardize daily as described in Section 22.

21.4 *Methanol*, anhydrous, water content less than 0.05%.

21.5 *Water Solution*, for use if back-titration is desired. Prepare by adding 1.0 mL of water to 1 litre of anhydrous methanol. Mix well and store in an all-glass assembly protected from contamination by atmospheric moisture. Standardize as described in Section 22.

22. Standardization of Reagents

22.1 *Back-Titration*—Standardize the Karl Fischer reagent and water solution daily. It is important that the same technique and end point be used both in the standardization and in subsequent analyses. The following directions apply to the visual detection of the end point, but with obvious modification, depending upon the particular apparatus used, they may be followed for instrumental end point detection.

22.1.1 Pass a stream of dry air or nitrogen through the flasks during all additions and titrations unless the titration assembly provides protection from atmospheric moisture.

22.1.2 Add Karl Fischer reagent to 50 mL of anhydrous methanol contained in a dry 250-mL Erlenmeyer flask until a dark brown color has been reached (A_F). Back-titrate with water solution until the end point described in Section 23 has been reached (A_W). Reserve this solution, refill burets, and accurately measure approximately 15 mL of Karl Fischer reagent into this same flask (W_F). Back-titrate with water solution as described above (W_W).

22.1.3 Weigh to the nearest 0.0001 g, by difference (conveniently from a Sattler weighing pipet), from 0.15 to 0.20 g of water (G) (Note 6) into a dry 250-mL Erlenmeyer flask containing 50 mL of anhydrous methanol. Titrate with Fischer reagent to a dark brown color (S_F), indicating an excess of 2 to 5 mL of the reagent. Back-titrate with water solution to the end point (S_W). If the end point should be passed, more Fischer reagent may be added and the titration with water solution continued until the exact end point has been reached.

Note 6—If desired, standard Rochelle salts or other material with known water of hydration, may be used instead of weighing the water directly.

22.1.4 Calculate the water equivalent, T , of the Karl Fischer reagent as follows:

$$A = W_F/W_W \quad (7)$$

where:

A = millilitres of Karl Fischer reagent equivalent to 1 mL of water solution,

W_F = millilitres of Karl Fischer reagent measured into the titrated methanol, and

W_W = millilitres of water solution required to titrate the excess Karl Fischer reagent.

$$B = A_F - (A \times A_W) \quad (8)$$

where:

B = millilitres of Karl Fischer reagent required by 50 mL of methanol,

A_F = millilitres of Karl Fischer reagent added to 50 mL of methanol, and

A_W = millilitres of water solution required to titrate the excess Karl Fischer reagent in 50 mL of methanol.

$$T = G/[S_F - (S_W \times A) - B] \quad (9)$$

where:

T = water equivalent of the Karl Fischer reagent, g/mL,

G = grams of water added (21.1.3),

S_F = millilitres of Karl Fischer reagent added to the weighed water, and

S_W = millilitres of water solution required to titrate the excess Karl Fischer reagent.

22.2 Direct Titration:

22.2.1 Alternatively, add from an automatic pipet or from an oven-dried pipet, 50 mL of methanol to each of four oven-dried titrating flasks or bottles. Weigh by difference to the nearest 0.1 mg, 0.10 to 0.25 g of distilled water from a weighing bottle fitted with a dropper, into each of two of the flasks. Titrate the solvent-water standards directly to the end point with Karl Fischer reagent (T_1). Titrate the solvent blanks to the same end point as for the standards (T_2).

22.2.2 Calculate the water equivalent of the Karl Fischer reagent as follows:

$$T = A/(T_1 - T_2) \quad (10)$$

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."

where:

- T = water equivalent of the Karl Fischer reagent, g/mL,
 A = grams of water added (22.2.1),
 T_2 = millilitres of Karl Fischer reagent required for titration of solvent blank, and
 T_1 = millilitres of Karl Fischer reagent required for titration of solvent and water.

22.2.3 Average the duplicate results and round off the average to three significant figures. Duplicate values of T should agree within 0.0001.

23. Procedure

23.1 Weigh to the nearest 0.0001 g an amount of sample estimated to contain from 0.10 to 0.15 g of water. For samples containing from 4 to 6 % water, a test specimen weighing from 2.5 to 3.0 g is satisfactory. Transfer the weighed specimen to a dry, 250-mL Erlenmeyer flask with minimum exposure to the atmosphere and add 100 mL of anhydrous methanol. Stopper the flask and shake slowly for 15 to 20 min, or allow the stoppered flask to stand for 1 h with occasional shaking.

23.2 With an oven-dried pipet, transfer 50 mL of the supernatant liquid to a 125-mL oven-dried, glass-stoppered bottle. Exposure to the atmosphere may be minimized by covering all flask openings with rubber sheeting during transfer and titration operations. Pipets and buret tips may be inserted through a small slit in the rubber sheeting.

23.3 Titrate with Karl Fischer reagent to the same end point as in the standardization. If back-titration is to be used, rapidly titrate with Karl Fischer reagent until the solution has a dark brown color. Stopper the flask and allow to stand for 15 min. If the brown color should fade during the standing period, add more Karl Fischer reagent. Record the amount of reagent added.

23.4 Pass a stream of dry air or nitrogen through the flask and titrate with water solution until the end point has been reached, as shown by an instrument or indicated visually. In the latter case, the titrated solution will have a yellow color with just a trace of brown at this point. If the end point should be overtitrated, add more Karl Fischer reagent dropwise to the first appearance of the brown color.

23.5 Alternatively, titrate directly to the end point with Karl Fischer reagent. The visual end point is a color change from light brown to dark reddish-brown.

NOTE 7—An excellent visual standard of comparison is an 0.016 N iodine solution (prepared by mixing 15 mL of 0.1 N iodine solution and 75 mL of water). The end point is best observed by examining the solution by transmitted light from a 15-W fluorescent tube.⁶ It is essential to titrate to the same end point in the standardization and the same titration.

⁶ General Electric Gold 15-W fluorescent tube, or its equivalent, has been found satisfactory for this method.

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.

23.6 Run a blank on 50 mL of the methanol by titrating as described in 23.3 and 23.4 or 23.5. The titrated alcohol may be stoppered and used as an end point color standard when the end point is detected visually.

23.7 If calculation on the dry mass basis is desired, transfer the cellulose specimen to a coarse, fritted-glass crucible previously washed with methanol or with alcohol conforming to Formula 30 of the U. S. Bureau of Internal Revenue, dried at 120°C, and weighed to the nearest 1 mg. Wash, using suction, with about 15 mL of methanol or Formula 30 alcohol. Suck as dry as possible to remove all alcohol vapors.

23.8 Place the crucible on a hot plate or steam bath (be sure the surface is clean) for 3 to 5 min to flash off any remaining alcohol, and then place it in an oven at 120°C to dry for 30 min. Cool the crucible containing the cellulose in a desiccator and weigh to the nearest 1 mg. Determine the mass of the dry pulp by difference.

24. Calculation

24.1 Calculate the percent moisture as follows:

24.1.1 For Back-Titration Method:

$$P = [(C - DA - B) \times T \times R] / W \times 100 \quad (11)$$

24.1.2 For Direct Titration Method:

$$P = [(C - B) \times T \times R] / W \times 100 \quad (12)$$

and,

$$\text{Moisture, dry basis, (regain), \%} = [P / (100 - P) \times 100] = [(W - Z) / Z] \times 100 \quad (13)$$

where:

- P = moisture content, %,
 C = millilitres of Karl Fischer reagent required for titration of the sample,
 D = millilitres of water solution added (23.4),
 A = millilitres of Karl Fischer reagent equivalent to 1 mL of the water solution,
 B = millilitres of Karl Fischer reagent required for titration of 50 mL of methanol (23.6),
 T = grams of water equivalent to 1 mL of the Karl Fischer reagent,
 W = grams of sample used,
 R = aliquot factor (Note 8), and
 Z = grams of dried cellulose.

NOTE 8—The aliquot factor is 2 when 100 mL of methanol is taken to extract the water from the cellulose and 50 mL is titrated.

25. Precision and Bias

25.1 Moisture in cellulose determined by Fischer titration is approximately 0.6 % (absolute) higher than by oven-drying, and the higher value is believed to represent the true moisture content of the sample with greater accuracy. The standard deviation assignable to a single test by a single operator is ± 0.41 .



Standard Practice for Reporting Particle Size Characteristics of Pigments¹

This standard is issued under the fixed designation D 1366; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

¹ NOTE—Keywords were added editorially in July 1991.

1. Scope

1.1 This practice for reporting the fineness characteristics of pigments is designed to apply in most cases where well-known methods for determining these particle size characteristics in the subsieve range are employed, such as microscopic, sedimentation, and turbidimetric methods; and partially to absorption and permeability methods.

1.2 Lamellar, plate-like pigments and composite pigments having a definite bimodal distribution are not considered within the scope of this practice.

1.3 *Parameters*—The fineness characteristics are reported in the following three parameters:

1.3.1 *Particle Size Parameter*.

1.3.2 *Coarseness Parameter*—A parameter descriptive of the coarseness character of the pigment, making use of a limiting value in the subsieve range similar to that used in the sieve ranges.

1.3.3 *Dispersion Parameter*—A parameter descriptive of the uniformity of the particle size distribution.

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 Document

2.1 *ASTM Standard:*

E 20 Practice for Particle-Size Analysis of Particulate Substances in the Range of 0.2 to 75 μm by Optical Microscopy²

3. Terminology

3.1 *Definitions:*

3.1.1 *particle size parameter* (specific surface diameter, *SSD*)—diameter d_3 used elsewhere in ASTM test methods. This parameter is the same as that frequently reported as "Average Particle Size by Surface Mean," and "Specific Particle Size," and is defined as follows:

$$SSD = \Sigma d^3 f / \Sigma d^2 f$$

where:

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 D01.31 on Pigment Specifications.

Current edition approved June 27, 1986. Published August 1986. Originally published as D 1366 - 55 T. Last previous edition D 1366 - 65 (1981)¹.

² *Annual Book of ASTM Standards*, Vol 14.02.

SSD = specific surface diameter, μm ,

d = mean class size, μm , and

f = frequency.

Therefore the *SSD* is the diameter of a sphere having the specific surface characteristic of the pigment. The true specific surface of all pigments involves a shape factor. Report *SSD* whether or not the effect of shape has been considered in the calculations. Presumably, as the effect of shape is better understood, it will figure more and more in calculations involving particle size, but in the meantime it will of necessity be ignored in many cases.

3.1.2 *coarseness parameter (CP)*—that diameter, expressed in micrometres, below which 99.5 % of the pigment falls.

3.1.3 *dispersion parameter (DP)*—the ratio of the micrometre size within which 50 % of the pigment lies, to the specific surface diameter, *SSD*. The larger the *DP* number, the greater the dispersion parameter and the lower the uniformity. Report the dispersion parameter in all cases when a distribution curve can be prepared from the original data. The dispersion parameter cannot be calculated from data obtained by absorption or permeability methods. Determine as follows:

3.1.3.1 Prepare a cumulative-size distribution curve on 3-phase log paper, using the vertical axis for the percent falling below the size indicated, and the horizontal axis (log scale) for diameter in micrometres. Use the upper class limit corresponding to the cumulative weight percentage. Subtract the micrometre size at 25 % cumulative weight from the micrometre size at 75 %, and multiply the difference by 100 $\div$ *SSD*, as follows:

$$DP = (\mu\text{m at } 75 \% - \mu\text{m at } 25 \%) / (SSD) \times 100$$

4. Significance and Use

4.1 This practice is of value (1) to the producer of fine particles as a means of reporting particle characteristics with respect to quality control and (2) to the buyer to assure that the particle size and particle size distribution meet his requirements.

5. Procedure

5.1 *Particle Size by Microscopical Methods*—Procedures for determining particle size by microscopical methods are described in Practice E 20. When microscopical methods are employed, report the data in tabular form similar to that shown in Table 1.

5.2 *Particle Size by Sedimentation Methods*—Sedimentation methods provide data from which may be calculated the

TABLE 1 Example of Data Sheet for Microscopic Method

Class Limits, μm		Mean Class Size, d	Frequency, f	d^2f	d^3f	$d^2f, \%$	Cumulative Weight, %
Lower	Upper						
0.25	0.75	0.5	71.4	17.8	8.9	0.007	0.007
0.75	1.25	1.0	50.9	50.9	50.9	0.040	0.047
1.25	1.75	1.5	57.9	130.2	195.4	0.154	0.191
1.75	2.25	2.0	36.9	147.5	295.0	0.233	0.424
2.25	2.75	2.5	41.1	256.8	641.9	0.506	0.93
2.75	3.25	3.0	34.5	310.9	932.7	0.738	1.69
3.25	3.75	3.5	34.5	423.2	1 481.2	1.17	2.86
3.75	4.25	4.0	21.0	336.1	1 344.5	1.06	3.92
4.25	4.75	4.5	42.0	850.8	3 828.8	3.02	6.94
4.75	5.25	5.0	81.2	2 030.8	10 154.0	8.02	14.96
5.25	5.75	5.5	283.8	8 586.3	47 225.0	37.25	52.21
5.75	6.25	6.0	155.9	5 613.4	33 680.0	26.60	78.81
6.25	6.75	6.5	71.0	2 998.1	19 487.9	15.38	94.19
6.75	7.25	7.0	7.9	368.9	2 722.4	2.15	96.34
7.25	7.75	7.5	5.1	268.8	2 166.3	1.71	98.05
7.75	8.25	8.0	4.2	268.9	2 151.4	1.71	99.76
8.25	8.75	8.5	0.5	33.7	286.8	0.23	99.99
Totals			999.8	22 733.1	126 653.1		

mean class size and percentage by weight for each of these class sizes. Table 2 presents in the second and third columns data normally obtained by sedimentation methods. Column 3 of Table 2, which gives the distribution by weight of the class sizes, is identical with the function d^3f . Therefore, the d^2f function in Column 4 equals d^3f/d . The fifth column, cumulative weight, percent, is obtained from the values in Column 3.

5.3 Particle Size by Turbidimetric Methods—Like the sedimentation method, the turbidimetric methods provide class sizes and a percentage by weight for each class size, and the calculation is the same as that in Table 2. However, frequently a weight distribution curve is obtained from data not directly convertible into the class-size distribution table. In such cases, reconstruct the size-weight distribution table from the distribution by weight curve.

5.4 Particle Size by Absorption and Permeability Methods—The absorption and permeability methods, and a few others, provide no means of making a distribution curve. Therefore, obtain specific surface either as square metres per cubic centimetre, or as square metres per gram of material. In the first case, where s equals square metres per cubic centimetre, the equation is as follows:

$$SSD = 6/s$$

In the second case, where S equals square metres per gram (Note), the equation is as follows:

$$SSD = 6/(sp \text{ gr} \times s)$$

NOTE : Example—Channel black has a specific gravity of 2.0. If the specific surface in square metres per gram is reported as 94.0, then:

$$SSD = 6/(2 \times 94) = 0.032$$

6. Calculation and Report

6.1 To determine the coarseness and dispersion parameters from the microscopical data of Table 1, draw Curve I of Fig. 1, using the cumulative weight percentage and the upper class size units of Table 1. From this curve, read the CP at the point where the curve crosses the 99.5 % line, that is, 8.7, and obtain the DP from the sizes where the curve crosses the 25 and 75 % lines; then calculate as follows:

$$SSD = 126 \ 653.1/22 \ 733.1 = 5.57 \ \mu\text{m}$$

$$p_1 = d_{75} \% - d_{25} \% = 0.70 \ \mu\text{m}$$

$$DP = 100 \ p_1/SSD = (100 \times 0.70)/5.57 = 12.6$$

$$CP = 8.7 \ \mu\text{m}$$

6.2 To determine the coarseness and dispersion parameters from the sedimentation data of Table 2, draw Curve II of Fig. 1, using the cumulative weight percentage and upper class size units of Table 2. From this curve, determine the coarseness and dispersion parameters as described in 5.1, as follows:

$$SSD = \Sigma d^3f/\Sigma d^2f = 100.0/84.47 = 1.183 \ \mu\text{m}$$

$$DP = 17.0 \ \mu\text{m}$$

$$p_2 = 5.30 - 1.18 = 4.12 \ \mu\text{m}$$

$$DP = 100 \ p_2/SSD = 348.0$$

$$CP = 17.0 \ \mu\text{m}$$

6.3 While theoretically the size of pigment particles is independent of the method of determination used, it is recognized that various methods of determining subieve particle size distribution give somewhat different results, depending on the assumption made in the particle method. It is recommended, therefore, that the method of determination always be made a part of the report of the size distribution determined under this practice.

TABLE 2 Example of Data Sheet for Sedimentation Method

Diameter Class Size Limits		Class Size Diameter, $d, \mu\text{m}$	Weight %, d^3f	d^2f	Cumulative Weight, %
Lower	Upper				
0.0	0.5	0.25	11.0	44.00	11.0
0.5	1.0	0.75	10.0	13.33	21.0
1.0	1.5	1.25	10.0	8.00	31.0
1.5	2.0	1.75	9.0	5.14	40.0
2.0	3.0	2.50	15.0	6.00	55.0
3.0	4.0	3.50	9.5	2.71	64.5
4.0	5.0	4.50	8.5	1.89	73.0
5.0	7.5	6.25	12.5	2.00	85.5
7.5	10.0	8.75	8.0	0.91	93.5
10.0	15.0	12.50	5.3	0.42	98.8
15.0	20.0	17.50	1.2	0.07	100.0
20.0	30.0	25.00	0.0	0.00	
Totals			100.0	84.47	

Keywords

microscopic; pigments; sedimentation; turbidimetric permeability absorption

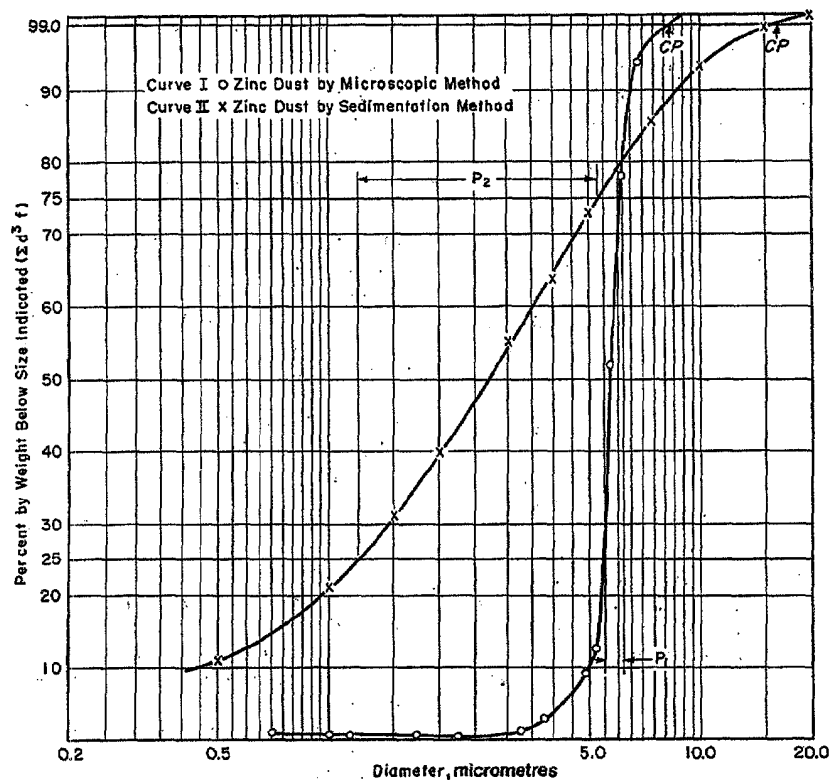


FIG. 1 Curves for Obtaining Coarseness and Dispersion Parameters

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Standard Test Methods for Chemical Analysis of White Titanium Pigments¹

This standard is issued under the fixed designation D 1394; the number immediately following the designation indicates the year of original adoption 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 which has been adopted by the Department of Defense.

^{e1} NOTE—Keywords were added editorially in June 1991.

1. Scope

1.1 These test methods cover procedures for the chemical analysis of white titanium dioxide pigments.

1.2 The analytical procedures appear in the following order:

	Sections
Preparation of Sample	4
Qualitative Analysis	5 and 6
Moisture	7
Total Titanium:	
Jones Reductor Method	8 to 12
Aluminum Reduction Method	13 to 17
Aluminum Oxide	18 to 22
Silica	23 to 29

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. A specific hazard statement is given in Section 19.*

2. Referenced Documents

2.1 ASTM Standards:

- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 1193 Specification for Reagent Water³
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴

3. Reagents

3.1 *Purity of Reagent*—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.

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

4. Preparation of Sample

4.1 The sample shall, in all cases, be thoroughly mixed and comminuted before taking portions for analysis.

QUALITATIVE ANALYSIS

5. Reagents

5.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

5.2 *Ammonium Sulfate* ((NH₄)₂SO₄).

5.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.4 *Hydrogen Peroxide* (30 %)—Concentrated hydrogen peroxide (H₂O₂).

5.5 *Hydrogen Sulfide* (H₂S).

5.6 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

5.7 *Sulfuric Acid* (1+19)—Carefully mix 1 volume of H₂SO₄ (sp gr 1.84) with 19 volumes of water.

5.8 *Tartaric Acid*.

5.9 *Tin or Zinc Metal*.

6. Procedure

6.1 Place about 0.5 g of the sample in a 250-mL glass beaker,⁶ and add 20 mL of H₂SO₄ (sp gr 1.84) and 7 to 8 g of (NH₄)₂SO₄. Mix well and boil for a few minutes. The sample should go completely into solution; a residue denotes the presence of silicon dioxide (SiO₂) or siliceous matter. Cool the solution, dilute with 100 mL of water, heat to boiling, let settle, filter, wash with hot H₂SO₄ (1+19) until free of titanium, and test the residue for lead, etc.

6.2 Test the filtrate for calcium, zinc, iron, chromium, etc., by the regular methods of qualitative analysis.⁷ For the iron determination add to a portion of the filtrate 5 g of tartaric acid, render slightly ammoniacal, pass in H₂S in

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paints and Related Coatings and Materials and are the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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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 03.05.

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."

⁶ Borosilicate glass has been found satisfactory for this purpose.

⁷ Treadwell, F. P., and Hall, William T., *Qualitative Analysis*, John Wiley & Sons, Inc., New York, NY, Vol. 1, Ninth English Ed., 1937.

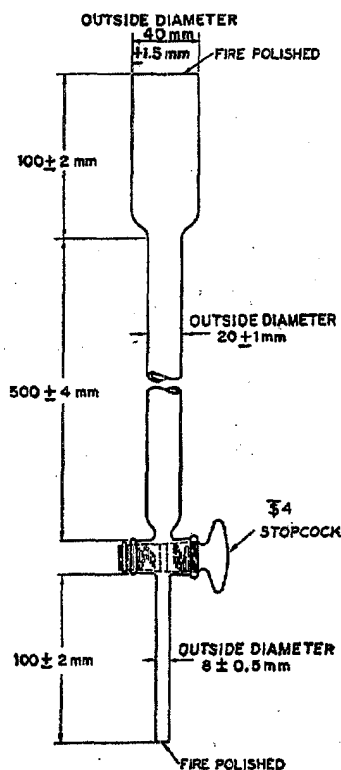


FIG. 1 Jones Reductor

excess, and digest on a steam bath. No precipitate after 30 min indicates the absence of iron, nickel, cobalt, lead, copper, etc. A black precipitate readily soluble in dilute HCl denotes iron. For titanium, test a small portion of the original filtrate with H_2O_2 (a clear yellow-orange color should result) and another portion with metallic tin or zinc (a pale blue to violet coloration should result). Negative results should be shown for sulfide, carbonate, or appreciable water-soluble matter.

MOISTURE

7. Procedure

7.1 Determine moisture and other volatile matter in accordance with Test Method A of Test Methods D 280.

TOTAL TITANIUM BY THE JONES REDUCTOR METHOD

8. Scope

8.1 This method gives results similar to those obtained with the Aluminum Reduction Method, Sections 13 to 17.

9. Apparatus

9.1 *Jones Reductor*⁸ having a zinc column at least 450 mm in length, and 19 mm in diameter (Figs. 1 and 2). The

⁸ Directions for preparing a Jones Reductor may be found in Hillebrand, W. F., et al., *Applied Inorganic Analysis*, John Wiley & Sons, Inc., New York, NY, Second Ed., 1953, p. 108.

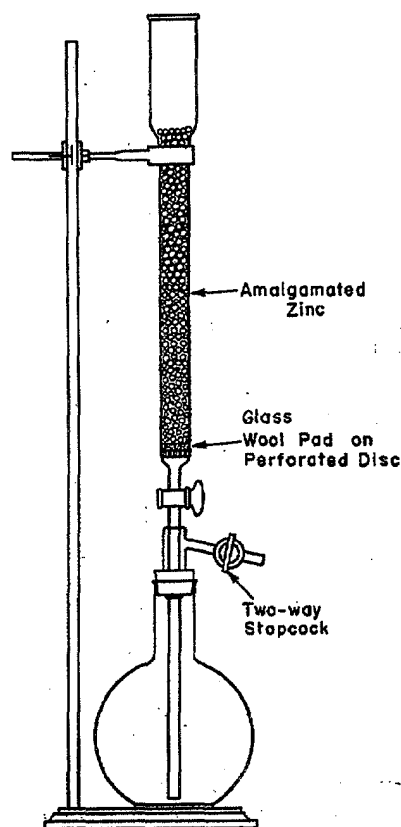


FIG. 2 Jones Reductor, Assembled

filtering pad must be tight enough to hold all the particles of amalgamated zinc resting on it, and may be made of asbestos or, preferably, glass-wool supported by platinum gauze or a perforated porcelain plate. Use the least amount (0.1 to 1.0 %) of mercury that will enable satisfactory control of hydrogen evolution, since heavy amalgamation tends to reduce the rate of reaction. Prepare the amalgam by washing 20-mesh zinc for 1 min in enough 1 N HCl to cover it, adding the proper amount of 0.25 M mercuric nitrate or chloride solution, and stirring rapidly for 3 min. Decant the solution and wash the amalgam with water and store under water to which a few drops of HCl have been added. After using, keep the reductor filled with water when not in use, in order that basic salts will not be formed and clog it.

10. Reagents

10.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

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

10.3 *Carbon Steel or Iron*—Pure iron or plain carbon steel.

10.4 *Ferric Sulfate Solution* (1 mL = 0.02 g Fe)—Dissolve 20 g of iron or carbon steel in a slight excess of HCl, oxidize with approximately 12 mL of HNO_3 , add about 80 mL of H_2SO_4 , and heat to dense white fumes. Cool, dilute with water to 1 L, digest on a steam bath until sulfates are

dissolved, and filter if necessary. To oxidize any ferrous iron that may be present, add 0.1 N KMnO_4 solution until a faint pink color persists for 5 min. Ferric ammonium sulfate ($\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) may also be used to prepare this solution (See 15.4).

10.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

10.6 *Iron or Carbon Steel*—Pure iron or plain carbon steel.

10.7 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

10.8 *Sodium Oxalate*—National Institute of Standards and Technology standard reference material No. 40 of sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$).

10.9 *Potassium Permanganate, Standard Reference Material* (0.1 N, 1 mL = 0.008 g TiO_2)—Dissolve 3.16 g of KMnO_4 in water and dilute to 1 L. Let stand 8 to 14 days, siphon off the clear solution (or filter through sintered glass, medium porosity), and standardize against the National Bureau of Standards standard sample No. 40 of sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) as follows: In a 400-mL beaker dissolve 250 to 300 mg $\text{Na}_2\text{C}_2\text{O}_4$ in 250 mL of hot water (80 to 90°C) and add 15 mL of H_2SO_4 (1+1). Titrate at once with the KMnO_4 solution, *stirring the liquid vigorously and continuously*. The KMnO_4 solution 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 solution shall not be below 60°C by the time the end point has been reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small hot plate during the titration. The use of a small type thermometer as a stirring rod is most convenient.) Keep the KMnO_4 solution in a glass-stoppered bottle painted black to keep out light or in a brown glass bottle stored in a dark place. Calculate the TiO_2 equivalent in grams of TiO_2 per millilitre of the KMnO_4 solution as follows:

$$\text{TiO}_2 \text{ equivalent} = (W \times 1.192)/V$$

where:

W = $\text{Na}_2\text{C}_2\text{O}_4$ used, g, and

V = KMnO_4 solution required for the titration, mL.

10.10 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

10.11 *Sulfuric Acid* (1+1)—Carefully mix 1 volume of H_2SO_4 (sp gr 1.84) into 1 volume of water with rapid stirring.

10.12 *Sulfuric Acid* (1+19)—Carefully mix 1 volume of H_2SO_4 into 19 volumes of water with rapid stirring.

11. Procedure

11.1 Determine the dry weight of a weighing bottle and cap to 0.1 mg. Weight to 0.1 mg 300 to 350 mg of the sample to be analyzed into the weighing bottle.

11.2 Dry the specimen in the opened weighing bottle for 2 h at 105 to 110°C. Cool in a desiccator, cap the bottle, and weigh as rapidly as possible. Calculate the dry weight of the specimen and use in the actual calculation.

11.3 Transfer the dried specimen to a dry 250 mL chemical- and heat-resistant glass beaker,⁶ add 20 mL of H_2SO_4 (sp gr 1.84) and 7 to 8 g of $(\text{NH}_4)_2\text{SO}_4$. Mix well and heat on a hot plate until dense white fumes are evolved, and

then continue the heating over a strong flame until solution is complete (usually requires not over 5 min of boiling) or it is apparent that the residue is composed of SiO_2 or siliceous matter. Caution should be observed in visually examining this hot solution. Cool the solution, dilute with 100 mL of water, stir, heat carefully to boiling while stirring, let settle, filter through paper, and transfer the precipitate completely to the paper.

11.4 Wash the insoluble residue with cold H_2SO_4 (1+19) until titanium is removed. Dilute the filtrate to 200 mL and add about 5 mL of NH_4OH to lower the acidity to approximately 10 to 15 % H_2SO_4 (by volume). Wash out the Jones reductor with H_2SO_4 (1+19) and water, leaving sufficient water in the reductor to fill to the upper level of the zinc. (These washings should require not more than one or two drops of 0.1 N KMnO_4 solution to obtain a pink color.) Empty the receiver, and put in it 25 mL of ferric sulfate solution. Reduce the prepared titanium solution as follows:

11.4.1 Run 50 mL of H_2SO_4 (1+19) through the reductor at such a uniform rate as to require 5 to 10 min for passage.

11.4.2 Follow this with the titanium solution at such a uniform rate as to require 10 min to pass through the reductor.

11.4.3 Wash out with 100 mL of H_2SO_4 (1+19).

11.4.4 Finally run through about 100 mL of water. Take care that the reductor is always filled with solution or water to the upper level of the zinc.

11.5 Gradually release the suction, wash thoroughly the glass tube that was immersed in the ferric sulfate solution, remove the receiver, and titrate immediately with 0.1 N KMnO_4 solution. Run a blank determination, using the same reagents and washing the reductor as in the above determination.

12. Calculation

12.1 Calculate the percent of TiO_2 as follows:

$$\text{TiO}_2, \% = \frac{(V_1 - B) \times T}{S} \times 100$$

where:

V_1 = KMnO_4 solution required for titration of specimen, mL

B = KMnO_4 solution required for titration of the blank, mL

T = TiO_2 equivalent of the KMnO_4 solution, g/mL, and

S = dried specimen, g.

12.2 The results calculated in accordance with 12.1 will include iron, chromium, arsenic, and any other substance that is reduced by zinc and acid. However, appreciable quantities of interfering materials are not likely to be encountered in normal, white titanium pigments.

TOTAL TITANIUM BY THE ALUMINUM REDUCTION METHOD

13. Scope

13.1 This method gives results similar to those obtained with the Jones Reductor Method (Sections 8 to 12).

14. Apparatus

14.1 *Delivery Tube*, made of about 4-mm inside diameter glass tubing bent so that there is a horizontal run of about 6

(152 mm) and a vertical drop of about 3 in. (76 mm) at one end, and a vertical drop of about 6 in. at the other end.

14.2 *Weighing Bottle*, wide-mouth, with an external-venting cap, and no larger than necessary for the required amount of sample.

5. Reagents

- 15.1 *Aluminum Metal Foil*, electrolytic grade.
- 15.2 *Ammonium Sulfate* $((\text{NH}_4)_2\text{SO}_4)$.
- 15.3 *Ammonium Thiocyanate Indicator Solution*—Dissolve 24.5 g of ammonium thiocyanate (NH_4CNS) in 80 mL of hot water, filter, bring to room temperature, and dilute to 100 mL. Keep in a well-stoppered, dark-colored bottle.
- 15.4 *Ferric Ammonium Sulfate Solution* (1 mL = 0.005 g TiO_2)—Dissolve 30.16 g of fresh ferric ammonium sulfate $(\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O})$ in 800 mL of water containing 15 mL of H_2SO_4 (sp gr 1.84). Add 5 mL of 3 % H_2O_2 and boil for at least 15 min then cool to room temperature. Dilute to exactly 1 L and mix well. Filter if cloudy. Standardize using 190 to 210 mg of NBS standard reference material No. 154 of titanium dioxide and proceeding as directed in Section 16. Calculate the TiO_2 equivalent of the solution in grams of TiO_2 per millilitre of solution, as follows:

$$\text{TiO}_2 \text{ equivalent} = (W_1 \times P) / (V_2 \times 100)$$

where:

- W_1 = National Bureau of Standards standard sample of TiO_2 used, g.
- P = percent TiO_2 in National Bureau of Standards standard sample, and
- V_2 = ferric ammonium sulfate solution required for the titration, mL.

15.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated, hydrochloric acid (HCl).

15.6 *Hydrogen Peroxide*—3 %.

15.7 *Sodium Bicarbonate Solution*—Make up a saturated solution at the time of analysis. About 10 g of sodium bicarbonate (NaHCO_3) to 90 g of water is required.

15.8 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4) .

15.9 *Titanium Dioxide* (TiO_2)—National Bureau of Standards standard sample No. 154 of titanium dioxide.

16. Procedure

16.1 Determine the dry weight of the weighing bottle and cap. Weigh to the nearest 0.1 mg, 190 to 210 mg of the sample to be analyzed into the weighing bottle.

16.2 Dry the specimen in the open weighing bottle for 2 h at 105 to 110°C. Cool in a desiccator, cap the bottle, and weigh as rapidly as possible. Calculate the dry weight of the specimen and use in the actual calculation.

16.3 Transfer the dry specimen to a 500-mL dry, wide-mouth Erlenmeyer flask. Add 7 to 9 g of $(\text{NH}_4)_2\text{SO}_4$ and 20 mL of H_2SO_4 . Mix well, heat on a hot plate until dense white fumes are evolved, and continue the heating over a strong flame until solution is complete (usually requires not over 5 min of boiling) or it is apparent that the residue is composed of SiO_2 or siliceous matter. Cool and, with caution, add 120 mL of water and 20 mL of HCl. Bring to a boil and remove from heat.

16.4 Insert the short end of the delivery tube into one hole of a two-hole rubber stopper suitable for the Erlenmeyer flask. Insert a glass rod with a slight hook or collar at the bottom end into the other hole of the stopper in such a way that the bottom end will be near the bottom of the flask when the stopper is inserted into the flask. Attach approximately 1 g of aluminum foil to the bottom end of the rod by crumpling or coiling the foil around the rod. It may be possible to use a thermometer instead of a collared glass rod and, if one ranging from 0 to 150°C is used, it can be used for determining temperature later. Insert the stopper, carrying the rod with the foil and the delivery tube, into the flask in such a way that the foil will be near the bottom of the flask at the same time that the long end of the delivery tube will be near the bottom of a 250-mL beaker containing about 150 mL of NaHCO_3 solution.

16.5 As soon as dissolution of the aluminum is complete, heat the flask to gentle boiling for 3 to 5 min without removing the delivery tube from the NaHCO_3 solution. Cool to about 60°C, preferably by partial immersion of the flask in a vessel of water. The NaHCO_3 solution should siphon into the flask during this cooling, giving an atmosphere of CO_2 over the reduced titanium solution. Withdraw the stopper, but rinse the glass rod attached to it with a little water, catching the rinse water in the flask before removing the stopper, rod, and delivery tube completely.

16.6 Add 2 mL of NH_4CNS indicator solution and titrate immediately with ferric ammonium sulfate solution (15.4) to a straw-colored end point. It is best to add the bulk of the ferric ammonium sulfate solution at once, shake well, and finish the titration drop by drop.

17. Calculations

17.1 Calculate the percent of TiO_2 as follows:

$$\text{TiO}_2, \% = (V_3 \times T_1 \times 100) / S$$

where:

V_3 = ferric ammonium sulfate solution required for titration of specimen, mL

T_1 = TiO_2 equivalent of the ferric ammonium sulfate solution, g/mL, and

S = dried specimen, g.

17.2 The results calculated in accordance with 17.1 will include chromium, arsenic, and any other substance which is reduced by aluminum and subsequently oxidized by ferric ion. However, appreciable quantities of interfering materials are not likely to be encountered in normal, white titanium pigments.

ALUMINUM OXIDE

18. Scope

18.1 This method covers the determination of aluminum oxide in titanium dioxide pigments.

19. Reagents

NOTE: Precaution—All solutions should be stored in polyethylene bottles.

19.1 *Acetic Acid*, glacial.

19.2 *Ammonium Acetate Solution* (Buffer Solution)—Dissolve 77 g of ammonium acetate in water, add 10 mL of glacial acetic acid and dilute with water to 1 L.

19.3 *Ammonium Hydroxide* (1+4)—Dilute 1 volume of concentrated ammonium hydroxide (sp gr 0.90) with 4 volumes of water.

19.4 *Ammonium Phosphate, Dibasic Solution*—Dissolve 150 g of $(\text{NH}_4)_2\text{HPO}_4$ in 700 mL of water. Adjust pH to 5.5 with HCl (1+1). Dilute with water to 1 L.

19.5 *EDTA Solution* (0.02 M)—Dissolve 7.45 g of disodium ethylenediamine tetraacetate dihydrate in water and dilute to 1 L.

19.6 *Hydrochloric Acid* (1+1)—Dilute 1 volume of concentrated hydrochloric acid (sp gr 1.19) with 1 volume of water.

19.7 *Methyl Orange Indicator Solution*—Dissolve 0.1 g of methyl orange in 100 mL of water, in accordance with Practices E 50.

19.8 *Sodium Bisulfate Monohydrate* ($\text{NaHSO}_4 \cdot \text{H}_2\text{O}$).

19.9 *Sodium Fluoride* (NaF).

19.10 *Sodium Hydroxide Solution* (6.25 M)—Dissolve 500 g of sodium hydroxide (NaOH) in water and dilute to 2 L.

19.11 *Sulfuric Acid* (1+1)—To 1 volume of water add slowly with stirring 1 volume of concentrated H_2SO_4 .

19.12 *Xylenol Orange Indicator Solution*—Dissolve 0.2 g of xylenol orange tetrasodium salt in 100 mL of water. Renew solutions weekly.

19.13 *Zinc Sulfate, Standard Solution* (0.01 M)—Dissolve 2.90 g of zinc sulfate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in water and dilute to 1 L. Standardize as follows:

19.13.1 Dissolve with the aid of heat 0.50 g of high-purity (99.8 %) aluminum wire, weighed to 0.1 mg, in 20 mL of concentrated HCl. Transfer to a 1-L volumetric flask and dilute to volume with water.

19.13.2 Place a 10-mL aliquot of this solution into a 500-mL Erlenmeyer flask containing approximately 90 mL of water and 3 mL of HCl. Add 1 drop of methyl orange indicator solution. Continue with step 20.4.

19.13.3 Calculate the titre of the ZnSO_4 solution as follows:

$$A = (18.8955 \times W_1) / V_4$$

where

A = Al_2O_3 per millilitre of ZnSO_4 solution, mL

W_1 = weight of aluminum wire dissolved in 19.13.2, g,

V_4 = ZnSO_4 solution consumed in the second titration, mL, and

$$18.8955 = \frac{\text{mol weight of } \text{Al}_2\text{O}_3 \times 10}{2 \times \text{mol weight of Al}}$$

20. Procedure

20.1 Fuse about 1 g of pigment weighed to 0.1 mg with 10 g of $\text{NaHSO}_4 \cdot \text{H}_2\text{O}$ in a 250-mL Erlenmeyer flask until the melt is clear. Use a 250-mL high-silica glass Erlenmeyer flask to prevent cracking. Do not use more sodium bisulfate than specified since excess concentrations of salt will interfere with the EDTA titration. Heat on a hot plate starting at low heat, then gradually raise the heat until full heat is reached. When the spattering has stopped and light fumes of SO_3 appear, heat the flask in the full flame of a Meker burner, with the flask tilted so that the fusion is concentrated at one end of the flask. Swirl constantly until the melt is clear. Avoid prolonged heating to prevent precipitation of titanium

dioxide. Cool and add 25 mL of H_2SO_4 (1+1). Heat until the mass has dissolved, and a clear solution results. (If silica is present, a little insoluble silica may remain.) Cool and add 120 mL of water.

20.2 Measure out 200 mL of 6.25 M NaOH solution. Add 65 mL of this NaOH solution to the sample solution while stirring constantly with a magnetic stirrer. Pour the remaining NaOH solution into a 500-mL volumetric flask. Slowly, and with constant stirring, add the sample solution to the NaOH solution. Police with water, cool, and dilute to volume. (If the procedure is delayed at this point for more than 2 h, transfer the contents of the volumetric flask to a polyethylene bottle.) Either centrifuge for 5 min, or allow most of the precipitate to settle out, then filter the supernatant liquid through a very fine filter paper until a little more than 100 mL have been collected.

20.3 Place a 100-mL aliquot of the above solution in a 500-mL Erlenmeyer flask, add 1 drop of methyl orange indicator solution and acidify with HCl (1+1) until the color changes to red; add approximately 3 mL in excess.

20.4 Add 25 mL of EDTA solution. (If the approximate alumina level is known, use the following mathematical formula for determining the amount of EDTA to add for best results: $4 \times \% \text{Al}_2\text{O}_3 + 5 = \text{mL of 0.02 M EDTA}$.) Add, dropwise, NH_4OH (1+4) until the solution color is just completely changed from red to orange-yellow. Add 10 mL of buffer solution and 10 mL of $(\text{NH}_4)_2\text{HPO}_4$ solution, boil for 5 min, and cool quickly to room temperature in running water. Add 3 drops of xylenol orange indicator solution. If the solution is purple, yellow-brown, or pink, bring the pH to 5.3 to 5.7 with acetic acid. If the pH is correct, a pink color indicates insufficient EDTA; repeat with a new aliquot, starting with 20.3 and using 50 mL of EDTA solution in 20.4.

20.5 Titrate with ZnSO_4 solution to a yellow-brown or pink end point. This titration should be performed quickly near the end point by rapidly adding 0.2-mL increments until the first color change occurs. This color will fade in 5 or 10 s, but is the true end point. This step is critical, and failure to observe the first color change will result in an incorrect value. The fading end point does not occur in the second titration. This first titration must be greater than 8 mL of ZnSO_4 solution. For most accurate work this first titration should require 10 to 15 mL of ZnSO_4 solution.

20.6 Add 2 g of NaF, boil for 2 to 5 min, and cool in running water. Titrate the EDTA, released from its aluminum complex by the fluoride, with ZnSO_4 solution to the same end point as in 20.5.

21. Calculation

21.1 Calculate the aluminum oxide content of the pigment sample as follows:

$$A = (Z \times T) / (2 \times S)$$

where:

A = percent Al_2O_3 ,

Z = ZnSO_4 solution consumed in the second titration, mL

T = Al_2O_3 per millilitre of ZnSO_4 solution, g, and

S = specimen used, g.

22. Precision

22.1 Based on interlaboratory studies the following cri-

aria should be used for judging the acceptability of results at the 95 % confidence level:

22.1.2 *Repeatability*—Two results obtained by the same operator on the same sample should be considered suspect if they differ by more than 0.22 % relative.

22.1.3 *Reproducibility*—Two results, each the mean of duplicates obtained by operators in different laboratories should be considered suspect if they differ by more than 0.62 % relative.

SILICA

23. Scope

23.1 This method covers the determination of silica in titanium dioxide (TiO_2) pigments.

24. Summary of Method

24.1 The fusion of TiO_2 pigment with sodium bisulfate leaves only the silica insoluble when the melt is dissolved in sulfuric acid. To assure no loss of the silica the sulfuric acid is taken to fuming to dehydrate the silica. The silica content is determined by volatilizing the silica in the weighed filtration residue with hydrofluoric acid.

25. Apparatus

- 25.1 *Erlenmeyer Flask*, 250-mL, high silica.
- 25.2 *Filter Paper*, very fine, ashless, acid washed.
- 25.3 *Platinum Crucible and Cover*.
- 25.4 *Oven*, controlled at 120°C.
- 25.5 *Muffle Furnace*, controlled at $1000 \pm 25^\circ\text{C}$.

26. Reagents

- 26.1 *Hydrofluoric Acid* (sp gr 1.15)—Concentrated hydrofluoric acid (HF).
- 26.2 *Sodium Bisulfate* ($\text{NaHSO}_4 \cdot \text{H}_2\text{O}$).
- 26.3 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).
- 26.4 *Sulfuric Acid* (1+1)—To 1 volume of water add slowly with stirring 1 volume of concentrated H_2SO_4 .
- 26.5 *Sulfuric Acid* (1+9)—To 9 volumes of water add slowly with stirring 1 volume of concentrated H_2SO_4 .

27. Procedure

27.1 Transfer 1 g of pigment weighed to 0.1 mg to a 250-mL high silica Erlenmeyer flask containing 10 g of $\text{NaHSO}_4 \cdot \text{H}_2\text{O}$. If an SiO_2 content in excess of 5 % is expected a 0.5-g specimen of pigment may be used to facilitate complete fusion with 10 g of $\text{NaHSO}_4 \cdot \text{H}_2\text{O}$.

27.2 Heat over a Meker burner, frequently swirling the flask until decomposition and fusion is complete and clear (except for SiO_2). *Be careful of overheating at start and of spattering of the fusion.*

27.3 Allow to cool and to the cold melt, add 25 mL of H_2SO_4 (1+1), and heat very carefully and very slowly until the fusion is dissolved. Carefully evaporate to fumes of H_2SO_4 .

27.4 Cool and carefully add 150 mL of water. Pour very small amounts of water down the sides of the flask with frequent swirling of the contents to avoid overheating and spattering. Let cool and filter through fine ashless filter paper, using a 60° gravity funnel.

27.5 Wash out all silica from the flask onto the filter paper with H_2SO_4 (1+9). Police the flask carefully.

27.6 Place the filter paper in a platinum crucible and dry in a 120°C oven. Heat the partly covered crucible over a bunsen burner. Avoid flaming the filter paper by heating first the cover from above and then the crucible from below. When the filter paper is consumed, heat at 1000°C for 30 min in a muffle furnace. Cool in a desiccator and weigh the crucible.

27.7 Add 2 drops of H_2SO_4 (1+1) and 5 mL of HF (sp gr 1.15). Carefully evaporate to dryness, first on a low heat hot plate to remove the HF and then over a bunsen burner to remove the H_2SO_4 . Avoid spattering, especially after removal of the HF.

27.8 Ignite at 1000°C for 10 min. Cool in a desiccator and weigh the crucible again. The difference in weight is silica.

28. Calculation

28.1 Calculate the silica content as follows:

$$\text{SiO}_2, \% = (W_2/S_3) \times 100$$

where:

W_2 = SiO_2 found, g, and

S_3 = specimen used, g.

29. Precision

29.1 On the basis of an interlaboratory test of this test method in which six laboratories tested, in duplicate, five samples of titanium dioxide ranging in silica content from 1.5 to 8.2 %, within-laboratory standard deviation was found to be 1.79 % and between-laboratories standard deviation was found to be 3.44 %. Based on this, the following criteria should be used for judging the precision of results at the 95 % confidence level:

29.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 5.1 % relative.

29.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 9.7 % relative.

30. Keywords

30.1 aluminum oxide; aluminum reduction; Jones Reductor; titanium pigment, chemical analysis

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Standard Test Methods for Chemical Analysis of Poly(Vinyl Butyral)¹

This standard is issued under the fixed designation D 1396; the number immediately following the designation indicates the year of original adoption 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 renumbering of Sections 4 through 6 in November 1987.

1. Scope

1.1 These methods cover procedures for the determination of poly(vinyl alcohol), poly(vinyl acetate), and butyraldehyde in poly(vinyl butyral).

1.2 The procedures appear in the following order:

	Sections
Poly(vinyl alcohol)	4
Poly(vinyl acetate)	5
Butyraldehyde	6

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. 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.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.

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

4. Poly(Vinyl Alcohol)

4.1 Reagents:

4.1.1 *Ethylene Dichloride*, technical grade.

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 Varnish and Resins, Including Shellac.

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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."

4.1.2 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %), methanol, or isopropanol.

4.1.3 *Potassium Hydroxide, Standard Alcoholic Solution* (0.5 N)—Dissolve 33 g of KOH in methanol and dilute to 1 L. Standardize against potassium acid phthalate using phenolphthalein indicator solution.

4.1.4 *Pyridine Acetic Anhydride Reagent*—Mix slowly 1000 mL of pyridine and 87 mL of acetic anhydride. Make only about a week's supply, and keep it in a brown bottle.

4.2 Procedure:

4.2.1 Transfer 2.2 g of the dry sample to a clean, dry, 500-mL glass-stoppered flask. Add 25.0 mL of pyridine-acetic anhydride reagent. Insert the stopper, and heat the flask on a sand or steam bath below the boiling point for 5½ h. Swirl gently until the sample is completely dissolved. Vent the flask occasionally during the first part of the heating period to prevent the stopper from blowing out.

4.2.2 At the end of the 5½-h period, add 25 mL of ethylene dichloride and shake well. Add 100 mL of water, and shake vigorously immediately after adding the water. Let the flask stand for ½ h.

4.2.3 Add a few drops of phenolphthalein solution and titrate with 0.5 N alcoholic KOH solution. Shake vigorously during the titration.

4.2.4 *Blank*—Run a blank determination on the reagents, following the same procedure as for the sample.

4.3 *Calculation*—Calculate the percentage of poly(vinyl alcohol) as follows:

$$\text{Poly(vinyl alcohol), \%} = [(B - V) N \times 4.4] / S$$

where:

B = KOH solution required for titration of the blank, mL,

V = KOH solution required for titration of the sample, mL,

N = normality of the KOH solution, and

S = specimen weight, g.

5. Poly(Vinyl Acetate)

5.1 Apparatus:

5.1.1 *Flask, Wide-Mouth*, 500-mL capacity, equipped with a metal reflux condenser.

5.2 Reagents:

5.2.1 *Hydrochloric Acid Standard Alcoholic Solution* (0.5 N).

5.2.2 Methanol:

5.2.3 *Phenolphthalein Solution* (10 g/L)—See 4.1.2.

5.2.4 *Potassium Hydroxide Alcoholic Solution* (28 g KOH/L)—Dissolve 33 g of potassium hydroxide (85 % KOH in ethyl alcohol and dilute to 1 L with methanol. This

solution is approximately 0.5 *N* so standardized solution (4.1.3) may be used if desired.

5.3 Procedure:

5.3.1 Transfer 2.15 g of the dry sample to the 500-mL flask. Add 200 mL of methanol, measured in a graduate. Measure from a buret 25.0 mL of KOH solution (4.2.4). Reflux on a sand or water bath for 2 h.

5.3.2 Rinse down the condenser and flask with a small amount of water, add a few drops of phenolphthalein solution, and backtitrate, using 0.5 *N* alcoholic HCl.

5.3.3 *Blank*—Run a blank determination on the reagents, following the same procedure as for the sample.

5.4 *Calculation*—Calculate the percentage of poly(vinyl acetate) as follows:

$$\text{Poly(vinyl acetate), \%} = [(B - V)N \times 8.6]/S$$

where:

B = HCl required for back-titration of the blank, mL,
V = HCl required for back-titration of the sample, mL,
N = normality of the HCl, and
S = sample used, g.

6. Total Butyraldehyde

6.1 Reagents:

6.1.1 *Bromphenol Blue Indicator Solution*—Dissolve 0.2 g of bromphenol blue indicator solution in 100-mL of methanol, ethanol, or isopropanol.

6.1.2 *n-Butanol*.

NOTE 1—The same lot of *n*-butanol should be used for all determinations that are to be compared.

6.1.3 *Hydroxylamine Hydrochloride Solution* (0.5 *N*).

6.1.4 *Methanol*.

6.1.5 *Sodium Hydroxide Standard Solution* (0.5 *N*)—Dissolve 20 g of NaOH in water and dilute to 1 L. Standardize against potassium acid phthalate.

6.2 Blank Determination:

6.2.1 Place 50 mL of *n*-butanol and 50 mL of the hydroxylamine hydrochloride solution in a 500-mL Erlenmeyer flask, and fit by means of the ground-glass joint to a 12-in. (305-mm) reflux condenser. Be sure the ground-glass joint is dry. Add a porous plate boiling chip, and reflux for 2 h on a hot plate. Cool to 24 ± 2°C.

6.2.2 Add exactly 5 drops of bromphenol blue indicator solution and 50 mL of methanol to the solution and titrate with 0.5 *N* NaOH solution to a green end point (Note 2). Run at least two blank determinations checking to within 0.5

mL of each other, titrating to the same end point at 24 ± 2°C (Note 3).

NOTE 2—The pH value of the blank and sample vary with temperature; if the temperature is raised the green end point will turn yellow and if the temperature is lowered the green end point will turn blue. The pH value of the blank changes more quickly than the pH of the sample solution for a given change in temperature. Therefore, the blank is titrated and kept at 24 ± 2°C and the sample is titrated at the same temperature.

NOTE 3—The color change of the indicator will be a gradual one from yellow to green to blue. No sharp transition point will be found. Choose a point of yellow green to green coloration. A buffer solution containing 2.05 g of potassium acid phthalate 24 mL of 0.1 *N* H₂SO₄, and 50 mL of methanol gives a color matching a blank titrated with 6.85 mL of 0.0938 *N* NaOH solution. The color in the buffer solution will fade out but the regular blank may be kept as a standard for the determination.

6.3 Procedure for Analysis of Sample:

6.3.1 Transfer 2 g of the dry sample, weighed to the nearest 0.1 mg, to the 500-mL Erlenmeyer flask. Add 50 mL of *n*-butanol and 50 mL of hydroxylamine hydrochloride solution, and attach the 12-in. (305-mm) reflux condenser. Be sure the ground-glass joint is dry. Add a boiling chip, and reflux for 2 h.

NOTE 4—The resin gradually forms a milky emulsion. At this point, the reaction tends to bump and must be watched. After approximately 1.5 h the bumping lessens considerably and the mixture clears.

6.3.2 Cool, add 5 drops of bromphenol blue solution and titrate with 0.5 *N* NaOH solution until at least two-thirds of the expected requirement has been run in. Add 50 mL of methanol. At the first appearance of a green tinge, bring the flask to 24 ± 2°C (Note 5). Titrate to the same color as the blank (Note 6).

NOTE 5—The heat of neutralization changes the temperature during the titration.

NOTE 6—The blank may be diluted to give the same intensity of color as in the analysis of the sample if the percentage of butyraldehyde in all determinations is fairly constant. Otherwise, adjust the intensity of color by the amount of indicator present.

6.5 *Calculation*—Calculate the percent of total butyraldehyde as follows:

$$\text{Butyraldehyde, \%} = [(V - B)N \times 7.2]/S$$

where:

V = NaOH solution required for titration of the specimen, mL,
B = NaOH solution required for titration of the blank, mL,
N = normality of the NaOH solution, and
S = specimen weight, g.

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Standard Test Method for Unsaponifiable Matter in Alkyd Resins and Resin Solutions¹

This standard is issued under the fixed designation D 1397; the number immediately following the designation indicates the year of original adoption 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 7012.1 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 covers the determination of unsaponifiable matter in alkyd resins and resin solutions. This test method is not applicable to alkyd resins containing modifying agents such as urea, melamine, phenols, rosin, and styrene.

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 4.

2. Referenced Document

- 2.1 *ASTM Standard:*
D 1193 Specification for Reagent Water²

3. Significance and Use

3.1 The unsaponifiable matter in alkyd resins controls the properties of the final film.

4. Apparatus

4.1 *Aluminum Beaker*, having a capacity of 125 mL.³

4.2 *Flask and Condenser*—A 200-mL Erlenmeyer flask fitted with a water-cooled glass reflux condenser. The connection between the flask and condenser shall be a standard 24/40 taper ground-glass joint.

4.3 *Separatory Funnels*—Three 500-mL capacity fitted with standard-taper, ground-glass stoppers and stopcocks. Stopcocks should be lubricated sparingly with ether-insoluble stopcock grease. Alternatively, funnels fitted with tetrafluoroethylene (TFE-fluorocarbon) stopcocks may be used.

4.4 *Steam Bath.*

4.5 *Vacuum Drying Oven*—A small, laboratory-size vacuum oven, thermostatically controlled to operate at 80 $\pm$ 5°C. A water aspirator vacuum source is satisfactory.

5. Reagents and Materials

5.1 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.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.

5.2 Unless otherwise indicated references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

5.3 *Benzene.*

5.4 *Benzene-Alcohol Mixture*—Mix equal volumes of benzene and ethyl alcohol, add 2 drops of phenolphthalein indicator solution, and neutralize with 0.02 *N* sodium hydroxide (NaOH) solution to a persistent faint pink color.

5.5 *Ethyl Alcohol (95 volume %)*—Pure ethyl alcohol or denatured alcohol conforming to Formula No. 2B of the U. S. Bureau of Internal Revenue.

5.6 *Ethyl Ether.*

5.7 *Phenolphthalein Indicator Solution (10 g/L)*—Dissolve 1 g of phenolphthalein in ethyl alcohol (95 %) and dilute to 100 mL with ethyl alcohol.

5.8 *pH Indicator Paper*, universal type.

5.9 *Sodium Hydroxide Solution (50 %)*—Dissolve sodium hydroxide (NaOH) in an equal weight of water.

5.10 *Sodium Hydroxide, Standard Solution (0.02 N)*—Prepare and accurately standardize a 0.02 *N* aqueous NaOH solution.

6. Procedure

6.1 Weigh by difference, from a closed container into the 200-mL Erlenmeyer flask, a portion of resin or resin solution containing from 0.05 to 0.2 g of unsaponifiable matter (Note 1) (8 to 10 g of resin solution usually is sufficient).

NOTE 1—The maximum specimen size is limited to 10 g of nonvolatile matter; otherwise saponification or separation difficulties may arise. The specimen should be weighed to the nearest milligram.

6.2 Add 10 mL of benzene, and warm to dissolve the sample. Add 50 mL of alcohol, swirl gently to mix, and then add slowly 5 mL of the NaOH solution (50 %), while

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.

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² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ Aluminum beakers, Catalog No. 2100, manufactured by the A. H. Thomas Co., W. Washington Square, Philadelphia, PA 19105, have been found satisfactory for this purpose.

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."

stirring gently. Add 5 mL of water, attach to the condenser, and allow to reflux gently on the steam bath for 2 h.

6.3 Remove from the heat source, cool to room temperature, and wash down the condenser and joint with a few millilitres of water from a wash bottle. Transfer the contents of the flask to a 500-mL separatory funnel with the aid of water from the wash bottle. Finally, rinse the flask with three 25-mL portions of ether, adding the ether washes to the sample in the separatory funnel. Add sufficient water to bring the volume of the lower aqueous layer to 300 mL, and add 10 mL of alcohol.

6.4 Stopper the separatory funnel, shake gently, and allow the layers to separate. Draw off the lower aqueous layer into the second separatory funnel (Note 2). Continue the extraction of the aqueous layer with successive 20-mL portions (not less than three) until a colorless ether extract is obtained, combining the ether extracts in the first funnel, and using the second and third funnels for the successive extractions.

NOTE 2—If the layers do not separate easily, carefully draw off the lower, clear, aqueous layer and add 2 to 3 mL of alcohol, by means of a pipet, to the ether-emulsion phases in the separatory funnel. Swirl gently to break the emulsion, and continue to draw off the lower layer. This procedure for breaking the emulsion may be repeated on subsequent extractions, if necessary.

6.5 Wash the final combined ether extracts with 25-mL portions of water until the washings are neutral when tested with the indicator paper or solution. Transfer the final ether extract portion-wise into the 125-mL beaker containing a small boiling stone and previously weighed to the nearest 1 mg, evaporating the ether from each portion on the steam bath (Note 3). (Precaution—See Note 4.) Finally, rinse the separatory funnel with a few successive millilitres of ether, adding these washes to the extract in the beaker.

NOTE 3—The metal top of the steam bath should be covered with clean aluminum foil to prevent corrosion of the aluminum beaker during the evaporation.

NOTE 4: Precaution—In addition to other precautions, be sure to use a hood.

6.6 Evaporate the final portion of ether; then transfer the beaker and its contents to the vacuum oven, previously heated to 80°C. Heat to constant weight, allowing to cool to room temperature in a desiccator before weighing.

6.7 After weighing, take up the residue in 50 mL of warm (approximately 50°C) benzene-alcohol mixture. Titrate with 0.02 N NaOH solution to the same persistent faint pink color as in the neutralization of the benzene-alcohol mixture (see 5.3).

7. Calculation and Report

7.1 Calculate the unsaponifiable matter as follows, and report the results to the nearest 0.1 %:

$$F = VN \times 0.280$$

where:

F = fatty acids in extract, g,

V = NaOH solution required for titration of the residue, mL,

N = normality of the NaOH solution, and

0.280 = the factor used for normal 18-carbon atom fatty acids. If cocoanut, lauric, pelargonic, or other short chain fatty acids are suspected or known to be present, use the arbitrary factor 0.216 in the above equation.

$$U = [(R - F)/S] \times 100$$

where:

U = unsaponifiable matter, %,

R = residue, g, and

S = specimen, g.

8. Precision and Bias

8.1 On the basis of an interlaboratory study of this test method in which the within-laboratory standard deviation was found to be 0.08 % absolute and the between-laboratory standard deviation was found to be 0.11 % absolute 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, should be considered suspect if they differ by more than 0.25 % 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 0.33 % absolute.

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Standard Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions¹

This standard is issued under the fixed designation D 1398; the number immediately following the designation indicates the year of original adoption 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 test method covers the gravimetric determination of the total fatty acids in alkyd resins and alkyd resin solutions. The test method is not applicable to alkyd resins containing modifying agents such as urea, melamine, phenols, rosin, and styrene.

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. Specific precaution statements are given in 5.4.1 and 6.1.*

2. Referenced Documents

2.1 ASTM Standards:

- D 563 Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions²
- D 1193 Specification for Reagent Water³
- D 1615 Test Methods for Glycerol, Ethylene Glycol, and Pentaerythritol in Alkyd Resins²
- D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints⁴

3. Significance

3.1 This test method is used to determine total fatty acid content of alkyd resins and this solution, in the absence of interfering compounds, as a means whereby the relative applicability of the alkyd resin to the particular end use may be estimated by buyer and seller.

4. Apparatus

- 4.1 *Beakers*, having capacities of 150 and 400 mL.
- 4.2 *Desiccator*, containing concentrated H_2SO_4 as the desiccant.
- 4.3 *Filter Paper*, rapid, low-ash filter paper, to fit the filtering funnel.
- 4.4 *Filtering Funnel*, 75-mm diameter, long-stem.

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.

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

³ *Annual Book of ASTM Standards*, Vols 06.01 and 11.01.

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

4.5 *Flask*, 250-mL, flat-bottom, with standard taper opening.

4.6 *Nitrogen Gas Supply*.

4.7 *Separatory Funnels*, three 500-mL capacity glass-stoppered, fitted with standard-taper ground-glass stoppers and stopcocks.

4.8 *Steam Bath*.

4.9 *Water Bath*.

4.10 *Vacuum Drying Oven*, small, laboratory-size, thermostatically controlled to operate at $60 \pm 2^\circ C$. A water-aspirator vacuum source is satisfactory.

4.11 *Cotton*, absorbent, USP.

5. Reagents and Materials

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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

5.3 *Ether*, anhydrous.

5.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.4.1 *Caution*—Concentrated hydrochloric acid is corrosive and may cause burns to the skin and eyes; the vapor is irritating to mucous membranes. Use care in handling this acidic substance. In case of contact, flush skin or eyes with plenty of water while removing contaminated clothing. Call a physician. Wash clothing before reuse. See supplier's Material Safety Data Sheet for further information.

5.5 *Indicator Paper*, universal-type.

5.6 *Sodium Sulfate* (Na_2SO_4), anhydrous.

6. Procedure

6.1 Saponify a portion of resin or resin solution in accordance with Test Method D 563. After filtering the potassium phthalate alcoholate (6.3), transfer the combined

⁵ "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."

filtrate and washings to the 400-mL beaker with the aid of 25 mL of water from a wash bottle. Concentrate to a volume of approximately 25 mL on the steam bath under a blanket of nitrogen to prevent oxidation of the fatty acids. (Caution—be sure to use a hood.) Transfer to a 500-mL separatory funnel with the aid of water from a wash bottle, dilute with water to approximately 300 mL, and add 10 mL of alcohol.

6.2 Extract the unsaponifiable and volatile thinners with successive 50-mL portions of ether (not less than three, or until a colorless ether extract is obtained), combining the ether extracts in the first separatory funnel and using the other two funnels for the successive extractions (Note 1). Finally, wash the combined ether extracts with three 15-mL portions of water, adding the water washes to the main aqueous phase. Discard the combined ether extracts.

NOTE 1—If the layers do not separate easily, carefully draw off the lower, clear aqueous layer and add 2 to 3 mL of alcohol, by means of a pipet, to the ether emulsion phases in the separatory funnel. Swirl gently to break the emulsion and continue to draw off the lower layer. This procedure for breaking the emulsion may be repeated on subsequent extractions, if necessary.

6.3 Acidify the aqueous phase to a pH of approximately 2 by slowly adding HCl, cooling under running tap water. When the mixture has cooled to room temperature, extract the fatty acids with successive 25-mL portions (not less than three) of ether until a colorless ether extract is obtained, combining the ether extracts in the first separatory funnel and using the other two funnels for the successive extractions. Wash the combined ether extracts with successive 10-mL portions of water until free of mineral acid when tested with an indicator paper. Discard the aqueous phase (unless Test Method D 1615 is being followed).

6.4 Dry the combined ether extracts in the separatory funnel by the addition of successive small quantities of anhydrous Na_2SO_4 .

NOTE 2—The free water has been removed when, by the addition of a small quantity of Na_2SO_4 and gentle swirling, the excess Na_2SO_4 is seen to disperse as a freely moving powder.

6.5 By either of the following methods, filter the ether extract portionwise into a tared (to 1 mg) 250-mL standard-taper flat-bottom flask or 150-mL beaker, containing a small boiling aid.

6.6 *Method A*—Filter the dried ether extract, portionwise through a rapid, low-ash paper by decanting from the top opening of the separatory funnel into the appropriate receiver. Cover the filter funnel with a watch glass between each filtration step.

6.7 *Method B*:

6.7.1 Invert the separatory funnel and open the stopcock, allowing any water in the bore of the stopcock to run back into the funnel.

NOTE 3—A small amount of ether applied to and evaporated from the outside of the funnel before the cock is opened will assure that the water will drain back into the funnel.

6.7.2 Close the cock and then shake the contents of the funnel sufficiently for the Na_2SO_4 to absorb all the water. If needed, add more Na_2SO_4 and gently swirl the funnel and contents. Swab out the stem of the funnel with a piece of cotton attached to a stiff wire.

NOTE 4—A small brass screw soldered to the end of a stiff brass wire will serve for this purpose.

6.7.3 Insert a clean plug of cotton moistened with ether into the funnel stem and filter the ether solution through the cotton plug into the appropriate receiver.

NOTE 5—Experience will show how tightly packed the cotton must be to hold back the Na_2SO_4 and still allow a sufficient flow of ether extract.

6.8 Evaporate the ether portionwise from the beaker or distill it from the flask on the steam bath, in a hood. Apply a blanket of nitrogen over the beaker during evaporation. Remove the last portions of fatty acids from the Na_2SO_4 in the funnel by washing with successive small portions of ether until a colorless extract is obtained. Remove the final traces of fatty acids from the filter paper or cotton plug by using several successive small portions of ether.

6.9 If a gas chromatographic analysis of the separated fatty acids is to be performed, observe the precautions given in 7.2 of Method D 2245.

6.10 Complete the evaporation of the fatty acid-ether solution on the steam bath, while maintaining a nitrogen atmosphere over the acids. Remove the final traces of ether by heating for successive 20-min periods in a vacuum oven at 60°C until minimum weight is obtained. After each heating period, allow the beaker and contents to cool in a desiccator and then weigh to 1 mg.

7. Calculation and Report

7.1 Calculate the percent total fatty acids content, T , as follows, and report the results to 0.1 %:

$$T = \frac{A - B}{S} \times 100$$

where:

A = weight of beaker plus residue,

B = weight of beaker, and

S = specimen weight taken for analysis.

8. Precision

8.1 On the basis of an interlaboratory study of this test method, the within-laboratory standard deviation was found to be 0.31 and the between-laboratories standard deviation was found to be 0.50. 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 same operator should be considered suspect if they differ by more than 0.9 % absolute.

8.1.2 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 1.5 % absolute.

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Standard Test Methods for Sodium Carboxymethylcellulose¹

This standard is issued under the fixed designation D 1439; the number immediately following the designation indicates the year of original adoption 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 2 was added editorially subsequent sections were renumbered in November 1989.

Scope

- 1.1 These test methods cover the testing of sodium carboxymethylcellulose.
- 1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 8
Degree of Etherification:	
Test Method A—Acid Wash	9, 10 to 15
Test Method B—Nonaqueous Titration	9, 16 to 21
Viscosity	22 to 27
Purity	28 to 34
Sodium Glycolate	35 to 42
Sodium Chloride	43 to 49
Density	50 to 54

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 Notes 1 and 2.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 1347 Test Methods for Methylcellulose²
E 1 Specification for ASTM Thermometers³

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.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.

3.2 Unless otherwise indicated, references to water shall be understood to mean distilled water.

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 D01.36 on Cellulosics.

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

³ *Annual Book of ASTM Standards*, Vol 14.03.

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."

MOISTURE

4. Scope

- 4.1 This test method covers the determination of the volatile content of sodium carboxymethylcellulose.
- 4.2 The results of this test are used for calculating the total solids in the sample; and, by common usage, all materials volatile at this test temperature are designated as moisture.

5. Apparatus

- 5.1 *Oven*—Gravity convection oven, capable of maintaining a temperature of $105 \pm 3^\circ\text{C}$.
- 5.2 *Weighing Bottles*, low-form, 50-mm inside diameter by 30-mm height, or equivalent.
- 5.3 *Analytical Balance*.

6. Procedure

- 6.1 Weigh 3 to 5 g of the sample to the nearest 0.001 g in a tared and covered weighing bottle.
- 6.2 Place the bottle in an oven at 105°C for 2 h with the cover removed. Cool the bottle in a desiccator, replace the cover, and weigh.
- 6.3 Replace the sample in the oven for 30 min, cool, and reweigh.
- 6.4 Continue this procedure to a mass loss of not more than 5 mg for 30 min drying time.

7. Calculation

- 7.1 Calculate the percent moisture, M , as follows:

$$M = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, g, and
 B = sample used, g.

8. Precision

- 8.1 Statistical analysis of interlaboratory test results on samples containing 2 to 10 % moisture indicates a precision of ± 0.2 % absolute at the 95 % confidence level.

DEGREE OF ETHERIFICATION

9. Scope

- 9.1 These test methods cover the determination of the degree of etherification (D.E.) of sodium carboxymethylcellulose.

9.2 Two test methods are included as follows:

- 9.2.1 *Test Method A (Acid Wash)*, for crude and purified grades of sodium carboxymethylcellulose with degrees of

etherification up to 0.85. Above 0.85 degree of etherification, slightly low results may be obtained.

9.2.2 *Test Method B (Nonaqueous Titration)*, for purified grades of sodium carboxymethylcellulose of all degrees of etherification. It is not applicable to the crude grades.

Test Method A—Acid Wash

10. Summary of Test Method

10.1 The water-soluble sodium carboxymethylcellulose is converted to the insoluble acid form, purified by washing, dried, and then a weighed sample is reconverted to the sodium salt with a measured excess of sodium hydroxide.

11. Apparatus

- 11.1 *Stirrer*, air-driven.
- 11.2 *Buchner Funnel*, 75-mm, fitted with a 70-mm fine-texture, heavy-duty filter paper. A 60-mm medium-porosity, fritted glass funnel may also be used.
- 11.3 *Drying Oven*, maintained at 105°C.

12. Reagents

12.1 *Diphenylamine Reagent*—Dissolve 0.5 g of diphenylamine in 120 mL of sulfuric acid (H_2SO_4 , 9+2). The reagent should be essentially water-white. It will give a deep blue coloration with traces of nitrate or other oxidizing agents.

12.2 *Ethyl Alcohol* (95 volume %)—Denatured ethyl alcohol conforming to either Formula 2B, 3A, or 30 of the U. S. Bureau of Internal Revenue.

12.3 *Ethyl Alcohol* (80 % by volume)—Dilute 840 mL of Formula 2B, 3A, or 30 denatured alcohol to 1 L with water.

12.4 *Hydrochloric Acid, Standard* (HCl, 0.3 to 0.5 N).

12.5 *Methanol*, anhydrous.

12.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

12.7 *Sodium Hydroxide, Standard Solution* (0.3 to 0.5 N)—Prepare and standardize a 0.3 to 0.5 N solution of sodium hydroxide (NaOH).

12.8 *Sulfuric Acid* (9+2)—Carefully mix 9 volumes H_2SO_4 with 2 volumes of water.

13. Procedure

13.1 Weigh approximately 4 g of the sample into a 250-mL beaker and add 75 mL of ethyl alcohol (95 %). Stir the mixture with an air-driven stirrer until a good slurry is obtained. Add 5 mL of HNO_3 , while agitating, and continue agitation for 1 to 2 min. Heat the slurry and boil for 5 min. (Warning—See Note 1.) Remove the heat and continue agitation for 10 to 15 min.

NOTE 1: Warning—Exercise care to avoid fire.

13.2 Decant the supernatant liquid through the filter and transfer the precipitate to the filter with 50 to 100 mL of ethyl alcohol (95 %). Wash the precipitate with ethyl alcohol (80 %) that has been heated to 60°C, until all of the acid has been removed.

13.3 Test for the removal of acid and salts (ash) by mixing a drop of the acid carboxymethylcellulose slurry from the filter with a drop of diphenylamine reagent on a white spot plate. A blue color indicates the presence of nitrate and the necessity for further washing. If the first drop of reagent does not produce a blue color, further drops should be added until

an excess of reagent is known to be present, noting the color after each drop. Four to six washings will usually suffice to give a negative test for nitrate.

13.4 Finally, wash the precipitate with a small amount of anhydrous methanol and draw air through it until the alcohol is completely removed. Transfer the precipitate to a glass or aluminum weighing dish provided with a cover. Heat the uncovered dish on a steam bath until the odor of alcohol can no longer be detected (in order to avoid fires due to methanol fumes in the oven), then dry the dish and contents, uncovered for 3 h at 105°C. Place the cover on the dish and cool to room temperature in a desiccator.

13.5 The sulfate ash content of the sample at this point should be less than 0.5 % when determined on 0.5 g of the sample by the procedure given in the Ash as Sulfate section of Test Methods D 1347. If the ash content is greater than 0.5 %, rewash the sample with ethyl alcohol (80 %). If necessary, repeat the procedure described in 13.1 to 13.4.

13.6 Weigh, to the nearest 0.01 g, about 1 to 1.5 g of the dried acid carboxymethylcellulose (depending on the normality of the acid and base to be used) into a 500-mL Erlenmeyer flask. Add 100 mL of water and 25.00 mL of 0.3 to 0.5 N NaOH solution, while stirring. Heat the solution to boiling, and boil for 15 to 30 min.

13.7 Titrate the excess NaOH, while the solution is hot, with the 0.3 to 0.5 N HCl to a phenolphthalein end point.

14. Calculation

14.1 Calculate the degree of etherification, G , as follows:

$$A = (BC - DE)/F \quad (2)$$

$$G = 0.162A/(1 - 0.058A) \quad (3)$$

where:

- A = milliequivalents of acid consumed per gram of sample,
- B = NaOH solution added, mL,
- C = normality of the NaOH solution,
- D = HCl required for titration of the excess NaOH, mL,
- E = normality of the HCl,
- F = acid carboxymethylcellulose used, g,
- 162 = gram molecular mass of the anhydroglucose unit of cellulose, and
- 58 = net increase in molecular mass of anhydroglucose unit for each carboxymethyl group substituted.

15. Precision

15.1 The precision of this test method is estimated to be ± 0.03 D.E. units.

Test Method B—Nonaqueous Titration

16. Summary of Test Method

16.1 This measurement is based upon a nonaqueous acid-base titration. The sample is refluxed with glacial acetic acid, and the resulting sodium acetate is titrated with a standard solution of perchloric acid in dioxane, to a potentiometric end point. Impurities containing alkaline sodium will also be titrated under these conditions. Sodium chloride does not interfere.

Apparatus

- 17.1 *pH Meter*, equipped with a standard glass electrode and a calomel electrode modified as follows:
- 17.1.1 Discard the aqueous potassium chloride solution, then rinse and fill with the calomel electrode solution as described in 18.2.
- 17.1.2 Add a few crystals of potassium chloride and silver chloride or silver oxide to the electrode.
- 17.2 *Buret*, micro, 10-mL capacity.

Reagents

- 18.1 *Acetic Acid*, glacial.
- 18.2 *Calomel Electrode Solution*—Add 2 g of potassium chloride (KCl) and 2 g of silver chloride (AgCl) or silver oxide (Ag₂O) to 100 mL of methanol and shake thoroughly to saturate. Use the supernatant liquid.
- 18.3 *1,4-Dioxane*.⁵
- 18.4 *Perchloric Acid* (0.1 *N*)—Add 9 mL of concentrated perchloric acid (HClO₄, 70 % to 1 L of dioxane, with stirring (Warning—See Note 2). Store in an amber glass bottle. Any slight discoloration that appears on standing may be disregarded.

NOTE 2: Warning—The solution of perchloric acid in dioxane should never be heated or allowed to evaporate.

18.4.1 Standardize the solution as follows: Dry potassium acid phthalate for 2 h at 120°C. Weigh 2.5 g to the nearest 0.0001 g into a 250-mL volumetric flask. Add glacial acetic acid, shake to dissolve, and then make up to volume and mix thoroughly. Pipet 10 mL into a 100-mL beaker and add 50 mL of acetic acid. Place on a magnetic stirrer and insert the electrodes of the pH meter. Add nearly the required amount of HClO₄ from a buret, then decrease the increments to 0.05 mL as the end point is approached. Record the millilitres of titrant versus millivolts, and continue the titration a few millilitres beyond the end point. Plot the titration curve and read the volume of titrant at the inflection point. Calculate the normality, *N*, as follows:

$$N = (A \times 10 \times 1000) / (B \times 204.22 \times 250) \quad (4)$$

where:

- A* = potassium acid phthalate used, g,
B = HClO₄ added, mL,
 204.22 = gram molecular mass of potassium acid phthalate,
 10 = potassium acid phthalate solution added, mL, and
 250 = glacial acetic acid used to dissolve potassium acid phthalate, mL.

18.5 *Potassium Acid Phthalate*, primary standard, National Institute of Standards and Technology Standard Sample No. 84.

Procedure

19.1 Weigh 0.2 g of the sample, to the nearest 0.0001 g, into a 250-mL Erlenmeyer flask with ground-glass joint. Add 75 mL of acetic acid, connect to a water-cooled condenser, and reflux gently on a hot plate for 2 h.

19.2 Cool, and transfer the solution to a 250-mL beaker

with the aid of 50 mL of acetic acid. Place on the magnetic stirrer and titrate to a potentiometric end point with 0.1 *N* HClO₄ in accordance with 18.4.

Calculation

20.1 Calculate the degree of etherification, *H*, as follows (Note 3):

$$M = (AN \times 100) / (G \times (100 - B)) \quad (5)$$

$$H = 0.162 M / (1.000 - (0.080 M)) \quad (6)$$

where:

- M* = milliequivalents of acid consumed per gram of sample,
A = HClO₄ added, mL,
N = normality of HClO₄,
G = sample used, g,
B = percent moisture, determined on a separate sample, in accordance with Sections 4 to 7,
 162 = gram molecular mass of an anhydroglucose unit of cellulose, and
 80 = net increase in molecular mass of an anhydroglucose unit for each sodium carboxymethyl group added.

NOTE 3—The result calculated in accordance with Section 18 includes the alkaline sodium from sodium glycolate; however, if the latter is less than 0.5 %, the interference is negligible.

Precision

21.1 Statistical analysis of interlaboratory test results indicates the precision of this test method as shown below:

Approximate D.E. Level	Precision, D.E. Units (95 % Confidence Level)
0.40	±0.010
0.80	±0.012
1.35	±0.038

VISCOSITY

Scope

22.1 This is an arbitrary test method for determining the viscosity of aqueous solutions of sodium carboxymethylcellulose in the viscosity range from 10 to 10 000 cP at 25°C.

22.2 The concentration to be used for the test should be agreed upon between the purchaser and the seller. It should be such that the viscosity of the solution will fall within the range of this test.

22.3 The results for the viscosity of sodium carboxymethylcellulose by this test method will not necessarily check with results from other types of instruments used for viscosity measurements.

22.4 The determinations are run on a calculated dry basis; that is, the amount of sodium carboxymethylcellulose required for the desired concentration on a dry basis is calculated from the known moisture content.

22.5 This test method is intended for referee purposes. The Brookfield spindles and speeds given in Table 1 are recommended for this purpose, but slight derivations from the table may occasionally be found convenient for individual application.

⁵ 1,4-Dioxane available as Eastman Kodak Catalog No. 2144 or Mateson, Coleman, and Bell Catalog No. CB 368 has been found satisfactory for this purpose.

TABLE 1 Viscometer Spindles Required for Given Speeds

Viscosity Range, cP	Spindle No.	Speed, rpm	Scale	Factor
10 to 100	1	60	100	1
100 to 200	1	30	100	2
200 to 1000	2	30	100	10
1000 to 4000	3	30	100	40
4000 to 10000	4	30	100	200

23. Apparatus

23.1 *Viscometer*, Brookfield type.⁶

23.2 *Container*—Glass jar, approximately 2½-in. (60-mm) in diameter and 5¼ in. (133 mm) deep, unstricted at the top, capacity 8 oz (2.3 m³).

23.3 *Analytical Balance*.

23.4 *Mechanical Stirrer*—Stirrer constructed of either stainless steel or glass (Fig. 1)⁷ attached to a variable speed motor capable of operating at 800 ± 100 rpm under varying load conditions.

23.5 *Water Bath*, constant-temperature, set at 25°C and capable of maintaining that temperature within ±0.2°C.

23.6 *Thermometer*—ASTM Saybolt Viscosity Thermometer having a range from 19 to 27°C and conforming to the requirements for Thermometer 17C, as prescribed in Specification E 1.

24. Procedure

24.1 Determine moisture in accordance with Sections 4 to 7.

24.2 Calculate the dry-basis sample mass, M , in grams necessary to make 240 g of test solution as follows:

$$M = 100A / (100 - B) \quad (7)$$

where:

A = desired dry mass of sample, g, and

B = moisture in the sample as received, %.

24.3 Calculate the quantity of distilled water required as follows:

$$V = 240 - S \quad (8)$$

where:

V = volume of distilled water, mL, and

S = mass of sample, g.

24.4 Add the calculated quantity of water to the jar. Position the stirrer in the jar allowing minimum clearance between the stirrer and the bottom of the container.

24.5 Begin stirring and slowly add the sodium carboxymethylcellulose specimen. Adjust the stirring speed to approximately 800 ± 100 r/min and mix for exactly 2 h. Do not allow the stirring speed to exceed 900 r/min since higher speeds tend to affect viscosity on certain grades of sodium carboxymethylcellulose.

NOTE 4—If the specimen is added too rapidly, agglomeration will occur. This may prevent complete dissolution within the required mixing time.

⁶ Model LVG, Brookfield Engineering Laboratories, Inc., Stoughton, MA or equivalent, has been found satisfactory for this test method.

⁷ Stirrers made with 1½-in. (38-mm), three-bladed propellers available from A. H. Thomas Co., P.O. Box 779, Philadelphia, PA 19105, Catalog No. 9240K have also been found satisfactory for this purpose.

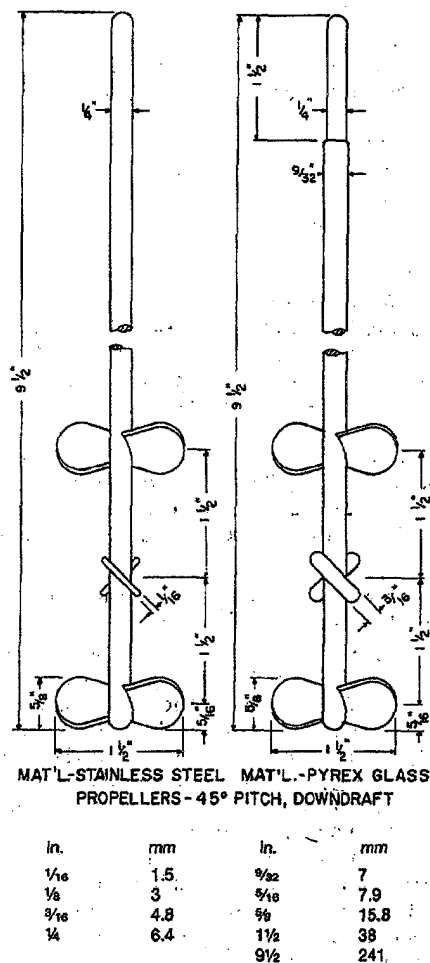


FIG. 1 Stirrer Blade

24.6 Remove the stirrer and transfer the specimen container to the constant-temperature bath for 1 h. Check the specimen temperature with a thermometer at the end of 1 h to ensure that the test temperature has been reached.

24.7 Remove the specimen container from the bath and shake vigorously for 10 s. Measure the viscosity with the Brookfield viscometer, selecting the proper spindle and speed from Table 1. Allow the spindle to rotate until a constant reading is obtained.

25. Calculation

25.1 Calculate the viscosity, V , in centipoises as follows:

$$V = \text{reading} \times \text{factor} \quad (9)$$

26. Report

26.1 Report the results as Brookfield viscosity at 25°C, stating the solution concentration and the spindle and speed used.

27. Precision

27.1 The difference between the average of the results

obtained by a given operator using a given viscometer and the average of the results obtained by a different operator using a different viscometer should not exceed 10 % of the mean of the averages.

PURITY OF CRUDE SODIUM CARBOXYMETHYLCELLULOSE

28. Scope

28.1 This test method covers the determination of purity or percent of active ingredient in crude sodium carboxymethylcellulose containing no phosphate. The test method has been standardized on materials having a degree of etherification of about 0.85 or less.

28.2 For determination of purity of refined sodium carboxymethylcellulose (purity approximately 98 % or higher), analysis for individual or combined impurities and calculation of purity by difference will give more reliable results.

29. Summary of Test Method

29.1 A 3-g sample is stirred mechanically in a beaker for 15 min with each of two 150-mL portions of ethanol (80 % by volume) at a temperature of 60 to 65°C. The supernatant liquid is decanted through a tared filtering crucible after each treatment. The undissolved matter is transferred quantitatively to the crucible, dried, weighed, and calculated as percent of sodium carboxymethylcellulose. The temperature of the ethanol during the leaching need not be closely controlled, but the concentration of the ethanol must be closely controlled (sp gr within 0.001).

30. Apparatus

30.1 *Filtering Crucible*, fritted glass, medium-porosity, 50-mL capacity.⁸

30.2 *Mechanical Stirring Motor*, electric or air-driven, with any convenient stirrer of appropriate size.

30.3 *Water Bath*, constant-temperature, maintained at 60 to 65°C.

30.4 *Cover*—A lid to keep a 400-mL beaker substantially covered during mechanical stirring in bath. A flanged lid, preferably of stainless steel, with a slot, wide enough to pass the shaft of the mechanical stirrer, cut from the rim to the center, has been found satisfactory. The center should be cut out somewhat larger than the shaft of the stirrer to permit free rotation of the stirrer. Such a lid serves to weight down the beaker as well as to minimize the evaporation losses during leaching.

31. Reagents

31.1 *Ethanol* (95 volume %)—Undenatured or specially denatured ethanol conforming to Formula 2B (Note 5) of the U. S. Bureau of Internal Revenue.

NOTE 5—Other grades of denatured alcohol, such as Formula 3A, are not satisfactory for this purpose.

31.2 *Ethanol* (80 volume %)—Dilute 840 mL of 95 % ethanol (30.1) to 1 L with water. The specific gravity should be 0.857 ± 0.001 at 25/25°C. If necessary, add water or

ethanol until the specific gravity is within the specified limits.

31.3 *Ethyl Ether*, anhydrous, ethanol-free.

32. Procedure

32.1 Weigh 3 to 5 g of the sample into a tared low-form, 65-mm diameter glass weighing dish fitted with a cover. Dry to constant mass at $105 \pm 1^\circ\text{C}$ in either a gravity or a mechanical convection oven. Weigh at the end of an initial 2-h heating period, then continue with 30-min heating periods until the change in mass during a 30-min heating period is not more than 0.10 %. If there is an increase in mass of the sample during one or more drying periods, record the lowest mass observed as the mass for use in the calculation of moisture content. Calculate the loss in mass as the percent of moisture in the sample.

32.2 Weigh 3 ± 0.1 g of the sample, in the "as-received" condition, to the nearest 0.001 g and transfer to a 400-mL beaker.

32.3 Add 150 mL of ethanol (80 %) that has been heated to between 60 and 65°C, and immediately place the beaker in a constant-temperature water bath maintained at 60 to 65°C. The level of the water in the bath should be somewhat higher than the level of the liquid in the beaker. Cover the beaker as completely as possible with a lid that will permit mechanical stirring. Lower a mechanical stirrer almost to the bottom of the beaker, and stir for 10 min at a rate suitable to provide good agitation without spattering material on the walls of the beaker above the liquid level.

32.4 Stop the stirrer. Allow the undissolved matter to settle with the beaker still in the bath, and then decant the hot supernatant liquid as completely as possible through a tared, fritted-glass filtering crucible.

32.5 Add 150 mL of ethanol (80 %), at 60 to 65°C to the beaker and proceed in accordance with 32.3 and 32.4.

32.6 After decanting the supernatant liquid as completely as possible, transfer the insoluble matter to the crucible with the aid of ethanol (80 %) at 60 to 65°C in a wash bottle, being careful to scrape all insoluble matter from the lid, the stirrer, and the beaker. A total of about 250 mL of ethanol (80 %) will normally be required to transfer the insoluble matter to the crucible and to further wash the insoluble matter in the crucible. During the operations prescribed in this paragraph, apply suction only while filtration is in progress. Make every effort to avoid drying-out of the filter cake. If fines appear to pass through the filter, use only gentle suction.

32.7 Wash the residue in the crucible with 50 mL of ethanol (95 %) at room temperature, and finally with several portions of ether at room temperature (Note 6). Without permitting suction to continue longer than necessary, place the crucible in a beaker or weighing bottle on the steam bath until no odor of ether can be detected.

NOTE 6—Thorough washing with ether is necessary to remove ethanol completely from the insoluble matter. If ethanol is not completely removed before oven drying, it may not be completely removed during the oven drying.

32.8 Place the crucible in an oven at $105 \pm 1^\circ\text{C}$ for 1 h. Stir the contents of the crucible with a dissecting needle or thin rod (preferably of smooth-surfaced metal) to break up the cake and facilitate complete drying. Again, dry at $105 \pm 1^\circ\text{C}$ for 1 h. Place the crucible in a desiccator. Cover it with a flat glass plate, weighing bottle cover, or other suitable cover

⁸ Corning No. 416120 available from A. H. Thomas Co., P. O. Box 779, Philadelphia, PA 19105, Catalog No. 4142-C 50 M, has been found satisfactory for this purpose.

to minimize absorption of moisture from the atmosphere in the desiccator; and cool to room temperature (at least 30 min). Weigh the uncovered crucible as rapidly as possible.

32.9 Dry the crucible for additional 1-h periods until the change in mass during a 1-h drying period does not exceed 0.003 g. If increases in mass are observed after such additional drying periods, record the lowest mass observed as the mass of the crucible plus dry sodium carboxymethylcellulose.

33. Calculation

33.1 Calculate the percent sodium carboxymethylcellulose, S , on the dry basis as follows:

$$S = (A \times 10\,000)/(B(100 - C)) \quad (10)$$

where:

A = mass of dried residue, g,

B = mass of specimen used, g, and

C = moisture in the specimen as received, %.

34. Precision

34.1 Statistical analysis of interlaboratory test results indicate a precision of $\pm 0.6\%$ absolute at the 95 % confidence level.

SODIUM GLYCOLATE

35. Scope

35.1 This test method covers the determination of the sodium glycolate content of purified sodium carboxymethylcellulose containing not more than 2.0 % sodium glycolate.

36. Summary of Test Method

36.1 The sodium carboxymethylcellulose is dissolved in acetic acid (50 %), precipitated with acetone and sodium sulfate and the insoluble material filtered off. The filtrate containing the sodium glycolate (as glycolic acid) is treated to remove the acetone and reacted with 2,7-dihydroxy naphthalene. The resulting color is measured at 540 nm with a spectrophotometer calibrated with known solutions.

37. Apparatus

37.1 *Spectrophotometer or Filter Photometer*, suitable for measuring absorbance at 540 nm.

37.2 *Absorption Cells*, for spectrophotometer, 1-cm light path.

37.3 *Aluminum Foil*—Cut to approximately 2-in. (51-mm) squares.

38. Reagents

38.1 *Acetic Acid*, glacial.

38.2 *Acetone*.

38.3 *Dihydroxy Naphthalene Reagent* (0.100 g/L)—Dissolve 0.100 g of 2,7-dihydroxy naphthalene in 1 L of sulfuric acid (H_2SO_4). Before using, allow the solution to stand until the initial yellow color disappears. If the solution is very dark, discard it and prepare a new solution from a different supply of H_2SO_4 . This solution is stable for approximately 1 month if stored in a dark bottle.

38.4 *Glycolic Acid, Standard Solution* (1 mg glycolic acid/mL)—Dry several grams of glycolic acid in a desiccator at room temperature overnight. Accurately weigh 0.100 g of

the dried material, dissolve in water, and make up to volume in a 100-mL volumetric flask. This solution will contain 1 mg of glycolic acid/mL. The solution is stable for approximately 1 month.

38.5 *Sodium Sulfate* (Na_2SO_4).

38.6 *Sulfuric Acid* (sp gr 1.84)—Concentrated H_2SO_4 .

39. Preparation of Calibration Curve

39.1 Into a series of five 100-mL volumetric flasks accurately introduce 1, 2, 3, and 4-mL aliquots of the standard glycolic acid solution, reserving the fifth flask for a blank. Add sufficient water to each flask to give a total volume of 5 mL. Add 5 mL of glacial acetic acid, make up to volume with acetone and mix. These solutions will contain 0, 1, 2, 3 and 4 mg of glycolic acid, respectively.

39.2 Pipet 2 mL of each of these solutions into individual 25-mL volumetric flasks. Place the uncovered flasks upright in a boiling water bath for exactly 20 min to remove the acetone. Remove the flasks from the bath and cool.

39.3 To each flask add 20 mL of 2,7-dihydroxy naphthalene reagent as follows: Add 5 mL of reagent initially, mix thoroughly, then add the remaining 15 mL of reagent and mix. Cover the mouth of the flasks with a small piece of aluminum foil and place upright in the boiling water bath for 20 min. Remove from the bath, cool, and make up to volume with H_2SO_4 .

39.4 Measure the absorbance of each solution at 540 nm against the blank solution. Plot the milligrams of glycolic acid in the original 100 mL of solution against absorbance to give a calibration curve.

40. Procedure

40.1 Weigh about 0.5 g of the sample (0.2 g for semirefined grades) to the nearest 0.001 g and transfer to a 100-mL beaker. Moisten the specimen thoroughly with 5 mL of acetic acid followed by 5 mL of water, and stir with a glass rod until solution is complete (usually about 15 min is required). Slowly add 50 mL of acetone, stirring during addition, followed by approximately 1 g of NaCl. Stir several minutes to ensure complete precipitation of the carboxymethylcellulose.

40.2 Filter through a soft, open-texture paper, previously wetted with a small amount of acetone, and collect the filtrate in a 100-mL volumetric flask. Use an additional 30 mL of acetone to facilitate transfer of the solids and to wash the filter cake. Make up to volume with acetone and mix.

40.3 Prepare a blank solution containing 5 mL of water and 5 mL of glacial acetic acid in another 100-mL volumetric flask. Make up to the mark with acetone and mix.

40.4 Pipet 2 mL of the solution from the specimen and 2 mL of the blank solution into separate 25-mL volumetric flasks. Develop the color and measure the absorbance in accordance with 39.2 to 39.4.

40.5 Using the observed absorbance, refer to the calibration curve and read the corresponding milligrams of glycolic acid.

41. Calculation

41.1 Calculate the percent sodium glycolate content, C , as follows:

$$C = (B \times 12.9)/[W \times (100 - A)] \quad (11)$$

where:

- A = glycolic acid, read from the calibration curve, mg,
 W = sample used, g,
 M = moisture in the sample as received, %, and
 2.9 = (gram molecular mass of sodium glycolate per gram molecular mass of glycolic acid) $\times 10$.

42. Precision

42.1 Statistical analysis of intralaboratory data on material containing less than 0.50 % sodium glycolate indicate a precision of ± 0.03 % absolute at the 95 % confidence level.

SODIUM CHLORIDE

43. Scope

43.1 This test method covers the determination of the sodium chloride content of purified sodium carboxymethylcellulose.

44. Summary of Test Method

44.1 The sodium carboxymethylcellulose is dissolved in water and titrated with a standard solution of silver nitrate to a potentiometric end point. Hydrogen peroxide is added to reduce the viscosity of the solution.

45. Apparatus

45.1 *pH Meter*, equipped with a silver electrode and a mercurous sulfate-potassium sulfate electrode.

45.2 *Buret*, micro, 10-mL capacity.

46. Reagents

46.1 *Hydrogen Peroxide* (30 mass %)—Concentrated hydrogen peroxide (H_2O_2).

46.2 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

46.3 *Silver Nitrate, Standard Solution* (0.1 *N*)—Dissolve 17.0 g of silver nitrate ($AgNO_3$) in 1 L of water. Store in an amber glass bottle. Standardize the solution as follows:

46.3.1 Dry the sodium chloride ($NaCl$) for 2 h at 120°C. Weigh 0.65 g to the nearest 0.0001 g, into a 250-mL beaker and add 100 mL of water. Place on a magnetic stirrer, add 10 mL of HNO_3 , and insert the electrodes of the pH meter. Add nearly the required amount of $AgNO_3$ solution from a buret, then decrease the increments to 0.05 mL as the end point is approached. Record the millilitres of titrant versus millivolts, and continue the titration a few millilitres beyond the end point. Plot the titration curve and read the volume of titrant at the inflection point. Calculate the normality, N , as follows:

$$N = (A \times 1000) / (B \times 58.45) \quad (12)$$

where:

- A = $NaCl$ used, g,
 B = $AgNO_3$ solution added, mL, and
 58.45 = gram molecular mass of $NaCl$.
 46.4 *Sodium Chloride* ($NaCl$).

47. Procedure

47.1 Weigh 5 g of the sample, to the nearest 0.0001 g, into

a 250-mL beaker. Add 50 mL of water and 5 mL of H_2O_2 (30 %). Place the beaker on a steam bath, stirring occasionally to achieve a nonviscous solution. If solution is not complete after 20 min, add 5 mL more of H_2O_2 and heat until solution is complete.

47.2 Cool the beaker, add 100 mL of water and 10 mL of HNO_3 . Place it on the magnetic stirrer and titrate to a potentiometric end point with 0.1 *N* $AgNO_3$ solution in 46.3.

48. Calculation

48.1 Calculate the percent sodium chloride content, C , as follows:

$$C = (AN \times 584.5) / [G \times (100 - B)] \quad (13)$$

where:

- A = $AgNO_3$ solution added, mL,
 N = normality of $AgNO_3$ solution,
 G = sample used, g,
 B = moisture, determined on a separate sample, %, in accordance with Sections 4 to 7, and
 584.5 = gram molecular mass of $NaCl \times 10$.

49. Precision

49.1 The precision of this test method is estimated to be ± 0.05 % absolute for material containing less than 0.50 % sodium chloride and ± 0.10 % absolute for material containing greater than 0.59 % sodium chloride.

DENSITY

50. Scope

50.1 This test method covers the determination of the bulk density of sodium carboxymethylcellulose.

51. Summary of Test Method

51.1 A weighed amount of sodium carboxymethylcellulose is transferred to a 250-mL graduated cylinder and the graduate vibrated to settle the powder.

52. Apparatus

52.1 *Vibrator*—A magnetic-type electric vibrator attached to the vertical support rod of a ring stand approximately 1 ft (0.3 m) above the base. A condenser clamp of sufficient size to hold a 250-mL graduated cylinder also shall be attached to the above rod. The base of the stand should be weighted.

53. Procedure

53.1 Place 50.0 g of sodium carboxymethylcellulose in a 250-mL graduated cylinder and place it in the condenser clamp. Turn on the vibrator and allow the cylinder to vibrate for 3 min. Record the level (in millilitres) to which the specimen has compacted.

53.2 Alternatively, the specimen may be compacted manually. Tap it on a hard surface by dropping the cylinder repeatedly from a height of about 1 in. (25 mm) until the volume of the specimen remains constant. In order to prevent cylinder breakage, cover the tapping surface with a $\frac{1}{8}$ to $\frac{1}{4}$ -in. (3 to 6-mm) thick rubber sheet or use a plastic graduated cylinder.

54. Calculation

$$D = 50/r$$

(14)

54.1 Calculate the density, D , in grams per millilitre as follows:

where:

r = observed reading, mL.

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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 Rosin Acids Content of Coating Vehicles¹

This standard is issued under the fixed designation D 1469; the number immediately following the designation indicates the year of original adoption 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 1988.

1. Scope

1.1 This test method covers the determination of total rosin acids content of rosin esters, varnishes, and alkyd resins, unmodified by such materials as maleic or fumaric acid, or phenols. Total rosin acids determined include free rosin, esterified rosin, and metallic salts of rosin.

1.2 This test method is primarily designed for material containing 0.5 to 5 % rosin on the nonvolatile basis.

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

D 1259 Test Methods for Nonvolatile Content of Resin Solutions³

D 1644 Test Methods for Nonvolatile Content of Varnishes⁴

3. Summary of Test Method

3.1 The specimen is saponified with potassium hydroxide-ethylene glycol reagent and acidified with hydrochloric acid. Heat is applied to hydrolyze metallic driers. This is necessary, especially when metallic rosins are present.

3.2 The mixture described in 3.1 is extracted with benzene (Note 1). The rosin and fatty acids and unsaponifiables pass into the benzene layer. The aqueous layer will contain certain dibasic acids, polyhydric alcohols, and other water-soluble products of saponification.

NOTE 1—While it has not been evaluated for this procedure, toluene has been found to be an acceptable alternative in similar procedures.

3.3 The benzene is removed by evaporation, the residue weighed, and the rosin acids are determined by a selective

esterification and titration method.⁵

4. Apparatus

4.1 *Air Condenser*, 30 in. (760 mm), with a 24/40 standard-taper joint.

4.2 *Buret*, automatic type, having a capacity of 25 mL, for the standard potassium hydroxide solution, fitted with soda-lime traps to protect against absorption of atmospheric carbon dioxide (CO₂).

4.3 *Erlenmeyer Flasks*, 250 and 500-mL capacity, with 24/40 ground joint.

4.4 *Separatory Funnels*, three of 1-L capacity.

4.5 *Steam Bath*, located in a fume hood, for evaporation of volatile solvents.

4.6 *Moisture Collection Trap*, constructed according to details shown in Fig. 1. Wrap with 1/2-in. (12.7-mm) asbestos tape.

4.7 *Pipet*, automatic, 50-mL capacity.⁶

5. Reagents and Materials

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.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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

5.3 *Benzene*—(Toluene may be an acceptable substitute, see Note 1.)

5.4 *Butyl Alcohol-Sulfuric Acid Esterification Reagent*—Add 500 mL of *n*-butyl alcohol, 500 mL of benzene, and 3.3 mL (6 g) of sulfuric acid (H₂SO₄) to a 2-L round-bottom

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 Rosins, Including Shellac.

Current edition approved July 27, 1973. Published October 1973. Originally published as D 1469 - 57. Last previous edition D 1469 - 70. Discontinued under 3-year tentative rule 1969. Reinstated and adopted as standard in 1970.

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 06.01.

⁵ Linder, A., and Persson, V., "Determination of Rosin Acids in Mixtures with Fatty Acids," *Journal, Am. Oil Chemists' Soc.*, Vol XXXIV, No. 1, 1957, pp. 24-27.

⁶ Pipets obtainable from the Arthur H. Thomas Co., W. Washington Square, Philadelphia, PA 19105 Catalog No. 8212, or from the Scientific Glass Apparatus Co., Catalog No. JP-6000, have 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."

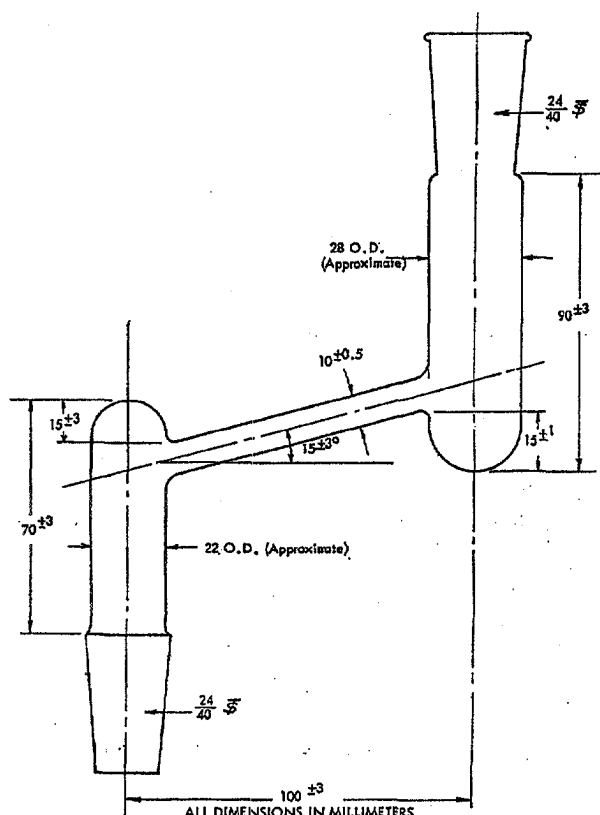


FIG. 1 Moisture Collection Trap

flask with ground joint, connect to a moisture trap and condenser, and reflux on a hot plate for 30 min to distill out the water and to form butyl-sulfuric acid. Cool and store in a glass-stoppered bottle.

5.5 *Ethyl Alcohol*, conforming to formula No. 30 or No. 3A of the U.S. Bureau of Internal Revenue.

5.6 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

5.7 *Potassium Hydroxide-Ethylene Glycol Solution (132 g/L)*—Dissolve 132 g of potassium hydroxide (KOH) pellets in 1 L of ethylene glycol in a 2-L Erlenmeyer flask. Insert a thermometer and boil to eliminate water until the temperature of the liquid reaches 190 to 195°C. Cool and store in a rubber-stoppered bottle.

5.8 *Potassium Hydroxide, Alcoholic Standard Solution (13.3 g/L)*—Dissolve 13.3 g of KOH pellets in 1 L of alcohol. Standardize against potassium acid phthalate primary standard.

5.9 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H₂SO₄).

5.10 *Thymol Blue Indicator Solution (10 g/L)*—Mix 1 g of thymol blue indicator with 100 mL of ethyl alcohol.

6. Procedure

6.1 Transfer to a 500-mL Erlenmeyer flask an amount of

TABLE 1 Precision

	Between Runs	Between Days	Between Labs
Degrees of freedom	17	11	4
Standard deviation	0.054	0.062	0.166
95 % confidence range	0.12	0.11	0.46

sample, weighed to the nearest 0.001 g, containing approximately 10 ± 1 g of nonvolatile material (Note 2). Add 150 mL of the potassium hydroxide-ethylene glycol solution and swirl to disperse the sample. Add a boiling stone, attach a condenser, and reflux on a hot plate for 2 h.

NOTE 2—Determine the nonvolatile content in accordance with either the foil method given in Test Methods D 1259 or the varnish method given in Test Method D 1644.

6.2 After refluxing, remove the flask from the hot plate and cool to room temperature under tap water. Add 100 mL of water, and while cooling under tap water, add 40 mL of HCl. Place on the hot plate again, reflux for 5 min, and cool under tap water.

6.3 Transfer the sample quantitatively to a 1-L separatory funnel with the aid of a total of 150 mL of water, followed by two 25-mL rinses with benzene. Shake, allow the layers to separate, and draw off the lower aqueous layer into a second 1-L separatory funnel. Extract the aqueous layer with a second 50-mL portion of benzene, and drain the aqueous layer into a third 1-L separatory funnel. Extract with a third 50-mL portion of benzene. Draw off and discard the water layer. Combine the benzene extracts, and wash with three 50-mL portions of water. Measure the pH of the third water wash. If it is less than 3.8, repeat with a fourth water wash. Discard the wash waters.

6.4 Transfer the washed benzene extract to a weighed (to the nearest 0.001 g) 250-mL Erlenmeyer flask, with the aid of 25 mL of benzene. Evaporate the benzene on the steam bath, preferably with the aid of a gentle stream of inert gas to a volume of approximately 20 mL. Add 5 mL of anhydrous ethyl alcohol and evaporate to dryness, to remove any water present by azeotropic distillation.

6.5 Cool and weigh to the nearest 0.001 g. To avoid oxidation, do not dry in an oven; also, the retention of a small amount of benzene does not affect the final calculation.

6.6 Using an automatic pipet, accurately measure 50 mL of the esterification reagent into the flask. Connect the flask to the moisture collection trap and condenser, place on a hot plate, heat to boiling, and reflux for 20 min. At the end of the heating period, allow the flask to cool somewhat, then remove and cool to room temperature.

6.7 Add 10 drops of thymol blue indicator solution and titrate with the alcoholic KOH solution to a blue end point.

6.8 Make a blank titration on 50 mL of the esterification solution, after refluxing it in the same manner.

7. Calculation

7.1 In order to apply properly the esterification correction factors to the isolated rosin acids-fatty acids mixture, it is necessary first to calculate the percent of rosin acids in the dried benzene extract, Y , and then to convert this value to the nonvolatile sample basis as follows (Note 3):

$$Y = [(A - B)N \times 30.24 \times 1.018]/S - 0.3$$

where:

- = alcoholic KOH solution required for titration of the sample, mL,
- = alcoholic KOH solution required for titration of blank, mL,
- = normality of the KOH solution used,
- = dried benzene extract, g,
- 0.24 = molecular weight of abietic acid multiplied by 100 and divided by 1000,
- 1.018 = experimentally determined factor to correct for the slight esterification of rosin acids, and
- 0.3 = experimentally determined factor to correct for unesterified fatty acids.

$$V = (S \times Y)/(W \times T)$$

where:

- = rosin acids in alkyd vehicle, nonvolatile basis, %,
- = original alkyd vehicle taken for analysis, g,
- = nonvolatile content of the vehicle, (Note 2) expressed as a decimal, and
- = dried benzene extract, g.

NOTE 3—If it is desired to express the total rosin acids as “commercial rosin,” use 35.0 instead of 30.2 as the factor in the calculation.

7.2 Report the results to one decimal place.

8. Precision

8.1 On the basis of an interlaboratory study of this test method in which one operator in four laboratories analyzed three samples containing different quantities of rosin, the within-laboratory standard deviation was found to be 0.05 % absolute at 11 degrees of freedom and the between-laboratory standard deviation was found to be 0.17 % absolute at 4 degrees of freedom. 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, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.2 % rosin content.

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 0.7 % rosin content.

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Standard Test Method for Oil Absorption of Pigments by Gardner-Coleman Method¹

This standard is issued under the fixed designation D 1483; the number immediately following the designation indicates the year of original adoption 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.

¹NOTE—Editorial changes were made throughout in March 1989.

1. Scope

1.1 This test method covers the determination of oil absorption of pigments by the Gardner-Coleman procedure.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 234 Specification for Raw Linseed Oil³

D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out⁴

3. Summary of Test Method

3.1 A soft paste is formed by the dropwise addition of linseed oil to the gently stirred pigment. The amount of oil required to form the paste is used to calculate an oil absorption value.

4. Significance and Use

4.1 The oil absorption value obtained by this test method provides information about the vehicle demand of the pigment when it is used in a pigment paste. Oil absorption values can be used to characterize batches of a given pigment.

4.2 This test method differs from Test Method D 281 in that D 281 requires a vigorous rubbing action whereas this test method involves only a gentle stirring and folding of the pigment. Because the end points are different, the values obtained from the two test methods generally differ.

5. Apparatus and Materials

5.1 *Balance*, capable of weighing to 0.01 g.

5.2 *Glass Container*, round-bottom, having a capacity of

½ pt (250 mL), or a low-form 250-mL beaker.

5.3 *Buret*, graduated in 0.1-mL divisions.

5.4 *Spatula*, sharp-edged steel, having a blade ½ or ¾ by 4 in. (15 or 20 by 100 mm).

5.5 *Linseed Oil*, Raw, conforming to Specification D 234 except that it shall have an acid number of 3 ± 1 .

6. Procedure

6.1 The weight of pigment used should correspond to a pigment volume of 3.0 ± 0.6 mL. Determine the weight needed by multiplying 3.0 ± 0.6 mL by the specific gravity of the pigment being tested. For example, a 20 g specimen normally is used for zinc oxide ($3.6 \text{ mL} \times 5.6 \text{ g/mL} = 20.2 \text{ g}$). Transfer the amount, weighed to 0.1 g, to the glass container.

6.2 Add oil from the buret at the rate of about 1 drop per second, stirring and "folding" the pigment continuously with the spatula during the addition. Try to provide dry pigment for the oil to strike. Do not rub or grind; as the particles of pigment become wetted, they collect in small lumps that gradually coalesce. As the coalescence proceeds, reduce the rate of oil addition. The end point is reached when the lumps, with a rolling action from the spatula, collect into a single ball or the excess of oil smears the wall of the container.

7. Calculation

7.1 Calculate the oil absorption, A , as follows:

$$A = \frac{M \times 0.93}{P} \times 100$$

where:

M = oil, mL, and

P = pigment, g.

0.93 represents density of oil (in grams per millilitre).

Express as grams of oil per 100 g of pigment.

8. Report

8.1 Report the oil absorption of the pigment as pounds (grams) of oil per 100 pounds (grams) of pigment.

9. Precision and Bias

9.1 On the basis of an interlaboratory study of this test method in which one operator in each of 5 laboratories tested 5 pigments with a broad range of oil absorption levels, the within-laboratory coefficient of variation was 3.4 % at 25 df and the between-laboratory coefficient of variation was 5.3 % at 20 df. Based on these coefficients, the following

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 Material.

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² See Gardner, H. A. and Coleman, R. E., "Oil Absorption of Pigments," *Scientific Section Circular 85*, Paint Manufacturers' Assoc. of the United States, February 1920.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.02.

Criteria should be used to judge the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 9.9 % relative.

9.1.2 *Reproducibility*—Two results obtained by different

operators in different laboratories should be considered suspect if they differ by more than 15.3 % relative.

10. Keywords

10.1 Gardner-Coleman Method; oil absorption; pigments (general properties)

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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.

[†] 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).

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.

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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Standard Test Method for Color of Transparent Liquids (Gardner Color Scale)¹

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.

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	0.4780	57	4
11	0.4842	0.4818	45	4
12	0.5077	0.4638	36	5
13	0.5392	0.4458	30	6
14	0.5846	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.

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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	
		0.13		13	13	-W
		0.16	A3	16	16	
		0.20		20	20	-Y
0.75	0.22	0.25	A2	25	25	-Z
		0.32	A1	32	32	-Z1
0.81	0.34	0.40		40	40	-Z2
		0.50	A	50	50	-Z3
0.90	0.50	0.63	B	63	63	-Z4
1.00	0.68	0.80	C	80	80	
1.15	0.92	1.00	D	100	100	-Z5
1.30	1.15	1.25	E	125	125	
		1.45	F	160	160	-Z6
1.55	1.45	1.80	G	200	200	
1.85	1.80	2.00	H	250	250	
2.20	2.15	2.60	J	320	320	
2.65 ^d	2.65 ^d	3.20	K	400	400	-Z7
		3.20	L	500	500	
3.20	3.20	4.00	M	630	630	-Z8
		4.00	N	800	800	-Z9
4.00	4.00	5.00	O	1000	1000	-Z10
		5.00	P			
5.00	5.00	6.30	Q			
		6.30	R			
6.30	6.30	8.00	S			
8.00	8.00	10.0	T			
		10.0	U			
			V			

^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 Test Methods for Glycerol, Ethylene Glycol, and Pentaerythritol in Alkyd Resins¹

This standard is issued under the fixed designation D 1615; the number immediately following the designation indicates the year of original adoption 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 methods cover the determination of glycerol, ethylene glycol, and pentaerythritol in alkyd resins and resin solutions. Other polyhydric alcohols that can be oxidized by periodic acid to formaldehyde or formic acid, or both, will interfere with the determination of glycerol and ethylene glycol. Urea, melamine, or phenolic resins interfere and render this procedure inapplicable.

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 563 Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions²
- D 1193 Specification for Reagent Water³
- D 1398 Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions²
- D 2456 Test Method for Identification of Polyhydric Alcohols in Alkyd Resins²
- D 2998 Test Method for Polyhydric Alcohols in Alkyd Resins²

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.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.

3.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.²

¹ These methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials.

Current edition approved Sept. 19, 1960. Originally issued 1958. Replaces D 1615 - 58 T.

² Annual Book of ASTM Standards, Vol 06.02.

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

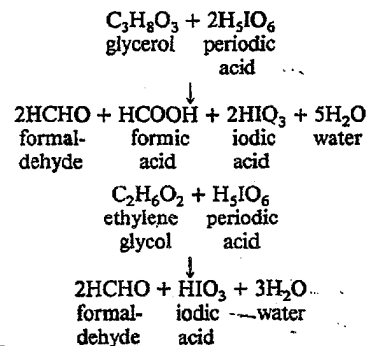
⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., and the "United States Pharmacopeia."

NOTE 1—The reagents and samples used in these methods may, under some conditions, be hazardous. Refer to the manufacturer's Material Safety Data Sheets for specific handling and safety precautions. Safe laboratory handling procedures and all applicable OSHA regulations are to be followed.

GLYCEROL AND ETHYLENE GLYCOL

4. Summary of Method

4.1 The primary hydroxyl groups of ethylene glycol and glycerol are oxidized to formaldehyde by periodic acid; the secondary hydroxyl group of glycerol is oxidized to formic acid. By acidimetric and iodometric titration, the proportions of formic acid and formaldehyde can be determined respectively, and calculated to glycerol and ethylene glycol by algebraic equations. The equations for the oxidation of the polyhydric alcohols are as follows:



With pentaerythritol, there is no reaction.

5. Apparatus

- 5.1 *Burets*, 50 and 100-mL capacity.
- 5.2 *Pipets*, 20 and 50-mL capacity.
- 5.3 *Beakers*, two of 400-mL capacity.
- 5.4 *Erlenmeyer Flasks*, four of 1000-mL capacity, glass-stoppered.
- 5.5 *Volumetric Flasks*, two of 100-mL capacity.
- 5.6 *Graduated Cylinder*, 10-mL capacity.
- 5.7 *Filter Paper*, fast, qualitative grade.
- 5.8 *Watch Glass*.

6. Reagents

- 6.1 *Methyl Purple Indicator Solution*.⁵

⁵ Methyl purple indicator manufactured by the Fleisher Chemical Co Benjamin Franklin Station, Washington 4, D.C., U.S. Patent No. 2,416,619, has been found satisfactory for this purpose.

Periodic Acid (11 g/L)—Dissolve 11 g of periodic acid in water and dilute to 1 L. Prepare fresh daily and store in a brown bottle.

Potassium Iodide Solution (200 g/L)—Dissolve 200 g potassium iodide (KI) in water and dilute to 1 L.

Sodium Hydroxide, Standard Solution (0.1 N)—Prepare and standardize a 0.1 N aqueous sodium hydroxide solution.

Sodium Thiosulfate, Standard Solution (0.2 N)—Prepare and standardize a 0.2 N aqueous sodium thiosulfate solution.

Starch Indicator Solution—Dissolve 5 g of soluble starch in water and dilute to 1 L. Preserve the solution with 1 mL of salicylic acid.

Sulfuric Acid (1+5)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) with 5 volumes of water.

Procedure

7.1 Determine the phthalic anhydride in accordance with Method D 563. Following this, extract the fatty acids in accordance with 5.6 through 7 of Method D-1398, except do not discard the aqueous phase.

7.2 Test the remaining water solution containing the polyalcohols for glycerol and ethylene glycol in the following manner:

7.2.1 Transfer the solution to a 400-mL beaker and evaporate to about 60-mL volume, using an electric hot plate as source of heat. Keep the beaker covered with a watch glass⁶ during boiling.

7.2.2 Cool to room temperature, and filter through a rapid paper into a 100-mL volumetric flask. (Take the sample for pentaerythritol determination from this same volumetric flask.) Fill to the mark and agitate.

7.2.3 Pipet 20 mL ($\frac{1}{5}$ aliquot) (Note 2) into a 1-L Erlenmeyer, glass-stoppered flask. Add 2 drops of methyl purple indicator solution and neutralize with NaOH. Pipet into the 1-L flask also 50 mL of HIO_4 solution, stopper, and swirl to mix thoroughly.

NOTE 2—The aliquot should be so chosen, if possible, that 15 to 20 % of the periodic acid is consumed during the oxidation. Considerable excess of periodic acid is required to complete the oxidation, and in case more than 20 % is consumed the results should be disregarded and a smaller aliquot taken. On the other hand, too small an aliquot is not advisable, for in such a situation the difference between titration and blank is small and any titration errors are magnified.

7.3 Simultaneously prepare two blanks containing 20 mL of water. Allow to stand 50 to 70 min at room temperature.

7.4 To the aliquot of the sample (7.2.3) and the blank, add 100 mL of water and 3 drops of methyl purple indicator and titrate with 0.1 N NaOH solution to neutrality. Use the 50-mL buret and record the volume to the nearest 0.01 mL.

7.5 To the solution that has just been titrated, add 150 mL of water, 30 mL of KI solution, and 25 mL of H_2SO_4 (1+5). Titrate with 0.2 N $Na_2S_2O_3$ solution to faint iodine color, add 10 mL of starch indicator and titrate to the disappearance of the blue color.

NOTE 3—If the end point is not stable, as indicated by return of the blue color in the stoppered flask in 5 min, add water and titrate to a stable end point. Use the 100-mL buret and record the volume to the nearest estimated 0.05 mL.

8. Calculations

8.1 Calculate the percentage of glycerol, G , as follows:

$$G = [(A - B)N \times 0.09206] / WF \times 100$$

where:

A = millilitres of NaOH solution required for titration of the sample,

B = millilitres of NaOH solution required for titration of the blank,

N = normality of the NaOH solution,

0.09206 = grams of glycerol equivalent to 1 mL of 1 N NaOH solution (acidimetric),

W = grams of sample used, and

F = aliquot fraction = $\frac{1}{5}$.

8.2 Calculate the percentage of glycerol and ethylene glycol, T , as a percentage of glycerol as follows:

$$T = [(B' - A')N \times 0.023015] / WF \times 100$$

where:

A' = millilitres of $Na_2S_2O_3$ solution required for titration of the sample,

B' = millilitres of $Na_2S_2O_3$ solution required for titration of the blank,

0.023015 = grams of glycerol equivalent to 1 mL of 1 N $Na_2S_2O_3$ solution (iodometric),

N = normality of the $Na_2S_2O_3$ solution,

W = grams of sample used, and

F = aliquot fraction = $\frac{1}{5}$.

8.3 If E is the percentage of ethylene glycol, then

$$E = 1.348(T - G)$$

When $E = 0$, the sample is free of ethylene glycol.

NOTE 4—In practice, samples containing no ethylene glycol have given calculated E values of up to 1 %. Therefore, samples with E values of 1 % or less should be considered as containing no ethylene glycol, and samples with higher E values may be corrected by 1 % at the discretion of the analyst.

9. Repeatability

9.1 The average difference between duplicate runs performed by the same analyst in the same laboratory should approximate the following values:

Percentage of glycerol	0.1
Percentage of glycol	0.4

Not more than once in twenty times would the difference be expected to exceed:

Percentage of glycerol	0.3
Percentage of glycol	1.0

10. Reproducibility

10.1 The average difference between results obtained by different analysts in different laboratories, when reported as the average of duplicate determinations, should approximate:

Percentage of glycerol	0.2
Percentage of glycol	0.5

10.2 Not more than once in twenty times should the difference be expected to exceed:

⁶ The Speedyvap watch glass has been found satisfactory for this purpose.

Percentage of glycerol
Percentage of glycol

0.6
1.2

PENTAERYTHRITOL

11. Summary of Method

11.1 Monopentaerythritol reacts with benzaldehyde to form a dibenzal which is crystalline and can be handled gravimetrically.

12. Apparatus

- 12.1 *Beakers*, 100-mL capacity.
- 12.2 *Delivery Pipets (for aliquots)*, 10 to 50-mL capacity.
- 12.3 *Steam Bath*.
- 12.4 *Ice Bath*.
- 12.5 *Crucible*, medium porosity, fritted glass.
- 12.6 *Suction Flask*.
- 12.7 *Water Aspirator*.
- 12.8 *Desiccator*.
- 12.9 *Drying Oven*, capable of maintaining a temperature of 105°C.
- 12.10 *Glass Rod*, flat-end.

13. Reagents and Materials

13.1 *Benzaldehyde-Methanol Reagent*—To 20 mL of benzaldehyde,⁷ add 100 mL of technical grade anhydrous methanol (CH₃OH).

13.2 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

13.3 *Methanol-Water Wash Solution* (1+1)—Mix equal volumes of technical anhydrous CH₃OH and water. (Because of heat of solution, prepare this solution in advance.) One fourth of the wash solution is to be stored in a refrigerator and used cold; the remainder is kept at room temperature.

14. Procedure

14.1 Transfer an aliquot of the 100-mL of sample solution (7.2.2) containing 0.15 to 0.55 g of pentaerythritol to a 100-mL beaker. Evaporate on a steam bath to a volume of approximately 5 mL. To the hot solution (incipient boiling) add 15 mL of benzaldehyde-methanol reagent and 12 mL of HCl. Swirl and allow to stand at room temperature 15 min, swirling occasionally during this time to prevent the precipitate from adhering to the bottom of the beaker. Place the beaker in an ice bath, maintaining the temperature at 0 to 2°C for 1 h or more.

⁷ Merck, N. F. grade benzaldehyde has been found satisfactory for this purpose.

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14.2 Remove the reaction mixture from the ice bath and immediately filter with suction through a weighed fritted-glass crucible of medium porosity. Rinse the beaker with 25 mL of the cold (0 to 2°C) methanol wash solution and add to the crucible. Continue to transfer and wash the precipitate with 100 mL of methanol-water wash solution at 20 to 25°C as follows:

14.2.1 Disconnect the vacuum line, pour a 10-mL portion of the wash solution from the beaker to the crucible, and stir the precipitate to form a homogeneous slurry, using a short flat-end glass rod. Connect the vacuum line and draw the wash solution through the crucible. Repeat this washing operation six times. With the last 30 mL of methanol-water wash solution, rinse the interior walls of the crucible and rinse and remove the short stirring rod.

14.3 Aspirate thoroughly and dry the precipitate at 105°C for 2 h. Cool in a desiccator and weigh.

15. Calculations

15.1 Calculate the percentage of monopentaerythritol, P , of the resin from the precipitate obtained in accordance with 14.3 as follows:

$$P = [(W_2 + 0.0269) \times 43.6] / W_1 F$$

where:

W_1 = grams of sample,

W_2 = grams of precipitate, and

F = aliquot fraction.

15.2 To convert the percentage of monopentaerythritol, P , to the percentage of commercial technical monopentaerythritol, use the following arbitrary formula which is based on the assumption that the commercial grade contains 85 % monopentaerythritol:

$$\text{Commercial monopentaerythritol, \%} = P/0.85$$

16. Repeatability

16.1 The average difference in percentage of monopentaerythritol found, between duplicate runs performed by the same analyst in the same laboratory, should approximate 0.25. Not more than once in twenty times would the difference be expected to exceed 0.6.

17. Reproducibility

17.1 The average difference in percentage of monopentaerythritol found, between results obtained by different analysts in different laboratories, when reported as the average of duplicate determinations should approximate 0.5. Not more than once in twenty times would the difference be expected to exceed 1.4.

Standard Specification for Basic Lead Silicochromate Pigment¹

This standard is issued under the fixed designation D 1648; the number immediately following the designation indicates the year of original adoption 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 covers two types of pigment commercially known as basic lead silicochromate.

Referenced Documents

2.1 ASTM Standards:

- D 1208 Test Methods for Common Properties of Certain Pigments²
- D 1366 Practice for Reporting Particle Size Characteristics of Pigments²
- D 1844 Test Methods for Chemical Analysis of Basic Lead Silicochromate²
- E 20 Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 μm by Optical Microscopy³

Composition and Properties

3.1 Both types of pigment shall consist of silica coated with lead silicates and lead chromates and shall conform to the requirements in Table 1.

3.2 The mass color and character of the tint obtained by mixing the pigment with a white pigment shall be the same as that of a reference sample mutually agreed upon by the purchaser and the seller.

3.3 The oil absorption shall be equal, within agreed upon tolerances, to that of a reference sample agreed upon by the purchaser and the seller.

3.4 Particle Size:

3.4.1 *Type 1*—This grade is characterized by major amounts of particles in the 6.5- to 28- μm range. Coarse particles retained on a 45- μm (No. 325) sieve shall be less than 0.3 %. The maximum specific surface diameter (SSD) shall be 8.5 μm .

3.4.2 *Type 2*—This grade is characterized by major amounts of fine-sized particles. In general, the maximum size is essentially below 1.0 to 2.5 μm . Coarse particles

retained on a 45- μm (No. 325) sieve shall be less than 0.1 %. The maximum Specific Surface Diameter (SSD) shall be 2.0 μm .

3.4.3 Where closer control within a grade is required, the fineness requirements shall be as agreed upon by the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of a production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon by the purchaser and the seller.

5.2 *Chemical Analysis*—Test Methods D 1844.

5.3 *Ignition Loss*—Determine the loss on ignition in accordance with the section on Procedure of Test Methods D 1208, but using an ignition temperature of 450 to 550°C.

5.4 *Particle Size by Microscopic Methods*—Procedures for determining particle size by microscopic methods are described in Practice E 20.

5.5 *Specific Surface Diameter*—Practice D 1366.

TABLE 1 Requirements for Pigments

	Weight %			
	Type 1 Regular Particle Size		Type 2 Fine Particle Size	
	Min	Max	Min	Max
Lead oxide (PbO)	46.0	49.0	42.5	46.0
Chromium trioxide (CrO ₃)	5.1	5.7	6.3	7.2
Silica (SiO ₂)	45.5	48.5	47.5	50.5
Moisture and other volatile matter, %	...	0.2	...	0.2
Ignition loss at 450 to 550°C, %	...	0.2	...	0.2

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 D01.31 on Pigment Specifications.

² Current edition approved April 25, 1986. Published June 1986. Originally published as D 1648 - 59 T. Last previous edition D 1648 - 81.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 14.02.

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Standard Specification for Strontium Chromate Pigment¹

This standard is issued under the fixed designation D 1649; the number immediately following the designation indicates the year of original adoption 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—The title of the specification and paragraph 4.1 were editorially changed in May 1987.

1. Scope

1.1 This specification covers the pigment commercially known as strontium chromate which is suitable for use in the manufacture of protective or decorative coatings.

2. Referenced Documents

2.1 ASTM Standards:

- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1208 Test Methods for Common Properties of Certain Pigments²
- D 1845 Test Methods for Chemical Analysis of Strontium Chromate Pigment²

3. Composition and Properties

3.1 *Dry Pigment*—The pigment shall be commercially pure strontium chromate free of extenders and organic material, except for materials introduced to improve those properties for which the pigment is used. The pigment shall conform to the following requirements:

	percent
Strontium as SrO, min	41

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 D01.31 on Pigment Specifications.

Current edition approved Oct. 29, 1982. Published January 1983. Originally published as D 1649 - 59 T. Last previous edition D 1649 - 77.

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

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	percent
Chromium as CrO ₃ , min	41
Chloride as Cl, max	0.1
Sulfate as SO ₃ , max	0.2
Water content, max	0.2
Coarse particles (total residue retained on No. 325 (45-μm) sieve), max	1.0

3.2 The mass color and character of the tint and tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller when tested in accordance with Test Method D 387.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Chemical Analysis*—Test Methods D 1845.

5.1.2 *Water Content*—Test Methods D 1208.



Standard Test Methods of Sampling and Testing Shellac Varnish¹

This standard is issued under the fixed designation D 1650; the number immediately following the designation indicates the year of original adoption 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 procedures for the sampling and testing of orange shellac and bleached lac varnishes.

1.2 The sampling procedures and methods of test appear in the following order:

	Sections
Sampling	5
General Requirements for Test Methods:	
Purity of Reagents	6
Preparation of Sample	7
Color	8 to 11
Drying Time	12 to 14
Nonvolatile Matter	15 to 17
Acid Value	18 and 19
Insoluble Matter	20 to 23
Wax	24 to 27
Alkali	28 and 29
Purity	30 to 34
Acid Value	35 to 37
Saponification Value	38 to 40

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 29 Test Methods of Sampling and Testing Lac Resins²
- D 1193 Specification for Reagent Water³

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *acid value*—the number of milligrams of potassium hydroxide required to neutralize 1 g of the nonvolatile portion of the lac varnish.

3.1.2 *lot*—For purpose of sampling, a lot shall consist of the entire amount of varnish manufactured as one batch and offered for delivery at one time.

3.1.3 *saponification value*—a measure of the alkali reactive groups in lac resins, it is expressed as the number of milligrams of potassium hydroxide that react with 1 g of the nonvolatile portion of the lac varnish.

4. Significance and Use

4.1 These test methods compile the common measure-

ments and procedures for characterizing shellac.

4.2 All shellac varnishes should be tested by the procedures contained in these test methods.

SAMPLING

5. Procedure

5.1 A single container shall be taken at random from each lot as representative of the whole.

5.2 In case the varnish is packaged in cans or drums of 5 gal (18.9 L) or more capacity, thoroughly mix the contents of the container and take a number of small samples from the top, bottom, and intermediate points by means of sampling tube. Mix these small samples to form a composite sample and transfer not less than 1 qt (0.94 L) to a clean, dry, glass bottle, securely stopper with a new clean cork or well-fitting cover or cap, seal, distinctly label, and transmit to the laboratory for test.

5.3 When the varnish is packaged in containers of 1 gal (3.8 L) or less capacity, send the original unopened container to the laboratory for test. When this cannot be done, thoroughly mix the contents of the container and transfer not less than 1 qt to a clean, dry, glass bottle, securely stopper with a new clean cork or well-fitting cover or cap, seal, distinctly label, and transmit to the laboratory for test.

5.4 Take precautions to ensure that the sampling apparatus and the samples themselves are neither contaminated with nor altered by any material not representative of the lot being sampled.

Test Methods

GENERAL REQUIREMENTS

6. Purity of Reagents

6.1 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.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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type IV.

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.

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² Annual Book of ASTM Standards, Vol 06.02.

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

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."

7. Preparation of Sample

7.1 Thoroughly mix the sample of varnish received for test by shaking the container immediately before the individual portions are removed for the various tests. Keep the unused portion in a tightly closed container (preferably glass) and in a cool, dark place.

COLOR

8. Apparatus

8.1 *Glass Tubes*, as prescribed in 19.1.1 of Test Methods D 29.

8.2 *Glass Plate*, as prescribed in 19.1.2 of Test Methods D 29.

9. Solvent

9.1 *Ethyl Alcohol, Denatured*, as prescribed in 8.2 of Test Methods D 29.

10. Color Comparison Standard

10.1 A shellac varnish mutually agreed upon by the purchaser and the seller for the color comparison. The varnish shall be stored in a tightly closed glass bottle and kept in a cool dark place.

11. Procedure

11.1 Weigh, to the nearest 0.1 g, approximately 35 g of the sample (Section 7) and the color comparison standard into separate stoppered flasks. Adjust both varnishes to the same nonvolatile matter content (preferably 33.3 %) by adding the proper amounts of the alcohol to the respective flasks. Thoroughly mix the contents of each flask by shaking and then allow the flasks to stand undisturbed for ½ h. Compare the color of the sample with that of the comparison standard in accordance with the procedure described in 19.4.2 or 19.4.3 of Test Methods D 29, as agreed upon by the purchaser and the seller.

DRYING TIME

12. Apparatus

12.1 *Film Applicator*—The applicator shall be made of smoothly finished metal, preferably corrosion-resistant, and accurately constructed. It shall spread a film of uniform width and shall have such clearance that the thickness of the resulting wet film of coating applied will be 1.5 mils (38 µm).

12.2 *Glass Plate*, as prescribed in 19.1 of Test Methods D 29.

13. Solvent

13.1 *Ethyl Alcohol, Denatured*, as prescribed in 8.2 of Test Methods D 29.

14. Procedure

14.1 Varnishes having more than 36 % nonvolatile matter shall be adjusted with the alcohol to a nonvolatile content of 35 ± 1 %. Varnishes having nonvolatile contents of less than 36 % shall be tested as received.

14.2 Place the glass plate in a horizontal position and the film applicator, having a clearance to give a wet-film thickness of 1.5 mils (38 µm) unless otherwise specified, at one end of the plate. Deposit a small quantity of the sample

(Section 7) on the glass plate immediately in front of the applicator. Spread the varnish the length of the panel by means of the applicator. Allow the test panel to remain in a horizontal position and dry in a well-ventilated room or chamber free from drafts and dust and in diffused light (not in direct sunlight). The temperature of the air within the room or chamber shall be between 21 and 32°C. If the time of drying is not within the specified limits of the product specification, repeat the test in a room or chamber maintained at a temperature of $223 \pm 2^\circ\text{C}$ and 50 ± 5 % relative humidity.

14.3 Test the film for set-to-touch and dry-hard times at points not less than 10 mm (½ in.) from its edges in accordance with 14.4 and 14.5.

14.4 *Set-to-Touch Time*—Lightly touch the test film with the tip of the finger and immediately place the finger tip against a piece of clean clear glass. Observe if any of the varnish is transferred to the glass. For the purpose of this test, the pressure of the finger shall not be greater than that required to transfer a spot of the varnish from ⅛ to ⅜ in. (3.2 to 4.8 mm) in cross section. The film shall be considered set-to-touch when it still shows a tacky condition but none of it adheres to the finger.

14.5 *Dry-Hard Time*—With one 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 shall be considered dry-hard when any mark left by the thumb is completely removed by the polishing operation.

NONVOLATILE MATTER

15. Materials

15.1 *Ethyl Alcohol, Denatured*, as prescribed in 8.2 of Test Methods D 29.

15.2 *Sand*—Sand that has been digested in hot-concentrated hydrochloric acid (HCl, sp gr 1.19) for 1 h, washed with water to remove all acid and soluble impurities, ignited, cooled in a desiccator, and stored in a clean-tightly closed container.

16. Procedure

16.1 Place a portion of the sample (Section 7) in a stoppered flask or weighing pipet and weigh to the nearest 0.1 mg. Transfer between 1 and 1.5 g of the sample to a weighed flatbottom glass or metal dish (petri dish or friction-top can plug) 75 to 80 mm in diameter, containing approximately 10 g of the sand, and a small glass rod. Weigh the container again and by difference obtain the exact weight of sample transferred to the dish.

16.2 Add 1 to 2 ml of the alcohol to the dish and thoroughly mix its contents with the rod. Heat the dish and its contents in a well-ventilated convection type oven maintained at $105 \pm 2^\circ\text{C}$ for 1 h. Transfer the dish to a desiccator to cool and weigh.

17. Calculation

17.1 Calculate the percentage of nonvolatile matter in the sample as follows:

$$\text{Nonvolatile matter, \%} = [(x - y)/S] \times 100$$

where:

= weight of dish plus sand, plus rod, plus nonvolatile

matter in sample used, g.

= weight of dish plus sand, plus rod, g. and

= weight of sample used, g.

IODINE VALUE

18. Reagents

18.1 See Section 10 of Test Methods D 29.

19. Procedure

19.1 Flow some of the sample (Section 7) over a 6 by 10 (152 by 254 mm) clean glass plate. Place the plate in a nearly vertical position and allow the film of varnish to dry in a well-ventilated room at 21 to 32°C for 1 h. Transfer the test plate to an oven maintained at $43 \pm 2^\circ\text{C}$ and allow to dry overnight. Cool to room temperature and scrape the dry lac resin from the plate with a razor blade, being careful not to use the thick edges or other areas of abnormal thickness.

19.2 Weigh, to the nearest 0.1 mg, 0.2 ± 0.005 g of the dry lac resin into a 250-ml, glass-stoppered bottle. Determine and calculate the iodine value in accordance with the procedures described in Section 10 of Test Methods D 29.

INSOLUBLE MATTER

20. Solvents

20.1 See 8.2 or 9.2 of Test Methods D 29, whichever is applicable.

21. Apparatus

21.1 See 8.1 or 9.1 of Test Methods D 29, whichever is applicable.

22. Procedure

22.1 Weigh, to the nearest 1 mg, an amount of the sample (Section 7) that will contain approximately 5 g of nonvolatile matter from a weighed stoppered flask or weighing pipet into a tall-form 200-ml beaker. Determine the insoluble matter in accordance with the procedure given in either 8.4 or 9.4 of Test Methods D 29, whichever is applicable.

23. Calculation

23.1 Calculate the percent of insoluble matter based on the nonvolatile matter content of the varnish as follows:

$$\text{Insoluble matter, \%} = [R/(S \times M)] \times 100$$

where:

R = grams of insoluble matter obtained,

S = grams of sample used, and

M = nonvolatile matter content of the sample, expressed as a decimal fraction.

WAX

24. Apparatus

24.1 See 16.1 of Test Methods D 29.

25. Reagents

25.1 See 16.2 of Test Methods D 29.

26. Procedure

26.1 Weigh, to the nearest 1 mg, an amount of the sample (Section 7) that will contain approximately 10 g of nonvolatile matter from a weighed stoppered flask or weighing pipet into a tall-form 200-ml beaker. Determine the wax content in accordance with the procedure given in 16.3 of Test Methods D 29.

27. Calculation

27.1 Calculate the percent of wax based on the nonvolatile matter content of the varnish, as follows:

$$\text{Wax, \%} = [R_1/(S_1 \times M)] \times 100$$

where:

R_1 = grams of wax obtained,

S_1 = grams of varnish used, and

M = nonvolatile matter content of the sample, expressed as a decimal fraction.

ASH

28. Procedure

28.1 Weigh, the nearest 1 mg, an amount of the sample (Section 6) that will contain approximately 5 g of nonvolatile matter from a weighed stoppered flask or weighing pipet into an ignited and weighed silica or porcelain dish or crucible. Determine the ash content in accordance with the procedure described in 18.1 of Test Methods D 29.

29. Calculation

29.1 Calculate the percent ash based on the nonvolatile matter content of the varnish as follows:

$$\text{Ash, \%} = [R_2/(S_2 \times M)] \times 100$$

where:

R_2 = grams of ash obtained,

S_2 = grams of varnish used, and

M = nonvolatile matter content of the sample, expressed as a decimal fraction.

PURITY

Qualitative Test for Rosin

30. Reagents

30.1 See 11.1 of Test Methods D 29.

31. Procedure

31.1 Weigh an amount of the sample (Section 7) that will contain 2 ± 0.1 g of nonvolatile matter into a 2-L Florence flask. Examine the test specimen for the presence of rosin in accordance with the procedure given in 11.2 of Test Methods D 29.

Qualitative Test for Copal

32. Reagents

32.1 See 12.1 of Test Methods D 29.

33. Procedure

33.1 To an appropriate amount (40 to 50 g) of the sample (Section 7) add a sufficient amount of the denatured alcohol to reduce the nonvolatile matter content of the test specimen

to approximately 33 %. Examine the reduced varnish for the presence of copal in accordance with the procedure described in 12.2 of Test Methods D 29.

Estimation of Adulteration

34. Procedure

34.1 When the qualitative test shows the presence of either rosin or copal in the sample, estimate their respective contents as prescribed in Section 13 of Test Methods D 29, using the iodine value obtained on the nonvolatile portion of the varnish.

ACID VALUE

35. Reagents

35.1 See 21.2 of Test Methods D 29.

36. Procedure

36.1 Weigh, to the nearest 1 mg, an amount of the sample (Section 7) that will contain approximately 1 g of nonvolatile matter from a weighed stoppered flask or weighing pipet into a 250-ml Erlenmeyer flask. Add 100 ml of the neutral alcohol and mix thoroughly. Titrate the lac solution with 0.1 *N* KOH or NaOH solution as prescribed in either 2.1.3.2 or 2.1.3.3 of Test Methods D 29, whichever is applicable.

37. Calculation

37.1 Calculate the acid value of the nonvolatile portion of the varnish as follows:

$$\text{Acid value} = (VN \times 56.1)/SM$$

where:

V = millilitres of KOH or NaOH solution required for titration,

N = normality of the KOH or NaOH solution,
S = grams of sample used, and
M = nonvolatile matter content of the sample, expressed as a decimal fraction.

SAPONIFICATION VALUE

38. Reagents

38.1 See 23.2 of Test Methods D 29.

39. Procedure

39.1 Weigh, to the nearest 1 mg, an amount of the sample (Section 7) that will contain approximately 1 g of nonvolatile matter from a weighed stoppered flask or weighing pipet into an alkali-resistant, standard-taper, ground-glass joint, 250-mL Erlenmeyer flask. Proceed in accordance with 23.3 of Test Methods D 29, also running a blank sample as prescribed therein. Titrate the sample and blank solutions in accordance with the procedure given in either 23.3.2 or 23.3.3 of Test Methods D 29, as applicable.

40. Calculation

40.1 Calculate the saponification value as follows:

$$\text{Saponification value} = [(B - V_1)N \times 56.1]/SM$$

where:

*V*₁ = millilitres of HCl required for titration of sample,

B = millilitres of HCl required for titration of blank,

N = normality of the HCl,

S = grams of sample used, and

M = nonvolatile matter content of the sample, expressed as a decimal fraction.

41. Keywords

41.1 iodine value; saponification value; shellac varnish

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 Epoxy Content of Epoxy Resins¹

This standard is issued under the fixed designation D 1652; the number immediately following the designation indicates the year of original adoption 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 7403 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 These test methods cover the quantitative determination of the epoxy content of epoxy resins.

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. For specific hazard statements, see Section 6.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 200 Practice for Preparation, Standardization, and Storage of Standard Solutions for Chemical Analysis³

3. Summary of Test Method

3.1 The resin is dissolved in a suitable solvent and the resulting solution is titrated with hydrogen bromide either directly or in situ. The hydrogen bromide reacts stoichiometrically with epoxy groups to form bromohydrins; therefore, the quantity of acid consumed is a measure of the epoxy content.

3.1.1 In Test Method A, the titration is direct with a standard solution of hydrogen bromide in glacial acetic acid.

3.1.2 In Test Method B, the titration is with standard perchloric acid in the presence of an excess of tetraethylammonium bromide. Hydrogen bromide generated in situ by the addition of perchloric acid to the quaternary ammonium halide rapidly opens the oxirane ring.

4. Significance and Use

4.1 The epoxy content of epoxy resins is an important variable in determining their reactivity and the properties of coatings made from them. These test methods may be used to determine the epoxy content of manufactured epoxy resins and confirm the stated epoxy content of purchased epoxy resins.

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.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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

6. Hazards

6.1 Hydrogen bromide and glacial acetic acid are corrosive. Chlorobenzene and chloroform are considered hazardous. In addition to other precautions, take care to avoid inhalation and skin or eye contact with these chemicals. Use goggles or a face shield, or both. Protect skin by use of suitable protective clothing. All specimen preparations shall be done in a well ventilated area, such as a fume hood.

TEST METHOD A

7. Apparatus

7.1 *Buret*, closed-reservoir type. The buret tip should be fitted with a rubber stopper of proper size to fit the neck of the Erlenmeyer flask and the stopper should have an additional small hole to permit escape of replaced air during titration.

7.2 *Magnetic Stirrer*, adjustable speed.

7.3 *Magnetic Stirring Bars*, polytetrafluoroethylene (PTFE) coated

8. Reagents and Materials

8.1 *Chlorobenzene* (Warning—See Section 6).

8.2 *Chloroform-Chlorobenzene Mixture* (1+1) (Warning—See Section 6).

8.3 *Crystal Violet Indicator Solution*—Prepare a 0.1 % solution of crystal violet in glacial acetic acid.

8.4 *Glacial Acetic Acid* (Warning—See Section 6).

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.

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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 "United States Pharmacopeia."

8.5 *Hydrogen Bromide (HBr)*, anhydrous (Warning—See Section 6).

8.6 *Potassium Acid Phthalate* ($\text{KHC}_8\text{H}_4\text{O}_4$)—Primary Standard grade.

8.7 *Hydrogen Bromide in Acetic Acid, Standard Solution* (0.1 N) (Warning—See Section 6)—Prepare by bubbling anhydrous HBr at a slow rate through glacial acetic acid until the desired normality is attained (approximately 8 g of HBr/L). Standardize each day used against 0.4 g of potassium acid phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) accurately weighed and dissolved by gently heating in 10 mL of glacial acetic acid.

NOTE—Reagent of 0.1 N concentration has been specified. As solutions exceed this concentration they became progressively less stable.

9. Procedure

9.1 Use a quantity of specimen that contains 0.001 to 0.002-g equivalents of epoxy groups. Weigh the appropriate amount, to within 1 mg, into an Erlenmeyer flask. Use a 50-mL flask for low-molecular-weight resins (liquid grades) and a 125-mL flask for high-molecular-weight resins (solid grades).

9.2 Dissolve the specimen in a solvent at room temperature. Use 10 mL of chlorobenzene for liquid grade resins or 25 mL of a 1+1 mixture of chloroform and chlorobenzene for solid grade resins. Place a magnetic stirring bar into the flask and mix on the magnetic stirrer to dissolve.

9.3 Add 4 to 6 drops of crystal violet indicator solution and attach the flask to the rubber stopper on the buret tip. Lower the buret tip to a point just above the solution and titrate with the hydrogen bromide in acetic acid solution to a blue-green end point with the magnetic stirrer rotating at a moderate speed to avoid splashing. Slow down the titration near the end point to allow ample time for the reaction to take place. Titrate, as nearly as possible, to the same color at the end point as that obtained during standardization of the reagent.

9.4 Make a blank determination on the reagents in an identical manner.

10. Calculation

10.1 Calculate the normality, N , of the HBr acetic acid as follows:

$$N = (W \times 1000) / (204.2 \times V) \quad (1)$$

where:

W = $\text{KHC}_8\text{H}_4\text{O}_4$ used, g, and

V = HBr solution used, mL.

10.2 Calculate the epoxy content, E , in gram equivalents of epoxy groups per 100 g of resin as follows:

$$E = N(V - B) / 10 \times W \quad (2)$$

where:

N = normality of the HBr in acetic acid,

V = HBr solution used for titration of the specimen, mL,

B = HBr solution used for titration of the blank, mL, and

W = specimen used, g.

10.3 Calculate the percent of oxirane oxygen, O , as follows:

$$O = 1.6N(V - B) / W \quad (3)$$

10.4 Calculate the weight per epoxy equivalent, WPE ,

that is, grams of resin containing 1 g equivalent of epoxy groups, as follows:

$$WPE = 1000W / N(V - B) \quad (4)$$

11. Precision

11.1 *Repeatability*—The difference between two results obtained by the same analyst will approximate 0.7 % of the epoxy content of the resin tested. Two such values should be considered suspect if they differ by more than 2 % absolute.

11.2 *Reproducibility*—The difference between two results, each the mean of two determinations, obtained by analysts in different laboratories, will approximate 2 % of the epoxy content of the resin tested. Two such values should be considered suspect if they differ by more than 6 % absolute.

TEST METHOD B

12. Apparatus

12.1 *Buret*, closed-reservoir type, bottom filling, 25 mL with $\frac{1}{10}$ -mL division, or potentiometric automatic titrator.

12.2 *Erlenmeyer Flasks*, 100-mL, 250-mL, and 500-mL.

12.3 *Magnetic Stirrer*, adjustable speed.

12.4 *Magnetic Stirring Bars*, polytetrafluoroethylene (PTFE) coated.

12.5 *Pipets*:

12.5.1 *Measuring Pipet*, 25-mL.

12.5.2 *Volumetric Pipet*, 50-mL.

12.6 *Volumetric Flask*, 1 L.

12.7 *Bottle*, 2 oz wide-mouth, or 100-mL disposable beaker, or equivalent.

13. Reagents

13.1 *Glacial Acetic Acid* (Warning—See Section 6).

13.2 *Tetraethylammonium Bromide*, anhydrous crystals.

13.3 *Perchloric Acid* (HClO_4), 60 % (Warning—See Section 6).

13.4 *Acetic Anhydride* (Warning—See Section 6).

13.5 *Methylene Chloride* (Warning—See Section 6).

13.6 *Crystal Violet Indicator*, crystals.

13.7 *Potassium Acid Phthalate* ($\text{KHC}_8\text{H}_4\text{O}_4$) primary standard grade.

13.8 *Diglycidyl Ether of Bisphenol-A*.

14. Reagent Preparation

14.1 *Perchloric Acid (0.1 N Solution in Glacial Acetic Acid)* (Warning—See Section 6)—Prepare in the following manner and sequence in order to avoid an excessive rise in temperature.

14.1.1 Place approximately 250 mL of glacial acetic acid into a 1 L volumetric flask. Add 13 mL of 60 % perchloric acid and mix. Add 50 mL of acetic anhydride, dilute to the mark with glacial acetic acid, and mix thoroughly.

14.1.2 Allow to stand at least 8 h for completion of reaction between the acetic anhydride and water. A shorter time period may be used if completion of the reaction is analytically verified.

14.2 *Tetraethylammonium Bromide Solution in Glacial Acetic Acid* (Warning—See Section 6):

14.2.1 Dissolve, with agitation at room temperature, 100 g of tetraethylammonium bromide in 400 mL of glacial acetic acid.

14.3 *Crystal Violet Indicator Solution*—Prepare 0.1 % solution of crystal violet indicator in glacial acetic acid. Warnings—See Section 6).

15. Standardization of 0.1 N Perchloric Acid Reagent

15.1 Procedure A—Standardization with Potassium Acid Phthalate:

15.1.1 Dissolve, by gentle heating, 0.4 g of potassium acid phthalate, weighed accurately to the nearest milligram, in 50 mL of glacial acetic acid, and add 6 to 8 drops of crystal violet indicator solution. Insert a clean stirring bar into the sample, and adjust the magnetic stirrer to effect solution. Continue agitation throughout the titration procedure. Titrate with perchloric acid reagent solution to the end point which is a sharp change in color from blue to green, stabilize for at least 2 min.

15.1.2 Calculate and record the perchloric acid reagent normality as follows:

$$N = (W \times 100)/(204.2 \times V) \quad (5)$$

where:

N = normality of perchloric acid reagent,

W = potassium acid phthalate used, g, and

V = volume of perchloric acid reagent required to titrate the standard, mL.

15.2 Procedure B—Standardization with Diglycidyl Ether of Bisphenol-A (DGEBA):

15.2.1 Dissolve 0.25 to 0.40 g of the DGEBA accurately weighed to the nearest milligram into a 2 oz wide-mouth bottle or 100 mL disposable beaker. Add 10 to 15 mL of methylene chloride. Insert a clean stirring bar into the sample and adjust the magnetic stirrer to effect solution. Continue agitation throughout the titration procedure.

15.2.2 Add 10 mL of tetraethylammonium bromide reagent and 6 to 8 drops of crystal violet indicator solution and titrate to a sharp blue-to-green end point with the perchloric acid reagent solution. The end point should be stable for at least 30 s.

15.2.3 Calculate and record the perchloric acid reagent factor, F , as follows:

$$F = (W_d \times E)/V \quad (6)$$

where:

W_d = diglycidyl ether of bisphenol-A standard used, g,

E = epoxide of the standard used (normally 25.19), weight %

The perchloric acid should be standardized initially by Procedure A and thereafter at least twice per week by Procedure A or B. Temperature of the reagent solution must be kept at $25 \pm 2^\circ\text{C}$ ($77 \pm 3^\circ\text{F}$).

16. Procedure

16.1 Weigh the required amount of specimen into a 2-oz disposable glass bottle or plastic beaker. The amount of

specimen weight used is dependent on the expected epoxide equivalent weight (EEW) as follows:

EEW	Approximate Specimen Size, g
170-375	0.4
375-600	0.6
600-1000	0.8
1000-1500	1.3
1500-2000	1.8
2000-2500	2.3
2500-5000	2.8

16.2 Add 10 to 15 mL of methylene chloride to the specimen. Insert a clean stirring bar and adjust the magnetic stirrer to effect solution. Continue agitation through the titration procedure.

16.3 Add 10 mL of tetraethylammonium bromide reagent and 6 to 8 drops of crystal violet indicator solution. Titrate with 0.1 N perchloric acid reagent to a sharp blue to green end point which is stable for at least 30 s. Record the volume of perchloric acid reagent used to titrate the specimen.

17. Calculation

17.1 If 15.1 is used for standardization, calculate weight percent epoxide, E , as follows:

$$E = 4.3 \times V \times N/W \quad (7)$$

17.2 If 15.2 is used for standardization, calculate weight percent epoxide, E , as follows:

$$E = F \times V/W_e \quad (8)$$

where:

W_e = weight of epoxy resin specimen used, g.

17.3 Calculate the epoxy equivalent weight, W_{EEW} , as follows:

$$W_{EEW} = 43 \times 100/E \quad (9)$$

where 43 = mol weight of the epoxy ring.

17.4 Calculate weight percent of oxirane oxygen, O , as follows:

$$O = 16/43 \times E = 1.6 \times V \times N/W \quad (10)$$

18. Precision

18.1 A liquid epoxy resin sample with approximately 24.1 % epoxide was tested by seven laboratories where ten analysts obtained the following results:

18.1.1 *Repeatability*—The difference between two results obtained by the same analyst should not vary by more than 1.22 % relative.

18.1.2 *Reproducibility*—The difference between two results, each the mean of two determinations obtained by analysts in different laboratories should not vary by more than 2.97 % relative.

19. Keywords

19.1 epoxide equivalent weight (EEW); liquid epoxy resin; oxirane; weight percent epoxide; weight per epoxy equivalent (WPE)

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Standard Terminology of Cellulose and Cellulose Derivatives¹

This standard is issued under the fixed designation D 1695; the number immediately following the designation indicates the year of original adoption 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—The title was changed editorially in October 1989.

This terminology is divided into three classes as follows:

- (A) Cellulosic Materials and Constituents,
- (B) Chemical Modifications and Derivatives of Cellulose,
- and
- (C) Properties of Cellulose and Associated Concepts that are applicable to both (A) and (B).

(A) CELLULOSIC MATERIALS AND CONSTITUENTS

acetylation pulps—pulps used in the manufacture of cellulose acetate or other esters, and subject to various specifications by the manufacturers, including those of purity, moisture content, sheet properties, and viscosity.

alpha-cellulose—(1) an imprecise and historic term (originally devised around 1900) that has been used to characterize cellulose purity. The currently preferred term is "R₁₀." (2) When used, alpha-cellulose is the portion of a cellulose pulp that remains insoluble after treatment with aqueous sodium hydroxide at 20°C. The term has meaning only if there is a precise statement of the initial sodium hydroxide concentration (usually about 18 %), the subsequent dilution, and other conditions employed in its determination.

alpha pulps—in a rather indefinite way, all those bleached wood pulps that have an alpha-cellulose content above 88 %. (Where possible, it is desirable to use a more specific term.)

araban—a pentosan yielding essentially only arabinose on hydrolysis.

arabinogalactan—a polysaccharide consisting of arabinose and galactose units, like the water-soluble polysaccharide of larch.

arabinose—a pentose that occurs as one of the sugar units in some hemicelluloses.

arabinomethylglucuronoxylan—a hemicellulose containing arabinose, 4-O-methylglucuronic acid, and xylose groups in its structure.

beta-cellulose—(1) an imprecise and historic term (originally devised around 1900) that has been used to characterize cellulose purity. The currently preferred term is "S₁₀-S₁₈." (2) When used, beta-cellulose is the portion of a cellulose pulp which is dissolved in the alkaline solution of the

alpha-cellulose test and is subsequently reprecipitated on neutralization of the alkaline solution. All conditions must be specified exactly.

carbohydrates not cellulose—the noncellulosic carbohydrates of a cellulosic material.

cellophane pulps—pulps used in the manufacture of cellophane, and subject to various specifications by the manufacturers, including those of purity, moisture content, sheet properties, and viscosity.

cellulose—(1) the main solid constituent of woody plants; it occurs widely elsewhere in the vegetable kingdom, and to a small extent in the animal kingdom. (2) chemically, cellulose is β -1-4 glucan of high degree of polymerization. It is desirable to apply "cellulose" to this material only and to designate the predominantly cellulosic residue obtained by subjecting woody tissues to various pulping processes as "cellulosic residues," "cellulosic pulps," or the like.

cellulose I—the crystalline modification of cellulose that normally occurs in nature.

cellulose II—the crystalline modification of cellulose that is found in mercerized cellulose, in regenerated cellulose, and in cellulose produced by the hydrolysis of various cellulose derivatives.

cellulose III—a crystalline modification of cellulose produced by treatment, under certain conditions, with ammonia or sometimes by amines. The method of removing the reagent determines the modification produced.

cellulose IV—a crystalline modification of cellulose produced by heat treatment of cellulose II.

cellulose X—a crystalline modification of cellulose produced by treatment of cellulose with strong hydrochloric acid or phosphoric acid.

cellulose, purified cotton—see cotton cellulose, purified.

cellulose, purified wood—see wood cellulose, purified.

chemical cellulose—a chemically purified cellulosic material that is intended for chemical treatment to produce derivatives.

chemical cotton—chemical cellulose prepared from cotton; generally, but not necessarily, cotton linters.

chemical pulps—in the paper industry, pulps produced by chemical processes, as contrasted to those produced by mechanical processes. (see also chemical wood pulp).

chemical wood pulp—a term used in the paper industry for pulps obtained by digestion of wood with solutions of various chemicals.

DISCUSSION—This term, which refers to pulp produced and purified by chemical processes, should not be confused with chemical cellulose which refers to pulp that is to be used in chemical processes.

¹ This terminology is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.36 on Cellulose.

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cotton cellulose, purified—chemical cellulose from cotton fiber or linters. (see also **chemical cotton**)

cotton linters—see **linters**.

dissolving pulps—see **pulps, dissolving**.

extractives—compounds occurring in plant materials, but not forming part of the structural elements, that are removed with neutral solvents such as ether, alcohol, and water.

galactan—a polysaccharide composed essentially of galactose units. (see also **arabinogalactan**)

galactoglucomannan—one of the hemicelluloses of softwoods, containing three types of sugar units—galactose, glucose, and mannose.

galactomannan—a polysaccharide containing galactose and mannose units. Galactomannans usually have a long chain of mannose units with galactose side chains and are found in seed gums (guar, locust bean).

gamma-cellulose—(1) an imprecise and historic term (originally devised around 1900) that has been used to characterize cellulose purity. The currently preferred term is "S₁₈." (2) When used, gamma-cellulose is the portion of a cellulosic material that remains dissolved after neutralization of the alkaline solution from the alpha-cellulose determination; all conditions must be specified exactly.

glucan—a macromolecular substance that can be hydrolyzed to give almost exclusively glucose.

glucomannan—a hemicellulose consisting essentially of glucose and mannose.

glucuronoxylan—a common designation for the xylose-containing hardwood hemicelluloses. (see also **methylglucuronoxylan**)

hemicellulose—any of a number of cell-wall polysaccharides that are removable by extraction with aqueous alkali and that may be hydrolyzed by boiling with dilute acids to give constituent monosaccharide units; any of the noncellulosic cell-wall polysaccharides.

hexosan—frequently used in contradistinction to pentosan, for a polysaccharide consisting mainly of hexose units.

holocellulose—the total polysaccharide fraction of extractive-free wood. The method of isolation or of determination should always be given.

kraft pulp—pulp cooked by the alkaline liquor consisting essentially of a mixture of caustic soda and sodium sulfide. The make-up chemical is traditionally sodium sulfate, which is reduced to the sulfide in the chemical recovery process; hence the alternative designation, sulfate pulp.

lignin—that part of plant material which is not saccharified by the action of 72 % sulfuric acid or 42 % hydrochloric acid, after the resins, waxes, and tannins have been removed.

linters—the short fibrous material adhering to cotton seed after the ginning operation. After removal from the seed it is used to a limited extent as a fibrous raw material for special papers. The principal use, however, is for chemical cellulose, that is, as the raw material for the manufacture of cellulose derivatives.

mannan—strictly, a polysaccharide composed entirely of mannose units, but used conventionally to distinguish the hexosan wood hemicelluloses from the pentosans (xylan). (see also **galactoglucomannan** and **glucomannan**)

mannogalactan—see **galactomannan**.

methylglucuronoxylan—the main hemicellulose of hardwood pulps; a polysaccharide containing xylose and 4-O-methylglucuronic acid groups. In the wood it is partially acetylated.

nitrating pulps—pulps used for the manufacture of cellulose nitrate and subject to various specifications by the manufacturers, including those of alpha-cellulose content and viscosity.

oligosaccharides—polymeric carbohydrates containing relatively few (compared to the polysaccharides) sugar units connected by glycosidal linkages. Two to nine units has been suggested as a suitable range. For longer chains the polymers cannot be readily separated into individual molecular species.

pectic substrates—complex polysaccharides containing a large proportion of galacturonic acid units.

pectins—colorless, amorphous, water-soluble polysaccharides occurring in plant tissues that yield pectic acid and methanol on hydrolysis.

pentosans—one of the groups of amorphous carbohydrates included under the general term "hemicellulose." Pentosans yield principally pentoses on acid hydrolysis. The principal pentosan in wood is xylan.

polyuronides—polysaccharides containing uronic acid groups.

pulps, dissolving—chemical cellulose from wood pulp.

pulps for chemical conversion—chemical cellulose from wood pulp.

pulps for manufacture of cellulose derivatives—chemical cellulose from wood pulp.

purified cotton cellulose—see **cotton cellulose, purified**.

purified wood cellulose—see **wood cellulose, purified**.

R₁₀—the portion of a cellulose pulp that is insoluble in 10 % sodium hydroxide using Test Method D 1696² or its equivalent.

DISCUSSION—R₁₀ is considered to be the main commercial long chain length portion of cellulose, similar to the intent in the imprecise term "alpha-cellulose" (see S₁₀).

rayon pulps—pulps used in the manufacture of rayon, and subject to various specifications by the manufacturers, including those of purity, moisture content, sheet properties, and viscosity.

S₁₀—the portion of a cellulose pulp that is soluble in 10 % sodium hydroxide using Test Method D 1696, or its equivalent.

DISCUSSION—S₁₀ is considered to contain both hemicellulose and degraded, short chain length cellulose (see R₁₀).

S₁₈—the portion of a cellulose pulp that is soluble in 18 % sodium hydroxide using Test Method D 1696, or its equivalent.

DISCUSSION—S₁₈ is considered to be mainly hemicellulose, similar to the intent of the imprecise term "gamma-cellulose."

S₁₀-S₁₈—an estimate of the portion of degraded cellulose in a pulp obtained by subtracting S₁₈ from S₁₀.

DISCUSSION—See S₁₀ and S₁₈ definitions. S₁₀-S₁₈ is similar to the intent in the imprecise term "beta-cellulose."

sulfate pulp—see **kraft pulp**.

² Annual Book of ASTM Standards, Vol 15.04.

sulfite pulp—wood pulp produced by cooking with a sulfite liquor made by dissolving sulfur dioxide in an aqueous base.

wood cellulose, purified—chemical cellulose from wood.

xylan—a pentosan giving almost exclusively xylose on hydrolysis.

(B) CHEMICAL MODIFICATIONS AND DERIVATIVES OF CELLULOSE

carboxymethylcellulose, CMC—the common name for a cellulose ether of glycolic acid. It is usually marketed as a water-soluble sodium salt, more properly called sodium carboxymethylcellulose. In the early literature, it is sometimes called cellulose glycolate or cellulose glycolic acid.

cellulose acetate—in the broad sense, any of several esters of cellulose and acetic acid. (see also **cellulose triacetate**)

cellulose acetate butyrate—a mixed ester of cellulose containing both acetate and butyrate groups.

cellulose acetate phthalate—a mixed ester of cellulose containing both acetate and phthalate groups.

cellulose acetate propionate—a mixed ester of cellulose containing both acetate and propionate groups.

cellulose derivative—a substance derived from cellulose by substitution of one or more of the hydroxyl groups with some other radical. Most derivatives are ethers or esters.

cellulose esters—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced acyl groups.

cellulose ethers—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced by alkyl groups.

cellulose lacquer—a liquid coating composition containing as the basic film-forming ingredients cellulose esters or ethers and plasticizers with or without resins.

cellulose mixed ester—a cellulose ester containing more than one type of acyl group.

cellulose nitrate—any of various nitrate esters of cellulose.

DISCUSSION—Cellulose nitrate is often and erroneously called "nitrocellulose" because it was formerly considered a nitro-compound.

cellulose plastics—plastics based on cellulose compounds, such as esters (cellulose acetate) and ethers (ethylcellulose).

cellulose propionate—any ester of cellulose with propionic acid.

cellulose sodium glycolate—see **carboxymethyl cellulose**.

cellulose triacetate—that form of cellulose acetate in which the degree of substitution approaches 3 sufficiently that the product is not soluble in acetone.

cellulose xanthates—the salts of cellulose xanthic acid. Commonly, cellulose xanthate refers to sodium cellulose xanthate, the essential constituent of the viscose solution, from which viscose rayon is spun.

dope—a composition, usually a cellulose lacquer, for application on textiles and leathers. Also a very viscous crude reaction product, as acetylation dope.

ethylcellulose—any of several ethyl ethers of cellulose. The one most generally used in industry has sufficient substitution to be soluble in organic solvents.

hydrocelluloses—water-insoluble products of the hydrolysis of cellulose with acids. They are molecularly heterogeneous in the sense that they are composed of molecules

varying in degree of polymerization. The average degree of polymerization (DP) and the DP distribution depend on the nature of the acid treatment and of the original cellulose. The term may also be applied to any insoluble polysaccharide so formed and separated as a more or less homogeneous fraction from the mixture of products, but the singular form "hydrocellulose" should not be used without an article, to avoid the implication of a molecularly homogeneous species.

(2-hydroxyethyl)cellulose—any of several cellulose ethers in which some of the hydroxyl groups have been substituted with hydroxyethyl groups. Hydroxyethyl cellulose, except at very low degrees of substitution, is water-soluble.

(2-hydroxyethyl)ethylcellulose—a mixed ether of cellulose containing both hydroxyethyl and ethyl groups.

(2-hydroxyethyl)methylcellulose—a mixed ether of cellulose containing both hydroxyethyl and methyl groups.

(2-hydroxypropyl)methylcellulose—a mixed ether of cellulose containing both hydroxypropyl and methyl groups.

ionic cellulose ethers—those water-soluble cellulose ethers which contain ionizable groups, in more than trace amounts. Sodium carboxymethyl cellulose is an example.

lacquer—see **cellulose lacquer**.

methylcellulose—any of several methyl ethers of cellulose. Commercially, the water-soluble ether (degree of substitution approximately 1.5 to 2.0).

methylethylcellulose—a cellulose ether containing both methyl and ethyl groups.

microcrystalline cellulose—a commercial name for cellulose that has been hydrolyzed to the limiting DP and that consists essentially of microcrystals.

nitrocellulose—see **cellulose nitrate**.

nonionic cellulose ethers—that class of cellulose ethers which does not contain any ionizable groups.

oxycelluloses—water-insoluble substances formed by the action of oxidizing agents on cellulose. The chemical nature of oxycelluloses varies with the oxidant used, and the type is indicated by attaching the name of the oxidant adjectivally to "oxycelluloses" as in "hypochlorite oxycelluloses." Any such mixture is "an oxycellulose" and the word should not be used in the singular without either the definite or indefinite article. In many respects, the phrases "oxidized cellulose" or "partially oxidized cellulose" are preferable.

propylene glycol ether of methylcellulose—see **(2-hydroxypropyl)methylcellulose**.

regenerated cellulose—cellulose regenerated from a solution of cellulose or from a cellulose derivative.

sodium carboxymethylcellulose—see **carboxymethylcellulose**.

viscose—a solution of sodium cellulose xanthate prepared by dissolving the reaction product formed by the interaction of carbon disulfide and alkali cellulose in an aqueous solution of sodium hydroxide. Viscose is used mainly in the manufacture of rayon or cellophane, where it is extruded through fine openings of the proper shape into a coagulating bath.

(C) PROPERTIES OF CELLULOSE AND ASSOCIATED CONCEPTS

accessibility—the fraction of total cellulose present that is accessible to certain reagents under certain specified conditions. The conditions of determination should always be indicated.

acetylation—substitution of an acetyl radical for an active hydrogen. Specifically, formation of cellulose acetate from cellulose.

acetyl groups—the characteristic groups of acetic acid; CH_3CO —.

acyl groups—radicals derived from carboxylic acids by removal of the hydroxyl group.

acid groups—functional groups having the properties of acids. In cellulose and its derivatives, these are usually carboxyl groups.

aging—in general, the change of properties with the passage of time. Specifically, the changes occurring in shredded alkali cellulose when allowed to stand exposed to air.

air-dry—a condition applied to paper or pulp whereby its moisture content is in equilibrium with the atmospheric conditions to which it is exposed. According to trade custom, pulps are generally understood to be air dry when they contain 10 % of moisture; for example, a pound of air-dry pulp contains 0.9 lb of oven-dry pulp and 0.1 lb of moisture (see *STP 60-B*).

alcohol-benzene solubility—solubility of a cellulosic pulp in a mixture of ethanol and benzene. The term is without precise meaning unless complete specification of an analytical procedure is attached explicitly or implicitly.

aldehyde groups—carbonyl groups to which a hydrogen atom is attached; the first oxidation stage of an alcohol; $-\text{CHO}$.

alkali resistance—for a cellulosic pulp, the fraction insoluble in alkali, usually sodium hydroxide, of a fixed concentration under specified conditions. The term is without precise meaning unless complete specification of an analytical procedure is attached explicitly or implicitly. (see also **alkali solubility**)

alkali solubility—for a cellulosic pulp, the fraction in alkali of a fixed concentration under specified conditions. This term is without precise meaning unless complete specification of an analytical procedure is attached explicitly or implicitly. Some related terms imply at least a partial specification, for example, "ten percent potassium hydroxide solubility" or "nondilution alkali solubility."

alkali staining—discoloration caused by the presence of an alkali.

alkyl groups—monovalent aliphatic radicals derived from aliphatic hydrocarbons by removal of a hydrogen.

amorphous regions—those regions within a cellulosic material which, on the basis of X-ray diffraction or other suitable technique, do not show any evidence of crystalline structure. The technique should be specified.

anhydroglucose units—the repeating unit of many polysaccharides, including cellulose; since the glucose molecules have combined with elimination of water, the unit is called "anhydroglucose" rather than "glucose."

ash—the inorganic residue obtained by igniting a specimen of pulp, paper, or other cellulosic material in such a way

that all combustible and volatile compounds are removed. Conditions of ashing should be specified.

bleachability—the capacity of a pulp to bleach to a given whiteness. This is approximately and indirectly related to lignin content.

bone-dry—see **oven-dry**.

brightness—as commonly used in the paper industry, the reflectivity of a sheet of pulp or paper for blue light measured under standardized conditions on a particular instrument designed and calibrated specifically for the purpose.

carbonyl group—the bivalent radical, $-\text{CO}-$, especially as it occurs in aldehydes or ketones.

carboxyl group—the radical $-\text{COOH}$ characteristic of most organic acids.

CED (cupriethylenediamine) viscosity—see **viscosity**, **cupriethylenediamine**.

chain length distribution—in a linear polymer like cellulose, the frequency distribution of molecular size, usually expressed in units of degree of polymerization.

chlorine number—the number of grams of chlorine gas or of bleaching powder (expressed as its equivalent in chlorine) that is consumed by 100 g of oven-dry pulp in a definite time under certain specified conditions. The chlorine number is an indication of the bleach requirement of the pulp and an indirect estimate of the lignin content.

color reversion—a process common to almost all bleached cellulose pulps in which the color darkens to a greater or lesser extent on standing.

commercial moisture regain—see **moisture regain**, **commercial**.

copper number—the number of grams of copper in the cuprous oxide reduced from a cupric compound by 100 g of pulp or paper (after deduction of moisture, ash, and sizing materials) treated under specified conditions with an excess of cupric solution. The copper number is an indication of the relative number of reducing groups in the pulp or paper.

cross linking—the reaction of a difunctional molecule with each of two molecules of a polymer. This change of the polymer from linearity produces profound changes in the physical properties.

crystalline regions—see **crystallinity**.

crystallinity—a regular arrangement of the atoms of a solid in space. In most polymers, including cellulose, this state is usually imperfectly achieved. The crystalline regions (ordered regions) are submicroscopic volumes in which there is more or less regularity of arrangement of the component molecules. In these regions there is sufficient geometric order to enable definite X-ray diffraction patterns to be obtained. (see also **degree of crystallinity**; **degree of order**)

crystallinity index—a number used to represent the state of crystallinity of cellulose as a whole. Unfortunately, it has been differently defined by different investigators and should not be used unless it is clearly stated which crystallinity index is meant. (see also **crystallinity**)

crystallite—a single crystalline region. (see also **crystallinity**)

cuam viscosity—see **viscosity**, **cuprammonium**.

cuene viscosity—see **viscosity**, **cupriethylenediamine**.

cuprammonium process—a process for making rayon by dissolving cellulose in an ammoniacal copper solution and

running the resulting solution into an acid bath, thereby regenerating the cellulose as fibers.

cuprammonium viscosity—see **viscosity**, cuprammonium.

diethylenediamine viscosity (*cuene* or *CED*)—see **viscosity**, cupriethylenediamine.

degradation—in general chemical use, the conversion of a complex compound to a simpler. Specifically for cellulose, the breakdown of the polymer chain, usually by hydrolysis or oxidation. Degradation is usually applied to changes in chemical structure. (see also **deterioration**)

degree of crystallinity—the fraction by mass of a cellulose sample occurring in crystalline regions. The method of determining crystallinity must be stated. (see also **crystallinity**)

degree of lateral order—the relative degree of molecular alignment. As for degree of crystallinity, quantitative values must be defined in terms of the experimental measurements. (see also **crystallinity**)

degree of polymerization, DP—in general, the average number of base units, or of monomeric units per molecule in linear polymers. Specifically, the average number of anhydroglucose units (or derivative units) per molecule of cellulose (or cellulose derivative). The type of average obtained depends upon the method used for the determination. Hence, the method must always be specified.

degree of substitution, DS—in a cellulose derivative, the average number of hydroxyl groups substituted per anhydroglucose unit. DS varies from zero to about 3.

density—the mass per unit volume at a specified temperature.

density, apparent—the mass per unit volume of a sheet of pulp or paper. It is commonly calculated by dividing the basis weight by the caliper, although it must be recognized that the numerical value thus obtained is dependent upon the definition of the ream.

density, bulk—the mass of a cellulosic material that will fill a unit volume of a container under specified conditions.

deterioration—a permanent impairment of the physical properties. (see also **degradation**)

dry weight—a term is usually applied to the mass of the oven-dry material, but it is ambiguous unless the method of drying is specified.

end groups—the functional groups at either end of the cellulose molecule. It is frequently used for the terminal glucose units that contain these groups. It is usually clear from the context which is meant, and the differentiation is seldom important. One end group is reducing (probably a hemiacetal) and one nonreducing (a free hydroxyl in the 4-position).

equilibrium moisture content—the percentage mass of moisture in a material when it has attained equilibrium with water vapor at a specified pressure or partial pressure. It is determined by successive weighings of the sample, either on the adsorption or desorption curve, and plotting moisture content against the logarithm of time. It should be designated as adsorption equilibrium or desorption equilibrium.

exchange capacity—see **ion-exchange capacity**.

fiber—the unit cell of vegetable growth, which is many times longer than its diameter and which consists largely of cellulose. It is the basic unit of pulps and papers.

fiber length—when applied to a pulp, this applies to the mean fiber length and both the experimental details and the calculations must be specified.

fiber length distribution—the frequency distribution of the individual fiber lengths in a material about the mean fiber length.

fiber saturation point—the moisture content in the absorption of water by cellulose when the cell walls are saturated, but virtually no free water is present in the grosser capillary structure. The concept is based on the hypothesis that a discontinuity occurs in the sorption curve at this point. Some careful studies of the region have failed to show a discontinuity, but it is still possible to define "fiber saturation point" as an extrapolation of the adsorption curve. In any case, it should only be used with reference to the method of determination.

fiber structure—the morphological structure of a fiber at the visual or microscopic level. (see also **fine structure**)

fibril—a fiberlike bundle of molecules, oriented in one direction either with the fiber axis or spirally around it. It makes up the fibrous structural unit of the single fiber. The cellulose fibrils are crystalline as shown by X-ray diffraction.

film—sheeting, including that composed of cellulose or its derivatives, having nominal thickness not greater than 10 mils. (see also **films**)

films—transparent sheeting. If this is used as a generic term, regardless of sheet thickness, the plural should be used and the singular restricted to a specified size. (see also **film**)

fine structure—a generic term denoting the submicroscopic structure that depends on molecular arrangement.

fluidity—the reciprocal of viscosity.

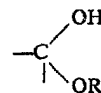
gamma number—degree of substitution (DS) $\times$ 100. This term is of widespread use in the cellulose xanthate field.

gloss—the geometrically selective reflectance of a surface responsible for its shiny or lustrous appearance. Surface reflectance is commonly at a maximum in or near the geometric directions in which a mirror would reflect light.

hardness—a term referring to the degree of cooking of a pulp. A hard pulp is one in which the residual lignin content is relatively high.

haze—the turbidity produced by the material in some cellulose derivatives that is not completely soluble in the medium. This turbidity may refer to solutions or to the solid plastic.

hemiacetal groups—functional groups derived from carbonyl groups by addition of one molecule of an alcohol, of the general structure:



hornification—the behavior of certain forms of cellulose and related materials on drying, which results in a lowered reactivity. This is apparently caused by the formation of new hydrogen bonds with a corresponding decrease in accessibility.

hydrodynamic specific surface—the specific surface of a fibrous material as measured by the filtration resistance of a compacted pad formed from a fiber suspension under specified conditions.

- hydrophilic**—having an affinity for water.
- hydrophobic**—having an antagonism to water.
- hydroxyl groups**—the monovalent group $-\text{OH}$, characteristic of hydroxides and alcohols.
- inherent viscosity**—see viscosity, inherent.
- intrinsic viscosity**—see viscosity, intrinsic.
- ion exchange**—a reversible process by which ions are interchanged between a solid and a liquid with no substantial structural changes in the solid.
- ion-exchange capacity**—for cellulosic ion-exchange materials, the number of milliequivalents of ions that can be exchanged by 1 g of the ion-exchange material.
- ion-exchange materials**—insoluble solid materials with the ability to exchange reversibly certain ions, in the structure or attached to the surface as functional groups, with ions in a surrounding medium.
- iron-sodium tartrate, EWNN or FeTNa**—a complex tartaric acid salt of sodium and ferric iron. Solutions in aqueous sodium hydroxide will dissolve cellulose. Both the German abbreviation, EWNN, and the English, FeTNa, are to be found in English articles. The details of preparation of the solution should always be given.
- iron-sodium tartrate viscosity**—the viscosity of a solution or dispersion of cellulose or pulp under standardized condition when dissolved in the iron-sodium tartrate solvent. (see also iron-sodium tartrate)
- kappa number**—the number of millilitres of 0.1 N potassium permanganate solution consumed by 1 g of oven-dry pulp under specified conditions. It is an indication of the hardness or bleachability of a pulp. An indirect estimate of lignin content.
- lateral order**—the degree of regularity of arrangement of atoms and atomic groups in the direction normal to the molecular chain axes in linear polymers. Quantitative evaluation is impossible without further specification and without description of the particular experimental technique.
- leveling-off degree of polymerization, LODP**—the nearly constant degree of polymerization of cellulose reached after very prolonged mild hydrolysis or short drastic hydrolysis.
- limiting viscosity number**—see viscosity, intrinsic.
- luster**—in the pulp and paper industry, synonymous with "gloss" or "sheen." (see also gloss)
- mercerization**—the process of subjecting a vegetable fiber to the action of a fairly concentrated aqueous solution of a strong base so as to produce great swelling with resultant changes in fine structure, dimensions, morphology, and mechanical properties.
- mesomorphous cellulose**—those portions of cellulose in which the segments of the individual molecules have some regularity of arrangement, but not enough to permit strict lattice order to give a distinct X-ray diffraction pattern.
- methoxyl group**—the monovalent group $-\text{OCH}_3$, characteristic of methyl alcohol and its esters or ethers.
- moisture content**—the moisture present in a cellulosic material, as determined by prescribed methods, conventionally expressed as a percentage of the total mass of the "wet" material.
- moisture equilibrium**—the condition reached by a sample when it no longer takes up moisture from, or gives up moisture to the surrounding air.
- moisture equilibrium for preconditioning**—the moisture equilibrium condition reached by a sample after free exposure to air controlled at the standard condition for preconditioning.
- moisture equilibrium for testing**—the equilibrium moisture condition reached by a sample after free exposure to moving air controlled at standard conditions.
- moisture regain**—the moisture present in a cellulosic material, as determined by prescribed methods, expressed as a percentage of the oven-dry mass.
- moisture regain, commercial**—an arbitrary figure formally adopted as the regain to be used in calculating the commercial or legal mass of shipments or deliveries of any specific material.
- moisture regain, standard**—the moisture regain of sample brought from a lower moisture regain into equilibrium with the standard atmosphere.
- moles of substituent combined, MS**—in a cellulose derivative, the average number of substituent molecules per glucose unit. For most derivatives, $\text{MS} = \text{DS}$, but for a few, such as hydroxyethylcellulose, the substituted group may also be reactive and MS may be greater than DS.
- nonreducing end groups**—those terminal glucose units of cellulose or its derivatives which contain a free hydroxyl in the 4-position and do not reduce Fehling's solution or similar reagents.
- oleoresins**—nonaqueous secretions of resin acids dissolved in terpenic hydrocarbons that are produced or exuded from the intercellular resin ducts of living trees, especially the conifers, and accumulated, together with oxidation products, in the wood of weathered limbs or stumps.
- order distribution**—see lateral order.
- orientation**—the angle made by the crystallites of the cellulose with the fiber axis. This is approximately the same angle as that made by the molecules or the fibrils.
- overdry**—the state of a cellulosic material that has been dried to constant mass at a temperature of 100 to 105°C.
- percent hydrolysis-resistant cellulose**—the residue after treatment of cellulose with acid under specified conditions, expressed as a percentage of the original.
- permanganate number**—the number of millilitres of 0.1 N potassium permanganate solution that is consumed by 1 g of oven-dry pulp under certain specified and carefully controlled conditions. It is an indication of the hardness or bleachability of a pulp. It also is an indirect estimate of lignin content.
- plastic**—(1) capable of being deformed continuously and permanently in any direction without rupture, under a stress exceeding the yield value. (2) made of, consisting of or pertaining to plastics. (3) a material containing as an essential ingredient an organic substance of large molecular mass, which is solid in its finished state and, at some stage in its manufacture or in its processing into finished articles, can be shaped by flow.
- porosity**—the existence in a material of connected air voids. It is frequently expressed as the ratio of void volume to total volume.

activity—the ability to react. For proper use of the term, the reaction in question and the conditions should be stated and the parameter used in measuring reactivity indicated, such as rate, uniformity, or the like.

reduced viscosity—see viscosity, reduced.

reducing end groups—those terminal glucose units in cellulose or its derivatives in which the 1-position is not substituted or involved in a glycosidic linkage. These end groups will reduce Fehling's solution or similar reagents.

regain—see moisture regain.

relative viscosity—see viscosity, relative.

reversion—in general, the tendency of pulp or paper properties to return to those of the material at some other stage. It is frequently specifically applied to the loss of brightness with time, after bleaching has produced a high brightness.

ripening—in the manufacture of viscose rayon, that stage where the cellulose xanthate solution is stored several days under controlled conditions to reach a state from which coagulation is easier.

roving—a continuous strand of loosely assembled fibers that is approximately uniform in cross-sectional area and without twist.

specific gravity—the ratio of the mass of a specimen to the mass of an equal volume of water, both at the same specified temperature. Generally, density is more useful. (see also density)

specific surface—the surface per unit mass (or less frequently per unit volume) of a moisture-free sample. The specific external surface is used as a measure of the degree of fineness of fibrillation.

specific viscosity—see viscosity, specific.

standard moisture regain—see moisture regain, standard.

surface area—the total area of the surface. As usually used for reactive pulps, this applies not to external surface, but to the internal surface as well, the total surface available to gases or penetrating liquids. It is useful to express this as specific surface, area per gram.

swelling—increase in volume and dimensions caused by penetration of a liquid.

total internal surface—the surface of a cellulosic material available for absorption of a given substance. A useful substance for this purpose is water; the value obtained with water is close to the potential maximum surface that can be developed without disruption of the crystalline structure.

unit cell—a parallelepiped element of crystal structure, containing a certain number of atoms, the repetition of which through space will build up the complete crystal.

uronic acids—oxidation products of the sugars in which a primary alcohol group has been oxidized to a carboxyl without changing the reducing functional group.

viscosity—in general, the resistance of a fluid to flow or motion within itself. As applied to cellulose or its derivatives, the viscosity of a solution of the substance.

viscosity, cuprammonium—the viscosity of a solution or dispersion of cellulose or pulp in cuprammonium hydroxide under standardized conditions.

viscosity, cupriethylenediamine—the viscosity of a solution or dispersion of cellulose or pulp under standard conditions when dissolved in a solution of copper hydroxide in ethylenediamine.

viscosity in absolute units, CGS poises—the force in dynes required to move, at a velocity of 1 cm/s, one surface having an area of 1 cm² past another parallel-like surface 1 cm away, overcoming the resistance to shear of the material filling the space between.

viscosity index—the ratio of the viscosity of a highly concentrated solution to that of a dilute solution. It is a measure of solvent power and, in derivatives, of uniformity of substitution.

viscosity, inherent—the quotient of the natural logarithm of relative viscosity by the concentration, that is, $\ln \eta_{rel}/c$. The concentration should be specified.

viscosity, intrinsic—the limiting value of reduced viscosity, η_{sp}/c , as c (concentration) approaches zero. In the cellulosic field the concentration is generally expressed as grams per decilitre. The IUPAC Committee of Nomenclature has recommended the expression "Limiting viscosity number" for this and the concentration is generally expressed as grams per millilitre.

viscosity number—see viscosity, reduced.

viscosity ratio—see viscosity, relative.

viscosity, reduced—the specific viscosity divided by the concentration. In the cellulosic field the concentration is generally expressed as grams per decilitre. The IUPAC Committee on Nomenclature has recommended the expression "viscosity number" for this, in which case the preferred expression of concentration is grams per millilitre.

viscosity, relative—the ratio of the viscosity of a solution to that of the pure solvent. The IUPAC Committee on Nomenclature has recommended the expression "Viscosity ratio" for this.

viscosity, specific—the difference between the viscosity of a solution and that of a solvent, divided by the latter.

viscosity, xanthate—for a cellulosic pulp, the viscosity of the viscose that will be obtained as a result of a specified series of processes.

viscous flow—flow, usually at low velocity, in which fluid elements flow in a straight line parallel to the direction of flow of the liquid in the absence of turbulence.

water vapor permeability—the rate of water vapor transmission per unit of thickness and per unit of vapor pressure differential. Test conditions must be specified.

whiteness—the degree of approach of the color of a substance to that of the ideal white, commonly represented by a standardized preparation of magnesium oxide.

xanthate viscosity—see viscosity, xanthate.

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 Solubility of Cellulose in Sodium Hydroxide¹

This standard is issued under the fixed designation D 1696; the number immediately following the designation indicates the year of original adoption 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 method² is intended for application to dissolving-type cellulose pulps prepared from cotton or wood. The procedure is not directly applicable to unrefined pulps for use in chemical conversion processes because solubility equilibrium may not be attained within the specified extraction 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 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 1193 Specification for Reagent Water³
- D 1347 Test Methods for Methylcellulose⁴
- D 1348 Test Methods for Moisture in Cellulose⁴

2.2 TAPPI Standard:

- T 429 Method for Alpha-Cellulose in Paper⁵

3. Summary of Test Method

3.1 Pulp is steeped in a sodium hydroxide solution of a specified concentration for 1 h at 20°C. The soluble fraction is estimated by dichromate oxidation of the filtered steeping alkali. The concentration of sodium hydroxide used in the pulp extraction process must be reported as part of the analytical result. Sodium hydroxide concentrations of 10, 18, and 21.5 % are most frequently used. Data are reported as percent of dry sample weight.

3.2 The extraction procedure avoids dilution sequences, and therefore, the results are not comparable to data obtained by the alpha, beta, gamma methods of pulp fractionation (see TAPPI Method T 429). The terms "alpha," "beta," or "gamma-" cellulose must not be applied to any test values obtained by this procedure since they are defined only by the method of their determination.

3.3 The essential feature of the method is to prepare sodium hydroxide extracts and oxidize the soluble material with dichromate as described. Alternative methods of estimating dichromate by titration with ferrous ammonium sulfate and sodium thiosulfate are described.

4. Significance and Use

4.1 The measurement of soluble oxidizable components of cellulose in sodium hydroxide is indicative of the purity of the cellulose sample, since pure cellulose is insoluble in sodium hydroxide. The extracted components are typically hemicelluloses, which are naturally present in the wood. Differences in pulp purity can have a dramatic impact on the processing and properties of the cellulose derivatives produced from the pulp.

5. Apparatus

5.1 *Constant-Temperature Bath*—A water bath maintained at $20 \pm 0.2^\circ\text{C}$.

5.2 *Stirrer*—Mechanical stirrer as shown in Fig. 1. All parts in contact with solutions must be of stainless steel. The stirrer motor shall be a variable speed laboratory motor with 1500 rpm max speed.

5.3 *Fritted-Glass Filter Crucible*—A fritted-glass filter crucible, coarse porosity (pore size 40 to 60 μm), of 30-mL capacity.

5.4 *Electrometric Titration Apparatus*—For estimation of dichromate by titration with ferrous ammonium sulfate. An indicator may be used as described in 8.6, but for rapid, accurate analysis an electrometric apparatus is recommended.

6. Purity of Reagents

6.1 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,⁶ where such specifications are available. 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

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 D01.36 on Cellulose.

Current edition approved May 25, 1990. Published July 1990. Originally published as D 1696 - 59. Last previous edition D 1696 - 61 (1985)⁴.

² This test method is an adaptation of the method designated CCA 8:55 by the Analysis Committee of the Central Committee of the Cellulose Industry of the Swedish Association of Pulp and Paper Engineers. This test method is also comparable with the TAPPI Tentative Standard T 235 m-58, Solubility of Pulp in Cold Sodium Hydroxide.

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

⁴ Annual Book of ASTM Standards, Vol 06.02.

⁵ Annual Book of ASTM Standards, Vol 15.09.

⁶ "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."

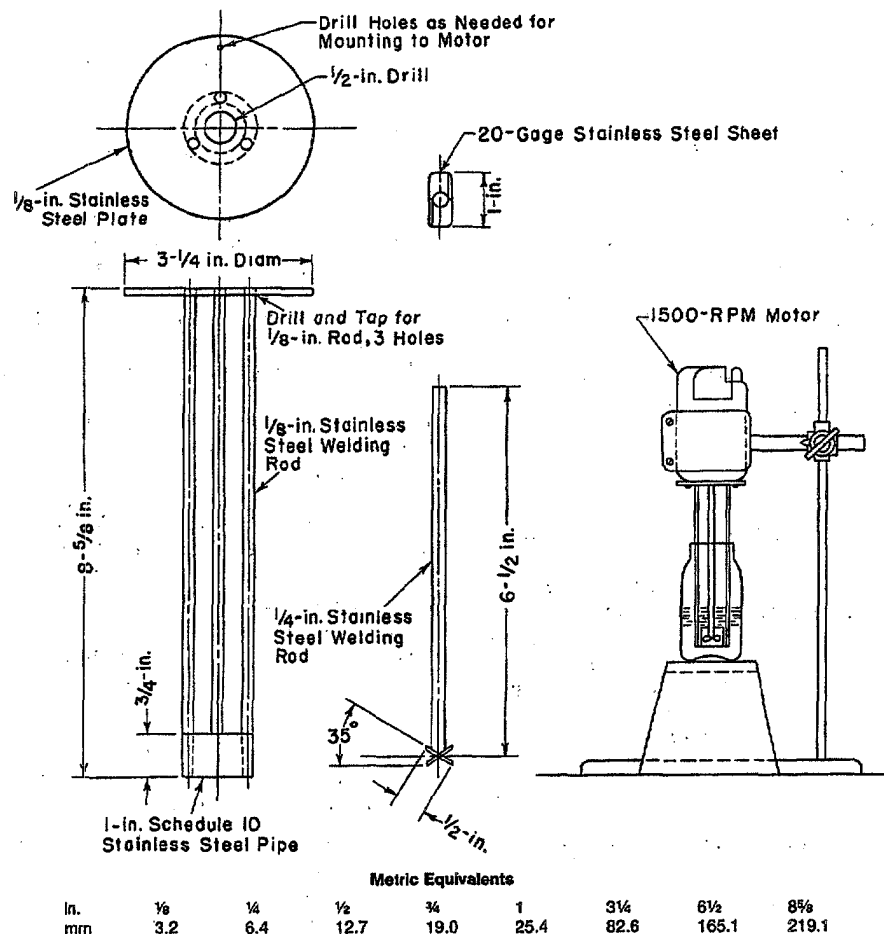


FIG. 1 Design Details of Mechanical Stirrer

7. Reagents

7.1 *Ferriin* (0.025 *M*)—Dissolve 1.48 g of orthophenanthroline monohydrate (or 1.624 g of the hydrochloride) with 0.695 g of ferrous sulfate (FeSO_4) in water and dilute to 100 mL.

7.2 *Ferrous Ammonium Sulfate Solution* (0.1 *N*)—Dissolve 40 to 41 g of ferrous ammonium sulfate ($\text{FeSO}_4(\text{NH}_4)_2 \cdot \text{SO}_4 \cdot 6\text{H}_2\text{O}$) in water containing 10 mL of H_2SO_4 and dilute to 1 litre in a volumetric flask. Standardize the solution daily against potassium permanganate (KMnO_4).

7.3 *Potassium Dichromate Solution* (20 g/L)—Weigh 20.0 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$), transfer to a 2-L beaker, and dissolve in approximately 700 mL of water. Add, with constant stirring, 150 mL of H_2SO_4 . Allow to cool to room temperature. Dilute to 1 L with water.

7.4 *Potassium Iodide* (KI).⁷

7.5 *Sodium Hydroxide Solution*—Dissolve solid sodium

hydroxide (NaOH) in an equal weight of water. Cover and allow to stand about one week to permit settling of sodium carbonate (Na_2CO_3). Prepare the dilute NaOH solutions listed below by approximate addition of freshly boiled water to portions of the concentrated stock solution. Use a siphon to withdraw the required volume of 50 % NaOH solution from the stock bottle; do not disturb the Na_2CO_3 precipitate. The Na_2CO_3 content of the dilute solutions should not exceed 1 g/L. Standardize the final dilute solutions by titration with standard acid.

7.5.1 *Sodium Hydroxide Solution* (10 %)—Prepare a solution containing 10.0 ± 0.1 g NaOH per 100 g of solution. Specific gravity at 20/4°C is 1.1089. This solution is 2.77 *N*.

7.5.2 *Sodium Hydroxide Solution* (18 %)—Prepare a solution containing 18.0 ± 0.1 g NaOH per 100 g of solution. Specific gravity at 20/4°C is 1.1972. This solution is 5.39 *N*.

7.5.3 *Sodium Hydroxide Solution* (21.5 %)—Prepare a solution containing 21.5 ± 0.1 g NaOH per 100 g of solution. Specific gravity at 20/4°C is 1.2356. This solution is 6.64 *N*.

7.6 *Sodium Thiosulfate Solution* (0.1 *N*)⁶—Prepare and standardize 0.1 *N* sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution as directed in 23.11 of Test Methods D 1347.

⁷ This reagent is required only for the iodometric method for measuring dichromate (see 8.7). Thyodene, supplied by Fisher Scientific Co., Pittsburgh, PA is an acceptable substitute.

7.7 Starch Solution (5 g/L).⁶

7.8 Sulfuric Acid (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

8. Preparation of Samples

8.1 Condition the air-dry pulp samples to obtain moisture equilibrium by exposing them to the atmosphere for 24 h in the room where the portions of this sample will be weighed for moisture and solubility analysis.

8.2 If the pulp is in sheeted form, tear it into pieces about 10 mm square. Do not use cut edges. Weigh a portion for moisture analysis and immediately also weigh, to the nearest 1 mg, about 1.6 g of the air-dry sample for the solubility determination.

8.3 Determine the moisture content of the air-dry sample in accordance with Test Methods D 1348. Calculate the oven-dry weight of the sample for the solubility determination.

9. Procedure

9.1 Precool the NaOH solutions to 20°C. Pulp solubility in 18 and 21.5 % NaOH solutions is not affected by a few degrees variation in temperature. Hence, temperatures of 20 ± 2°C are satisfactory in this case. However, pulp solubility in 10 % NaOH solution is very sensitive to temperature variation. The operator must control temperature to 20 ± 0.2°C in all phases of pulp extraction with 10 % NaOH solution.

9.2 With a pipet or buret, transfer 100 mL of the NaOH solution of the desired concentration (10, 18, or 21.5 %) into a 250-mL beaker. Add the 1.6 g air-dry pulp sample to the solution. Allow the pulp to swell for 2 min; then introduce the stirring apparatus into the beaker so that the surface of the liquid coincides with the draft tube top. Disintegrate the pulp by stirring for 3 min (Note 1). Adjust the stirring speed to prevent addition of air to the slurry (about 1500 rpm). Remove the stirrer. Carefully clean the stirring apparatus and walls of the beaker with a glass rod so that all pulp fibers are retained in the alkali. Cover the beaker with a watch glass and leave in the 20°C bath until a total of 60 min have elapsed from the time of addition of NaOH to the sample. For solubility determinations in 10 % NaOH solution, all extraction operations must be performed with the sample container in the constant-temperature water bath. In work with 18 or 21.5 % NaOH, the sample container may be removed from the bath for the disintegration process.

NOTE 1—Most pulps are completely disintegrated after 3 min. A reasonable increase in the time of disintegration does not sensibly affect the solubility. On the other hand, low values are obtained if the disintegration is not complete. The stirrer should, therefore, be run until complete disintegration is obtained, even if the time required exceeds 3 min.

9.3 At the end of the 1-h total extraction time, remove the beaker from the bath and immediately filter the slurry through a coarse fritted-glass filter. Apply suction but do not pull air through the cellulose mat on the filter. Discard the first 10 mL of filtrate. If suspended fibers are noted in the filtrate, recycle the filtrate through the cellulose mat to clarify. Retain the filtrate in a stoppered Erlenmeyer flask.

9.4 Transfer 10-mL aliquots of the NaOH filtrates to 250-mL Erlenmeyer flasks. Add 10 mL $\text{K}_2\text{Cr}_2\text{O}_7$ solution to

each flask; then carefully add 30 mL of H_2SO_4 to each flask. After 10 min (Note 2), cool to room temperature. Include a blank test with the original NaOH extraction solution in this operation.

NOTE 2—It is suggested that heat be applied to maintain a temperature in the range of 125 to 130°C for the entire 10-min period. A reflux system is highly satisfactory.

9.5 The 10-mL aliquot of the NaOH filtrates specified in 9.4 is generally suitable for dissolving-type pulps; however, if the alkali solubility is greater than 16 %, reduce the volume of filtrate to 5 mL and the volume of H_2SO_4 to 25 mL. For pulps with alkali solubility less than 5 %, use 20 mL of filtrate and 45 mL of H_2SO_4 .

9.6 After dichromate oxidation, add 50 mL of water to each sample and the blank. Cool again to room temperature. Titrate the excess $\text{K}_2\text{Cr}_2\text{O}_7$ with 0.1 N ferrous ammonium sulfate solution. An electrometric titrimeter is preferred for this operation. If this equipment is not available, ferroin indicator may be used to detect the end point.

9.7 An acceptable alternative for measuring dichromate concentration is to use iodometric methods. In this case, transfer the cooled solution with about 500 mL of water to a 1-L Erlenmeyer flask. Add about 2 g of KI and after 5 min titrate the solution to a visual end point with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution, using starch as the indicator. Make a blank test on the original NaOH extraction solution in the same manner.

10. Calculations

10.1 Calculate the results, when obtained by titration with ferrous ammonium sulfate, as follows:

$$\text{Alkali solubility, \%} = [(b - a)N \times 68.5]/mw \quad (1)$$

where:

- a = ferrous ammonium sulfate solution required for titration of the sample, mL,
- b = ferrous ammonium sulfate solution required for titration of the blank, mL,
- N = normality of the ferrous ammonium sulfate solution,
- 68.5 = cellulose equivalent to 1 milliequivalent of $\text{K}_2\text{Cr}_2\text{O}_7$, times 10 (Note 3), mg,
- m = filtrate used for oxidation, mL, and
- w = oven-dry weight of sample used, g.

NOTE 3—The factor 68.5 is the experimental value recommended in method CCA 8:55.² As an alternative, the problem of standardization of ferrous ammonium sulfate can be handled in terms of oxidation of a reference cellulose solution prepared at each occasion for analysis as follows: Dissolve 200 mg (oven-dry weight) of high-quality cotton linters pulp in H_2SO_4 (3+1) (prepared by mixing 3 volumes of H_2SO_4 (sp gr. 1.84) with 1 volume of water) and dilute to 100 mL with H_2SO_4 (3+1). Do not use heat in the preparation of this solution. Immediately after preparation, oxidize 2-mL aliquots of this solution in the procedure described in Section 8 for blank and sample treatment. Compare the cellulose solution and blank to express the concentration of ferrous ammonium sulfate solution in terms of milligrams of cellulose per millilitre of solution. The calculation then becomes:

$$\text{Alkali solubility, \%} = [10 C(b - a)]/mw \quad (2)$$

where:

- C = concentration of ferrous ammonium sulfate solution in terms of milligrams of cellulose per millilitre, and
- b , a , m , w are defined as in 10.1.

10.2 Calculate the results, when obtained by titration with $\text{Na}_2\text{S}_2\text{O}_3$ solution, as follows:

$$\text{Alkali solubility, \%} = [(V_1 - V_2)N \times 68.5]mw \quad (3)$$

where:

V_1 = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the sample, mL,

V_2 = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the blank, mL, and

N = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, and m and w are defined as in 10.1.

Report

1.1 Report the alkali solubility as a percentage of oven-pulp to one decimal place. Use the symbol S for alkali

solubility. Denote the strength of the sodium hydroxide solution by a numerical suffix, for example, designate the solubility in 10 % NaOH solution by the symbol S_{10} .

11.2 If it is of interest to report the insoluble fraction, subtract the S value from 100. Use the symbol R , with the appropriate subscript, to report this value. For example: $R_{10} = 100 - S_{10}$.

12. Precision and Bias

12.1 *Precision*—The relative standard deviation of the test method for all concentrations of sodium hydroxide is less than 4 %.

12.2 *Bias*—No statement of bias can be made as no suitable reference material exists for determining bias.

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Standard Test Method for Cellulose Chain Length Uniformity by Fractional Precipitation of Cellulose Nitrate¹

This standard is issued under the fixed designation D 1716; the number immediately following the designation indicates the year of original adoption 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.

1. Scope

1.1 This test method covers the determination of the chain length uniformity of cellulose by fractional precipitation of cellulose nitrate. It is recognized that this test method does not give absolute delineation of chain length; however, the curves obtained by this method afford a useful basis for comparing the relative chain length uniformity of different cellulose samples. It is applicable primarily to purified celluloses.

2. Referenced Documents

2.1 ASTM Standards:

- D 301 Test Methods for Soluble Cellulose Nitrate²
- D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 This test method is based on the following steps:

3.1.1 Conversion of the cellulose sample into cellulose nitrate by a nitration step that does not degrade the cellulose to any appreciable extent.

3.1.2 Separation of the cellulose nitrate into several viscosity components through stepwise precipitation by the addition of a nonsolvent to a solution of the sample.

3.1.3 Measurement of the weight and viscosity of each recovered fraction and calculation of its average chain length (degree of polymerization).

4. Purity of Reagents

4.1 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 available⁴. 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

PREPARATION OF CELLULOSE NITRATE

5. Apparatus

- 5.1 Bottles, 2-oz (60-mL), wide-mouth, with plastic caps.
- 5.2 Constant-Temperature Bath, $0.0 \pm 0.5^\circ\text{C}$ (ice water).
- 5.3 Erlenmeyer Flask, temperature-resistant glass, 200-mL, standard-taper 45/50 neck, with glass stopper.
- 5.4 Filter Funnels, Büchner-type, 50-mm diameter with fritted disk, coarse porosity.
- 5.5 Mixer.⁵
- 5.6 Tamper, stainless steel rod, 40-mm diameter by 100-mm.
- 5.7 Weighing Bottles, 40 by 100-mm, ground-glass stoppers.
- 5.8 Wire Baskets, 200-mesh stainless screen.
- 5.9 Oven, forced draft, 50°C .

6. Reagents

6.1 Ethyl Alcohol—Neutral 95 % ethyl alcohol or neutral denatured alcohol with 0.5 % benzene (Formula 2B) or 10 % methanol (Formula 30).

6.2 Methanol, absolute.

6.3 Nitrating Acid Mixture (64 % HNO_3 , 26 % H_3PO_4 , 10 % P_2O_5)—Add very slowly with a spatula 404 g of phosphorus pentoxide (P_2O_5) to 1000 g of cold-white fuming nitric acid, swirled in a 2-L Erlenmeyer flask. Keep the acid cold throughout the time of P_2O_5 addition by keeping the flask immersed in the ice-water bath.

6.4 Nitric Acid (sp gr 1.49 to 1.50)—White fuming nitric acid.

7. Procedure

7.1 Place 2.0 ± 0.1 g of the air-dry sample in the mixer and mix dry for about 30 s or until the material is uniformly fluffed. Transfer the fluffed material to a glass weighing bottle and dry for 1.5 h at $50 \pm 1^\circ\text{C}$. Remove the sample from the oven, cover, and cool in a desiccator.

7.2 Weigh a 100-g portion of the nitrating acid into a glass-stoppered weighing bottle. Stopper the bottle and place it in the 0°C constant-temperature bath. Allow time for the acid to equalize to the bath temperature.

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 D01.36 on Cellulosics.

Current edition approved Sept. 28, 1962. Published November 1962. Originally published as D 1716 - 60 T. Last previous edition D 1716 - 60 T.

² Annual Book of ASTM Standards, Vol 06.02.

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

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."

⁵ An Osterizer or Waring Blender has been found satisfactory for this purpose.

7.3 Weigh a 1-g portion (Note 1) of the dried cellulose sample to the nearest 0.001 g, and transfer it with stainless steel forceps to the bottle containing the nitrating acid (0°C). Swirl to mix. Allow the bottle and contents to remain in the bath for exactly 60 min, swirling at 10-min intervals.

NOTE 1—One gram of cellulose gives sufficient nitrate for viscosity measurement and a single fractionation. If duplicate fractionations, percentage nitrogen, or stability tests are desired, use a blend of nitrates from multiple 1-g nitrations.

7.4 At the end of the reaction period, transfer the contents of the bottle to the Büchner-type, fritted-glass funnel. Apply vacuum, pressing the cellulose nitrate down with the stainless steel tamper (Note 2). Continue pressing the cellulose nitrate with the tamper while applying suction until excess acid has just been removed. With forceps, quickly transfer the nitrate pad to 300 mL of water (0°C) and stir rapidly for several seconds. Wash the nitrate with at least five 300-mL changes of water (room temperature).

NOTE 2—The stainless steel tamper is used to provide uniform pressure and to exclude air while removing excess nitrating acids; it also tends to form a loose pad of cellulose, which in most cases may be handled quickly and conveniently with tweezers to transfer to the water used for drowning. An alternative procedure for removing the excess nitrating acid and drowning the nitrate is to apply vacuum cautiously while squeezing the cellulose nitrate until only the minimum amount of nitrating acid to cover the sample completely remains. The sample is then flooded in the funnel under vacuum with 50 % aqueous acetic acid previously chilled to -10°C.

7.5 Stabilize the cellulose nitrate by subjecting it to at least three 5-min boils in fresh 200-mL portions of water. Transfer the nitrate to the Büchner-type, fritted-glass funnel, and drain with suction. Place the nitrate in the wire basket and dry in the oven at 50°C for 1 h. Remove the nitrate from the oven, transfer to a weighing bottle, cool in a desiccator, and weigh. If the nitrate is to be stored for more than a few days, transfer it to a 2-oz (60-mL) wide-mouth bottle, saturate it with ethyl alcohol, seal the bottle with a cap lined with tin foil, and place in a cool, dark cabinet.

8. Measurements on Fractionated Nitrate

8.1 *Determination of Yield*—Determine the moisture content for the pulp as nitrated. Consider the dried cellulose nitrate for practical purposes to be moisture-free. Calculate the yield in percent by multiplying the quotient of nitrate weight divided by pulp weight by 54.6.

NOTE 3—Depending on the type of cellulose, the yield value gives some indication of the degree of nitration and the purity of the cellulose. As the specified nitration conditions give a high nitrogen content with pure cellulose, low yields should occur only with pulps of high pentosan content (xylan dinitrate) or with pulps of high lignin content.

8.2 *Determination of Percentage Nitrogen*—When desired, determine the degree of nitration (percent nitrogen) by Specification and Methods of Test D 301.

NOTE 4—Nitrates prepared by the procedure described in Section 7, from a pure cellulose, should contain 13.9 ± 0.1 % nitrogen; they should have a stability of 20 to 25 min by Specifications and Methods D 301. It should be noted that the degree of nitration does affect solution viscosity,

and for samples with nitrogen contents outside the limits specified an adjustment must be made in the calculation of intrinsic viscosity.⁶

MEASUREMENT OF VISCOSITY

9. Apparatus

9.1 *Bottles*, 4-oz, narrow-mouth, with plastic caps.

9.2 *Constant Temperature Bath*, $25.0 \pm 0.1^\circ\text{C}$.

9.3 *Shaker*.⁷

9.4 *Stop Watch*, reading to 0.1 s.

9.5 *Tin Foil*, 0.001 in. thick.

9.6 *Viscometers*, Cannon-Fenske, 0.4 to 0.5-mm bore capillary, 1-mL bulb,⁸ or Ubbelohde Type 1.

NOTE 5—It should be further specified that the viscometer have a flow time within the range of 70.0 to 100.0 s/mL at 25°C for the solvent being used in order to avoid a kinetic energy correction.

9.7 *Oven*, forced draft, 50°C .

10. Materials

10.1 *Ethyl Acetate*, reagent-grade.

11. Procedure

11.1 For a preliminary check of the viscosity, weigh a 0.0500 ± 0.0001 -g sample of nitrate (redried for 1 h at 50°C and cooled just prior to weighing), place it in a 4-oz (120-mL) plastic capped bottle, add 100 mL of ethyl acetate from a pipet, cover the bottle with a cap lined with tin foil, and place it on the shaker until solution is complete.

11.2 Measure the flow time of the solvent and the solution in the viscometer. For the final determination of viscosity, adjust the concentration of the nitrate solution to a value indicated by the following equation:

$$C \times DP = 50 \text{ (Section 12)}$$

11.3 If the approximate degree of polymerization (DP) of the sample is known, select the appropriate concentration at the start; the initial measurement will then suffice.

NOTE 6—The time needed to obtain complete solution depends on the degree of polymerization and the degree of subdivision of the sample, usually running from a few minutes for rayon or cellophane to several hours for very high viscosity wood pulps or raw cotton.

12. Calculation of Degree of Polymerization (DP)

12.1 Calculate the degree of polymerization, DP , as follows:

$$DP = K[\eta]$$

where:

$$K = 75,$$

$$[\eta] = \text{intrinsic viscosity} = (\eta_{sp}/C)[1/(1 + k'\eta_{sp})],$$

$$\eta_{rel} = \text{viscosity of solution/viscosity of solvent},$$

$$\eta_{sp} = \eta_{rel} - 1,$$

$$k' = 0.35, \text{ and,}$$

⁶ Lindsley, C. H., and Frank, M. B., "Intrinsic Viscosity of Nitrocellulose Related to Degree of Nitration," *Industrial and Engineering Chemistry*, Vol 45, 1953, p. 2491.

⁷ A Burrell Wrist Action or International Shaker has been found satisfactory for this purpose.

⁸ This viscometer can be obtained from the Cannon Instrument Co., P. O. Box 16, State College, PA 16801.

TABLE 1 Viscosity-Concentration Relationship

η_{sp}	$[\eta]C$	η_{sp}	$[\eta]C$	η_{sp}	$[\eta]C$	η_{sp}	$[\eta]C$	η_{sp}	$[\eta]C$	η_{sp}	$[\eta]C$
0.005	0.0050	0.190	0.178	0.375	0.332	0.560	0.468	0.745	0.591	0.930	0.702
0.010	0.0100	0.195	0.183	0.380	0.335	0.565	0.471	0.750	0.593	0.935	0.705
0.015	0.0149	0.200	0.187	0.386	0.339	0.570	0.475	0.755	0.597	0.940	0.707
0.020	0.0199	0.205	0.191	0.390	0.343	0.575	0.479	0.760	0.600	0.945	0.710
0.025	0.0248	0.210	0.196	0.395	0.347	0.580	0.482	0.765	0.603	0.950	0.713
0.030	0.0297	0.215	0.200	0.400	0.351	0.585	0.486	0.770	0.607	0.955	0.716
0.035	0.0346	0.220	0.204	0.405	0.355	0.590	0.489	0.775	0.609	0.960	0.719
0.040	0.0394	0.225	0.209	0.410	0.359	0.595	0.492	0.780	0.613	0.965	0.721
0.045	0.0443	0.230	0.213	0.415	0.362	0.600	0.496	0.785	0.616	0.970	0.724
0.050	0.0491	0.235	0.217	0.420	0.366	0.605	0.499	0.790	0.619	0.975	0.727
0.055	0.0540	0.240	0.221	0.425	0.370	0.610	0.503	0.795	0.622	0.980	0.730
0.060	0.0588	0.245	0.226	0.430	0.374	0.615	0.506	0.800	0.625	0.985	0.733
0.065	0.0636	0.250	0.230	0.435	0.378	0.620	0.509	0.805	0.628	0.990	0.735
0.070	0.0683	0.255	0.234	0.440	0.381	0.625	0.513	0.810	0.631	0.995	0.738
0.075	0.0731	0.260	0.238	0.445	0.385	0.630	0.516	0.815	0.634	1.000	0.741
0.080	0.0778	0.265	0.242	0.450	0.389	0.635	0.520	0.820	0.637	1.005	0.743
0.085	0.0825	0.270	0.247	0.455	0.393	0.640	0.523	0.825	0.640	1.010	0.746
0.090	0.0873	0.275	0.251	0.460	0.396	0.645	0.526	0.830	0.643	1.015	0.749
0.095	0.0919	0.280	0.255	0.465	0.400	0.650	0.529	0.835	0.646	1.020	0.752
0.100	0.0966	0.285	0.259	0.470	0.404	0.655	0.533	0.840	0.649	1.025	0.754
0.105	0.1013	0.290	0.263	0.475	0.407	0.660	0.536	0.845	0.652	1.030	0.757
0.110	0.1059	0.295	0.267	0.480	0.411	0.665	0.539	0.850	0.655	1.035	0.760
0.115	0.1103	0.300	0.272	0.485	0.415	0.670	0.543	0.855	0.658	1.040	0.763
0.120	0.1152	0.305	0.276	0.490	0.418	0.675	0.546	0.860	0.661	1.045	0.765
0.125	0.1198	0.310	0.280	0.495	0.422	0.680	0.549	0.865	0.664	1.050	0.768
0.130	0.1243	0.315	0.284	0.500	0.426	0.685	0.553	0.870	0.667	1.055	0.771
0.135	0.1289	0.320	0.288	0.505	0.429	0.690	0.556	0.875	0.670	1.060	0.773
0.140	0.1335	0.325	0.292	0.510	0.433	0.695	0.559	0.880	0.673	1.065	0.776
0.145	0.1371	0.330	0.296	0.515	0.436	0.700	0.562	0.885	0.676	1.070	0.778
0.150	0.1425	0.335	0.300	0.520	0.440	0.705	0.565	0.890	0.679	1.075	0.781
0.155	0.1470	0.340	0.304	0.525	0.444	0.710	0.569	0.895	0.682	1.080	0.784
0.160	0.1515	0.345	0.308	0.530	0.447	0.715	0.572	0.900	0.684	1.085	0.786
0.165	0.1560	0.350	0.312	0.535	0.451	0.720	0.575	0.905	0.687	1.090	0.789
0.170	0.1605	0.355	0.316	0.540	0.454	0.725	0.578	0.910	0.690	1.095	0.792
0.175	0.1649	0.360	0.320	0.545	0.458	0.730	0.581	0.915	0.693	1.100	0.794
0.180	0.1693	0.365	0.324	0.550	0.461	0.735	0.585	0.920	0.696	1.105	0.797
0.185	0.1737	0.370	0.328	0.555	0.465	0.740	0.588	0.925	0.699	1.110	0.799
										1.115	0.802

C = concentration in grams of nitrate per 100 mL of solution.

12.2 To facilitate the calculation of intrinsic viscosity the relationship between η_{sp} and $[\eta]C$ is given in Table 1.

FRACTIONATION OF CELLULOSE NITRATE

13. Apparatus

13.1 *Centrifuge*, with head and trunnion cups for 250-mL centrifuge bottles.

13.2 *Centrifuge Bottles*, 250-mL round-bottom, with temperature-resistant caps, rubber, 1.5-in. (38.1 mm).

13.3 *Constant-Temperature Bath*, $25 \pm 0.1^\circ\text{C}$.

13.4 *Laboratory Stirrer*, with a glass auger-type agitator, $\frac{1}{2}$ -in. (12.7-mm) diameter.

13.5 *Microburet*, 10-mL, with 0.02-mL subdivisions.

13.6 *Shaker*.⁶

13.7 *Spatula*, stainless steel.

13.8 *Oven*, forced draft, 50°C .

14. Material

14.1 *Acetone-Water Mixture*—Mix 91 volumes of acetone (reagent grade) with 9 volumes of water.

TABLE 2 Sample Weight Versus Degree-of-Polymerization Level of Unfractionated Sample

Degree-of-Polymerization Level	g of sample/200 mL of Solvent
3000	0.2
2000	0.4
1000	0.8
500	1.0
250	1.2

15. Procedure

15.1 Redry the nitrate sample for 1 h at 50°C . Select from Table 2 the appropriate sample weight corresponding to the DP level of the unfractionated sample, and weigh the specified amount to the nearest 0.0001 g. Transfer the specimen to an 8-oz plastic capped bottle, add 200 mL of the acetone-water mixture, close the bottle with a cap lined with tin foil, and place it on the shaker to dissolve.

NOTE 7—The use of the 91+9 acetone-water mixture instead of pure acetone in the solution step gives faster dissolving action and at the same time brings the solution to the point of threshold precipitation, avoiding the necessity of adding relatively large amounts of water prior to precip-

TABLE 3 Sequence of Water Addition

Fraction No.	Degree-of-Polymerization Level				
	3000	2000	1000	500	250
	For Precipitation of Fraction Add Millilitres of Water as Shown				
1	0.5	0.7	1.2	1.7	2.7
2	0.2	0.2	0.3	0.3	0.3
3	0.3	0.3	0.4	0.4	0.4
4	0.35	0.35	0.5	0.5	0.6
5	0.4	0.4	0.6	0.6	0.8
6	0.5	0.6	0.7	0.8	1.0
7	0.6	0.7	0.8	0.9	1.2
8	0.7	0.9	1.2	1.4	1.8
9	1.5	2.5	4.0	5.0	6.0
10	Evaporate to one half initial volume, cool, centrifuge, decant				
11	Evaporate to one fourth initial volume, cool, centrifuge, decant				
12	Evaporate to dryness				

ation of the first fraction. Mild heating (30°C) is sometimes required to effect solution, particularly of material of very high degree of polymerization. In such cases, the tendency to gel formation can be lessened by adding the nonsolvent water first followed by addition of the acetone. These additions would be in place of the 91+9 acetone-water mixture, but the quantities would remain the same.

15.2 Transfer the solution to a 250-mL, round-bottom centrifuge bottle, and place it in the constant-temperature bath at 25°C. Immerse the tip of a microburet filled with water in the solution, adjusting the position so that the auger stirrer and buret tip do not make contact. Stir the solution rapidly and add the desired increment of water slowly from the buret.

NOTE 8—The successive increments of water that need be added in order to separate the sample into about 12 fractions of roughly equivalent weight are critical. As a guide, Table 3 gives sequences of water additions that have been found to give satisfactory weight separation at several initial DP levels for the sample weights outlined in Table 2. Experience will help to judge minor adjustments that may be needed for various nitrates. Watch the precipitation carefully, and if any precipitate tends to cling to the buret nozzle, remove it promptly to prevent the formation of large stringy masses. This tendency is apparent only with the early fractions of nitrate having a high degree of polymerization, particularly if too high an initial concentration of nitrate has been chosen. With proper precipitation technique, the fractions appear as finely divided floc.

15.3 After the precipitation of a fraction, cap the centrifuge bottle and centrifuge the dispersion until the precipitate has completely settled (approximately 5 min at 500 g). Decant the supernatant liquor into a second centrifuge bottle

and set it aside for precipitation of the next fraction.

15.4 Transfer the settled residue, comprising the first fraction, to a weighing bottle. To facilitate drying, press the gel-like fraction into a thin film using the spatula to squeeze out the excess liquid. Add 1 to 3 mL of water to agglomerate (the higher degree of polymerization fractions agglomerate as films, the lower ones as powders). Whenever possible to do so without loss of nitrate, pour off the excess solution. Place the weighing bottle containing nitrate in the 50°C oven until constant weight is attained (usually about 2 h).

NOTE 9—For all but fractions with very low degree of polymerization, the centrifuged residue can be shaken out of the centrifuge bottle in a relatively solid piece that requires no rinsing for quantitative transfer to the weighing bottle. The fractions of a low degree of polymerization are somewhat more difficult to transfer, sometimes requiring a rinse for clean removal.

15.5 In a similar manner, precipitate and recover the next eight fractions. To recover fraction No. 10, evaporate on a water bath the supernatant liquor from fraction No. 9 to one half its initial volume, cool, centrifuge, and decant. In a similar manner, evaporate the supernatant liquor from fraction No. 10 to one half its volume to recover fraction No. 11. Evaporate to dryness the supernatant liquor from fraction No. 11 to obtain the final fraction, No. 12. The entire precipitation fractionation should be completed within one 6-h period.

16. Measurements on Cellulose Nitrate Fractions

16.1 Determine for each fraction the weight of cellulose nitrate precipitated and the degree of polymerization following the procedure described in Sections 9 to 12.

16.2 Plot the integral distribution curve (degree of polymerization *versus* cumulative percentage) and differential distribution curve (amount at each chain length *versus* degree of polymerization).

NOTE 10—To prepare the integral distribution curve, plot the cumulative percent as the abscissa and the degree of polymerization as the ordinate. To construct a differential plot draw a smooth curve through the stepwise integral plot, ignoring minor deviations from the mid-point of individual fractions. From this smooth curve pick the amount (in percentage of total) of material falling within successive suitably spaced degree-of-polymerization ranges, for example, 0 to 25, 25 to 50, 50 to 100, 100 to 200, etc. Divide each percentage by the increment range of degree of polymerization covered to give the average amount of each chain length in that increment. Plot this "amount for each increment range" against the degree of polymerization to give a stepwise differential curve. Draw a smooth curve through the stepwise curve ignoring minor deviations from the mid-point of individual steps in such a manner that the area under the curve totals 100 %.

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Standard Test Method for Viscosity of Resin Solutions¹

This standard is issued under the fixed designation D 1725; the number immediately following the designation indicates the year of original adoption 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—Editorial corrections were made throughout in April 1989.

1. Scope

1.1 This test method covers the measurement of the viscosity of resin solutions.

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 154 Guide for Testing Varnishes²

D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method³

3. Summary of Test Method

3.1 Solid resins are dissolved in organic solvents by cold-cut or hot-cut methods in the laboratory. The viscosity of such prepared solutions, or of commercial solutions of resins is then determined by the bubble time method (Test Method D 1545). The bubble seconds are approximately equal to stokes.

4. Apparatus

4.1 *Bath*, constant-temperature, consisting of a cylindrical glass jar of about 5-gal capacity, or an aquarium tank with controls capable of maintaining the temperature at $25 \pm 0.1^\circ\text{C}$ with water as the bath medium.

4.2 *Bottles*, 8-oz (225-mL), wide-mouth, screw cap.

4.3 *Cellophane*, sheet, cut into 4 or 5-in. (102 or 127-mm) squares.

4.4 *Corks*, No. 2 short taper, to fit viscosity tubes.

4.5 *Holder for Viscosity Tubes*,⁴ preferably a mechanical holder with rack and pinion for inverting the tubes. The holder should be checked with a small level and T-square to make certain it holds the tubes in an exact vertical position after being placed in the constant-temperature bath.

4.6 *Bottle Shaker*, preferably one which will give end-over-end agitation.

4.7 *Timing Device*, such as a stopwatch or electric stop clock capable of being read to a precision of 0.1 s.

4.8 *Viscosity Tubes*,⁵ of clear glass and flat bottoms, having 10.65 ± 0.025 -mm inside diameter and 114 ± 1 -mm outside length. Plainly legible lines shall be located on the tubes as follows:

27 ± 0.5 mm
 100 ± 0.5 mm
 108 ± 0.5 mm

All distances shall be measured from the bottom outside of the tube. The distance between the first and second lines shall be 73 ± 0.5 mm.

5. Solvent

5.1 The solvent used should be mutually agreed upon between the purchaser and the manufacturer. Toluene, xylene, mineral spirits, alcohols, etc., are used for the preparation of solutions of resins. For a given resin, the viscosity obtained will depend on the solvent used. In case of dispute, both laboratories should use portions of the same batch of solvent.

6. Preparation of Resin Solutions

6.1 The procedure employed for dissolving solid resins will be dependent upon the chemical nature of the resin under test. Some resins will dissolve readily in cold solvent with moderate agitation, while others must be hot-cut in order to effect solution. The method of solution shall be agreed upon mutually between the purchaser and the seller since there may be differences in solubility or viscosity between solutions prepared with and without heat. The solvent concentration may vary from 30 to 90 % and is generally selected to give a solution having a viscosity between 5 and 300 s. With hard resin use only material taken from freshly broken lumps. Do not use crushed or powdered material from the resin sample container.

6.2 *Cold-Cut Solutions*—Because it is not easy to tell when solution of the resin is complete, proceed exactly in accordance with the following directions, unless experience has shown that short cuts can be safely taken:

6.2.1 Prepare 100 g of solution as follows: Weigh an 8-oz (225-mL), screw-cap bottle, cap, and cellophane sheet 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 D01.33 on Polymers and Resins.

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² *Annual Book of ASTM Standards*, Vol 06.01.

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

⁴ A suitable mechanical holder is available from the Gardner Laboratory, Inc., 5521 Landy Lane, Washington, DC, Item 660.

⁵ Viscosity tubes are available from the Gardner Laboratory, Inc., 5521 Landy Lane, Washington, DC, Item 555, or from the R. P. Cargille Laboratories, Inc., 118 Liberty St., New York, NY 10805.

nearest 0.05 g. Weigh into the bottle to the nearest 0.05 g the appropriate amount of solvent (40 g for a 60 % solution).

6.2.2 Weigh into a beaker to the nearest 0.05 g the correct amount (60 g for a 60 % solution) of the freshly crushed resin, passing a No. 10 (2000- μ m) sieve.

6.2.3 Pour the resin slowly into the bottle containing the solvent, swirling gently so as to wet the resin as thoroughly as possible as it is added. Place the cellophane sheet over the mouth of the bottle and screw on the cap tightly.

6.2.4 Immediately shake vigorously by hand and place on shaker to give end-over-end agitation for an overnight period.

6.2.5 Check the weight of the bottle and solution to make certain that none was lost during the mixing. If appreciable loss has occurred, discard the solution and prepare a fresh one.

6.3 Hot-Cut Solutions:

6.3.1 Prepare 100 g of solution as follows: Weigh a 250-mL Erlenmeyer flask with a ground joint to the nearest 0.05 g. Weigh into the flask to the nearest 0.05 g the appropriate amount of solvent (40 g for a 60 % solution).

6.3.2 Weigh into the flask to the nearest 0.05 g the correct amount (60 g for a 60 % solution) of the freshly crushed resin passing a No. 10 (2000- μ m) sieve. Connect the flask to a suitable air or water condenser and warm gently on a hot plate with swirling as necessary to prevent any charring or scorching of the resin. Usually, the solution will be complete in 15 or 20 min.

6.3.3 When the solution is complete, cool the flask, disconnect, and weigh. If over 0.1 g of solvent has been lost, add the necessary amount and swirl to mix.

6.3.4 If any air bubbles are present, allow the solution to stand until they disappear.

6.4 *Resins Supplied in Solution*—These resins ordinarily can be tested for viscosity as received. However, if modification is necessary, such as a reduction to a specified solids

content, use a solvent or solvent combination suitable for use with resin under test, as agreed upon between the manufacturer and the purchaser.

7. Procedure

7.1 Determine the viscosity of the resin solution in accordance with Test Method D 1545, repeating the timing determination until three readings are obtained that agree within 10 s or 3 % relative, whichever is larger.

7.2 For viscosities of solutions which have a timed bubble travel of 5 s or less, more precise results can be obtained by comparison against reference standards having predetermined viscosity (Test Method D 154) or timed bubble travel (Test Method D 1545).

8. Report

8.1 Report the following information:

8.1.1 The mean bubble travel time, in seconds, as the viscosity at 25°C, including:

8.1.1.1 Solution concentration,

8.1.1.2 Solvent used, and

8.1.1.3 Whether cold- or hot-cut solutions were used.

9. Precision

9.1 *Repeatability and Reproducibility*—Two results, each the mean of three readings, should be considered suspect if they differ by more than 7 % relative (95 % confidence level).

NOTE—This estimate of precision is based on an interlaboratory study.⁶ Because the data showed no significant difference for between laboratory and within laboratory variances, the precision estimates for repeatability and reproducibility were pooled and a single value reported.

⁶ For the results of the interlaboratory study, see Report of Committee D-1, *Proceedings*, ASTM, Vol 60, 1960.

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Standard Test Methods for Hydrolyzable Chloride Content of Liquid Epoxy Resins¹

This standard is issued under the fixed designation D 1726; the number immediately following the designation indicates the year of original adoption 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 easily hydrolyzable chloride content of liquid epoxy resins which are defined as the reaction product of a chlorohydrin and a di- or polyfunctional phenolic compound.

1.1.1 In Test Method A, the easily hydrolyzable chloride is saponified with potassium hydroxide and directly titrated with hydrochloric acid. This test method can be used for concentrations of 1 weight % and below.

1.1.2 In Test Method B, the easily hydrolyzable chloride is again saponified with potassium hydroxide, then titrated potentiometrically with silver nitrate. This test method can be used for concentrations of 5 to 2500 ppm hydrolyzable chloride.

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.* For specific hazard statements see Sections 8 and 14.

2. Referenced Documents

2.1 ASTM Standards:

- D 841 Specification for Nitration Grade Toluene²
- D 1193 Specification for Reagent Water^{2,3}

3. Summary of Test Method

3.1 The sample is refluxed in the presence of a known amount of 0.1 N alcoholic potassium hydroxide. The amount of potassium hydroxide consumed in the hydrolysis is a measure of the hydrolyzable chloride content of the resin.

4. Significance and Use

4.1 The hydrolyzable chloride content of liquid epoxy resins is an important variable in determining their reactivity and the properties of coatings made from them. These test methods may be used to determine the hydrolyzable chloride content of manufactured epoxy resins and confirm the stated hydrolyzable chloride content of purchased epoxy resins.

5. Purity of Reagents

5.1 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,⁴ where such specifications are available. 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

TEST METHOD A

6. Apparatus

6.1 *Reflux Apparatus*, consisting of a 250-mL Erlenmeyer flask attached to a reflux condenser and a hot plate with variable heat control.

7. Reagents and Materials

7.1 *Hydrochloric Acid, Standard (0.1 N)*—Dilute 9 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 1 L with water. Standardize against 0.25 g of sodium carbonate (Na_2CO_3) accurately weighed and dissolved in 75 to 100 mL of water.

7.2 *Methyl Ethyl Ketone*.

7.3 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 mL of methanol, ethanol, or isopropanol.

7.4 *Potassium Hydroxide, Alcohol Solution (0.1 N)*—Dissolve 5.5 to 6.0 g of potassium hydroxide (KOH) in 1 L of methanol (99 %) or ethanol conforming to Formula No. SD-30 of the U.S. Bureau of Internal Revenue. No standardization of the solution is necessary.

7.5 *Toluene* (Warning—See Section 8.), conforming to Specification D 841.

8. Hazards

8.1 Hydrochloric acid and potassium hydroxide are corrosive. Toluene and methyl ethyl ketone are flammable and their vapors can be harmful. Precautions should be taken to avoid inhalation and skin or eye contact with these chemicals. All sample preparations should be done in a well ventilated area, such as a fume hood.

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.

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 11.01.

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."

Procedure

Weigh to the nearest 1 mg, 6 to 8 g of neutral specimen into a 250-mL glass-stoppered Erlenmeyer flask. By means of a pipet, transfer 50.0 mL of 0.1 *N* alcoholic KOH solution into the flask and add 15 mL of toluene. Stopper the flask and swirl to mix. Add a few boiling aids and attach to the reflux condenser on the hot plate.

2.2 Allow the solution to reflux gently for 15 ± 1 min. At the end of the reflux period, remove the flask from the hot plate and cool to room temperature with the condenser in place.

2.3 Remove the condenser, add 3 drops of phenolphthalein indicator solution to the specimen, and titrate with 0.1 *N* HCl. The end point is taken when 1 drop changes the solution from pink to colorless.

NOTE 1—Add 100 mL of methyl ketone to the specimen if required to ensure a homogeneous solution during titration of the excess KOH solution.

2.4 Make a blank determination on the reagents following the same procedure but omitting the specimen.

10. Calculation

10.1 Calculate the weight percent hydrolyzable chloride content, *H*, of the specimen as follows:

$$H = [(V - B)N \times 3.55]/W \quad (1)$$

where:

- B* = HCl required for titration of the blank, mL,
- V* = HCl required for titration of the hydrolyzed specimen, mL,
- N* = normality of the HCl,
- 3.55 = grams of chlorine per milliequivalent multiplied by the percentage factor of 100, and
- W* = specimen weight, g.

11. Precision

11.1 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 0.02 % absolute.

11.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 0.05 weight %.

TEST METHOD B

12. Apparatus

12.1 *Reflux Apparatus*, consisting of a 250-mL Erlenmeyer flask attached to a reflux condenser.

12.2 *Hot Plate*, with variable heat control.

12.3 *Magnetic Stirrer*, with polytetrafluoroethylene (PTFE)-coated stirring bar.

12.4 *Buret or Automatic Titrator*.

12.5 *Silver Electrode* or equivalent.⁵

12.6 *Boiling Chips*.

⁵ A Mettler DM 141 Silver Electrode, manufactured by Mettler Instruments, Princeton Hightstown Rd., P.O. Box 71, Hightstown, NJ 08520, or equivalent has been found satisfactory for this purpose.

13. Reagents and Material

13.1 *Acetone*.

13.2 *Bromcresol Green Indicator Solution (0.1 %)*—Dissolve 0.1 g of bromcresol green in 100 mL of water.

13.3 *Nitric acid*, (HNO₃) (1 + 1), diluted with water.

13.4 *Potassium Hydroxide*, alcohol solution (0.1 *N*)—Dissolve 5.6 g of potassium hydroxide (KOH) in 1 mL of methanol (99 %) or ethanol conforming to Formula No. SD-30 of the US Bureau of Internal Revenue. No standardization of the solution is necessary.

13.5 *Silver Nitrate*, alcohol solution (0.0025 *N*)—Dissolve 0.425 g of silver nitrate (AgNO₃), weighed to the nearest 1 mg, in 1 L of methanol (99 %) or ethanol conforming to Formula No. SD-30 of the US Bureau of Internal Revenue. Standardize against hydrochloric acid or sodium chloride solution (with traceability to an NIST standard).

13.6 *Toluene*.

14. Hazards

14.1 Nitric acid, potassium hydroxide, and silver nitrate are corrosive. Acetone and Toluene are flammable and their vapors can be harmful. Precautions should be taken to avoid inhalation and skin or eye contact with these chemicals. All sample preparations should be done in a well ventilated area, such as a fume hood.

15. Procedure

15.1 Weigh 2 to 3 g of specimen, to the nearest 0.005 g, into a 250-mL Erlenmeyer flask. Add 20 mL of toluene, 20 mL of acetone, and 50 mL of 0.1 *N* alcoholic KOH. Swirl or mix until dissolution is complete.

15.2 Add several boiling chips, connect the flask to the reflux condenser, and gently reflux for 15 ± 1 min on a hot plate.

15.3 Remove the hot plate from under the flask and allow the flask and contents to cool to room temperature. Rinse down the condenser with acetone then remove from the flask.

15.4 Quantitatively transfer the contents of the flask to a 250-mL beaker using acetone as wash solution. Dilute the solution to about 125 mL with acetone, insert a stirring bar, and place on a magnetic stirrer.

15.5 Add five drops of bromcresol green indicator. While stirring add 1 + 1 nitric acid dropwise just until the permanent color changes from blue to yellow.

NOTE 2—Caution—Do not add excess nitric acid. Do not acidify the solution until ready to begin the titration. Make certain that the solution is at room temperature before acidifying. These cautions are necessary to prevent the chloride results from being low due to recombination with the resin.

15.6 Titrate with 0.0025 *N* silver nitrate using the combination silver electrode and the automatic titrator or buret.

15.7 Reflux a blank, which includes all components except the sample, and titrate with 0.0025 *N* silver nitrate.

16. Calculation

16.1 Calculate the parts per million hydrolyzable chloride content, *H*, of the specimen as follows:

$$H = [(V - B)N \times 35.5 \times 10^3]/W$$

where:

- B = AgNO_3 required for the titration of the blank, mL,
 V = AgNO_3 required for the titration of the hydrolyzed specimen, mL,
 N = Normality of the AgNO_3 ,
 35.5×10^3 = grams of chlorine per milliequivalent multiplied by the ppm conversion of 1×10^6 , and
 W = specimen weight, g.

17. Precision

17.1 A liquid epoxy resin of approximately 150 ppm hydrolyzable chloride was sampled to five laboratories and

seven analysts obtained the following results.

17.1.1 *Repeatability*—The difference between two results obtained by the same analyst should not vary by more than 8.32 % at the 95 % confidence level.

17.1.2 *Reproducibility*—The difference between two results, each the mean of two determinations, obtained by analysts in different laboratories should not vary by more than 15.88 % relative at the 95 % confidence level.

18. Keywords

18.1 hydrolyzable chloride; liquid epoxy resin; saponifiable chloride

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Standard Test Method for Pentosans in Cellulose

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Scope

This test method covers the determination of pentosans in cellulose by the orcinol colorimetric method, in range from less than 1 to about 16 %.

NOTE 1—This test method is an adaptation of the method designated D 2456 by the Analysis Committee of the Cellulose Industry of the British Association of Pulp and Paper Engineers.

NOTE 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 1193 Specification for Reagent Water²
- D 1348 Test Methods for Moisture in Cellulose³

3. Summary of Test Method

3.1 The cellulose is treated with boiling hydrochloric acid (approximately 12 % or 3 + 7) to hydrolyze the pentosans to pentoses, which are then converted to furfural. The furfural is distilled and collected. Hydroxymethyl furfural is also produced during the distillation, but does not interfere in the following colorimetric determination.

3.2 An aliquot of the distillate is treated with orcinol reagent, and after standing for 2 h the absorbance is measured at 630 nm. By reference to a calibration curve prepared from known furfural solutions, the percent pentosans is calculated.

4. Significance and Use

4.1 This test method is used for measuring the level of hemicelluloses in wood pulps. The amount of pentosans, primarily arabinose and xylose, are measures of the purity of wood pulps used in chemical cellulose applications.

5. Apparatus

5.1 *Flask*, two-neck round-bottom, 500-mL, standard-taper 24/40 joint.

5.2 *Dropping Funnel*, 500-mL, with standard-taper 24/40 neck.

5.3 *Distillation Connecting Tube*, with standard-taper 24/40 inner joints to fit the round-bottom flask and the Graham condenser, respectively, with the proper angles to allow for the vertical position of the condenser. If a thermometer is to be used, the adapter should have a standard-taper 10/30 thermometer well.

5.4 *Condenser*, Graham, water-cooled, with standard-taper 24/40 connections.

5.5 *Adapter*, with standard-taper 24/40 joints with gas inlet.

5.6 *Receiving Flask*, 500-mL, with standard-taper 24/40 joint, to collect the distillate.

5.7 *Ice Bath*, for receiving flask to help prevent loss of furfural.

5.8 *Heating Mantle*, for reaction flask, controlled by a variable transformer. A gas burner may be substituted for the heating mantle.

5.9 *Constant-Temperature Bath*, capable of maintaining a temperature within $\pm 0.5^\circ\text{C}$ of the temperature selected for development of the color with orcinol, which temperature should be between 15 and 30°C .

5.10 *Filter Photometer or Spectrophotometer*, suitable for measuring absorbance at 630 nm.

6. Purity of Reagents

6.1 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.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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

6.3 *Ethanol*, 95 %, aldehyde-free.

6.4 *Furfural*—Distill furfural immediately before use. Use only freshly-distilled pale yellow material with a boiling point of 162°C . It is preferable to distill in vacuum.

6.5 *Hydrochloric Acid* (3 + 7)—Mix 3 volumes of concentrated hydrochloric acid (HCl, sp gr 1.19) with 7 volumes of water. The concentration will be approximately 12 %.

6.6 *Orcinol Solution*—Dissolve 0.40 g of orcinol and 0.5 g of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in 1 L of 11.0 N HCl. Discard solutions more than 2 weeks old.

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 D01.36 on Celluloses.

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² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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."

7. Preparation of Calibration Curve

7.1 Dissolve about 0.5 g furfural, weighed to an accuracy of ± 0.2 mg, in HCl (3 + 7) and dilute to 500 mL with HCl (3 + 7) in a volumetric flask. After mixing thoroughly, pipet 5, 10, 15, 20, 25, 30, and 50 mL of this solution into separate 100-mL volumetric flasks and dilute to the mark with HCl (3 + 7). This gives solutions with 50, 100, 150, 200, 250, 300, and 500 mg of furfural per litre.

NOTE 2—Higher or lower concentrations of standards may be prepared to fit specific conditions.

7.2 Pipet 10 mL of each of these diluted solutions into separate 50-mL volumetric flasks, and add 25 mL of orcinol solution to each. Place the flasks in a constant-temperature bath.

NOTE 3—The color intensity is dependent on the temperature during the reaction of furfural with orcinol. Any temperature between 15 and 30°C may be used, provided the temperature is the same during the pentosans determination as in the preparation of the calibration curve. If available, a constant-temperature room may be used.

7.3 Prepare a blank containing all the reagents except the furfural solution.

7.4 After 60 ± 5 min, almost fill the volumetric flasks with ethanol, mix, bring to the temperature of the bath, fill to the mark with ethanol, and again mix thoroughly. Place in the bath for a second 60 ± 5 min period. Measure the absorbance at 630 nm, and correct for the reading on the reagent blank.

7.5 Plot absorbance versus milligrams of furfural to give a calibration curve.

8. Procedure

8.1 The size of specimen to be taken depends on its pentosan content. Select the proper size specimen and suitable size aliquot for the subsequent colorimetric measurement as follows:

Pentosans Content, %	Specimen Size, g	Size of Aliquot, mL
<1	4	10
1 to 3	3	10
3 to 5	2	10
5 to 10	1	10
>10	1	5 (plus 5 mL of HCl (3+7))

8.2 Allow the specimen to come to moisture equilibrium with the atmosphere of the balance. Weigh to the nearest milligram and transfer it to the 500-mL round-bottom flask. At the same time weigh a specimen for moisture determination in accordance with Test Methods D 1348. Add 100 mL of HCl (3 + 7) to the flask, washing down any fibers adhering to the sides of the flask. Assemble the distillation apparatus and mark the flask to indicate the liquid level.

NOTE 4—If the room air temperature is above 20°C, cool the receiver with an ice bath.

8.3 Add 300 mL of HCl (3 + 7) to the dropping funnel. With a glass-marking pencil or permanent glass-marking ink, divide the volume into six portions, one portion for each 15-min period. (Any convenient number of divisions that will enable the operator to keep track of the amount of HCl (3 + 7) added with time is satisfactory.)

8.4 Start timing the distillation when the distillate first starts to appear, and distill for 90 min, collecting in that time 300 mL of distillate in the receiver. Allow the acid to drop into the distillation flask from the separatory funnel at such a

rate that the original 100-mL level is maintained. It is important that the proper rate of distillation be maintained and that 300 mL of distillate be collected. The receiver should be marked at the 300-mL level.

NOTE 5—It is important to avoid overheating the flask above the liquid level.

8.5 Transfer the contents of the receiver to a 500-mL volumetric flask. Rinse the receiver (Note 6) with HCl (3 + 7), and add the rinsings to the volumetric flask. Dilute to the mark with HCl (3 + 7) and mix well.

NOTE 6—If a 500-mL volumetric flask, marked at the 300-mL level, is used as the receiver, transfer of the distillate and rinsing of the receiver are avoided. Proper choice of specimen size and aliquot will allow the use of a 300-mL volumetric flask as the receiver. The distillation can be stopped just short of the mark to allow precise adjustment of the volume.

8.6 Pipet 10 mL of the diluted distillate into a 50-mL volumetric flask, add 25 mL of orcinol solution, and place the flask in the constant-temperature bath. After 60 ± 5 min almost fill the flask with ethanol, mix, bring to the temperature of the constant-temperature bath, fill to the mark with ethanol, and mix thoroughly. Place in the bath for a second 60 ± 5 -min period. Measure the absorbance of the blue color at 630 nm, using a blank containing 10 mL (or whatever size aliquot is used) of HCl (3 + 7), instead of the distillate, as a reference solution. Read the furfural concentration of the distillate from the calibration curve.

9. Calculation

9.1 The theoretical factor for converting pentosans to furfural is 0.727, which is the quotient of the molecular weight of furfural, 96, divided by the molecular weight of anhydroxylan, 132. Assuming that the pentosans content of wood consists principally of xylan that is converted to furfural with a yield of 88 percent, the empirical factor for converting furfural values to pentosans is:

$$1/(0.727 \times 0.88) = 1.563$$

9.2 Calculate the pentosans content of the specimen as follows:

$$\text{Pentosans, \%} = [A/(B \times [(100 - C)/100])] \times 1.563 \times 100$$

where:

A = furfural in the distillate, g,

B = specimen used, g, and

C = moisture in the specimen, %.

10. Report

10.1 Report the pentosans content of the cellulose as a percent of the moisture-free sample. Make all determinations at least in duplicate, and report the average value.

11. Precision and Bias

11.1 A general guide for the agreement between duplicates and the number of significant figures is as follows:

Pentosans, %	Agreement, %	Nearest Significant Figures to be Reported, %
<2	0.1	0.1
2 to 10	0.4	0.1
>10	1.0	0.5

11.2 No statement on bias can be made as no suitable reference material exists for determining bias.

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Standard Test Method for Alcohol-Benzene Soluble Matter in Cellulose¹

This standard is issued under the fixed designation D 1794; the number immediately following the designation indicates the year of original adoption 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.

1. Scope

1.1 This test method covers the determination of alcohol-benzene soluble matter in cellulose and is applicable to dissolving-type cellulose pulps prepared from cotton or wood.

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 1348 Test Methods for Moisture in Cellulose²

2.2 *Other Documents:*

Occupational Safety and Health Standards and Interpretations, Part 1910³

3. Summary of Test Method

3.1 A sample is extracted with a 1 + 2 alcohol-benzene solution in a Soxhlet apparatus as a measure of the waxes, fats, resins, and oils present.

4. Significance and Use

4.1 This test method provides an estimate of waxes, lipids, resins, and other organic soluble residues that were not removed from the fiber during the pulping process.

5. Apparatus

5.1 *Extraction Apparatus*, Soxhlet, consisting of a 300-mL flask, an extractor with a 50 by 250-mm body, and a Hopkins type condenser.

5.2 *Extraction Thimble*, either (1) standard thickness paper, 43 by 123 mm, (2) Alundum, 45 by 127 mm, medium-porosity, or (3) glass with coarse-porosity fritted-glass disk, 45 by 130 mm.

5.3 *Extraction Heater and Support*, heater with three-heat switches.

5.4 *Oven*, gravity-convection, maintained at $105 \pm 3^\circ\text{C}$.

5.5 *Dish*, evaporating, milk, aluminum, 75 mm in diameter by 20 mm deep.

5.6 *Desiccator* with efficient desiccant such as anhydrous calcium sulfate, activated alumina, or magnesium perchlorate. Calcium chloride is not satisfactory.

6. Reagent

6.1 *Alcohol-Benzene Solvent*—Mix 1 volume of Formula 30 ethyl alcohol with 2-volumes of benzene.

NOTE 1: Caution—Benzene is regulated as a "cancer hazard" as outlined in OSHA Regulations.

7. Procedure

7.1 Weigh 8 to 12 g of loose pulp, to the nearest 0.01 g, into an extractor thimble that has previously been extracted with the alcohol-benzene solvent. For sheet pulp, cut a sample into strips about $\frac{1}{4}$ in. (6.4 mm) wide and 3 in. (76 mm) long, and weigh about 20 g into a thimble. Weigh a separate portion for a moisture determination in accordance with Methods D 1348.

7.2 Place the extraction thimble with specimen in the extractor and connect the flask. Pour 250 mL of the alcohol-benzene solvent into the body of the extractor. Connect the assembled extractor to the condenser and place the flask on the extraction heater. Turn on the cooling water to the condenser and adjust the heater to cause the solvent to reflux at such a rate as to cause siphoning 6 to 8 times/h. Continue the extraction for 5 h, beginning the timing when the solvent first begins to drop from the condenser into the extractor.

7.3 When the extraction is complete, turn off the heater. Disconnect the flask at a time when most of the solvent has collected in the extractor.

7.4 Heat the evaporating dish in the oven at 105°C for 30 min, cool in a desiccator, and weigh to the nearest 0.0001 g. Filter the solution from the flask through a sheet of qualitative paper into the weighed drying dish. Rinse the flask and filter with three 5-mL portions of the alcohol-benzene solvent.

7.5 Place the dish on a steam bath and evaporate just to dryness. Then place the dish in the oven at 105°C for 1 h, cool in a desiccator, and weigh.

8. Calculation

8.1 Calculate the percent of alcohol-benzene soluble matter, S , on the dry basis as follows:

$$S = [(A - B)/W \times \{(100 - M)/100\}] \times 100$$

where:

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 D01.36 on Cellulosics.

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

³ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

- W_1 = weight of dish and residue, g,
- W_2 = weight of dish, g,
- W_3 = weight of specimen, g, and
- M = moisture in specimen, % (Test Methods D 1348).

9. Report

9.1 Report the alcohol-benzene soluble matter to the nearest 0.01 %.

10. Precision and Bias

10.1 At an extractive level of 0.13 %, the relative standard deviation is 10 %.

10.2 No statement on bias can be made as no suitable reference material exists for determining bias.

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Standard Test Method for Intrinsic Viscosity of Cellulose¹

This standard is issued under the fixed designation D 1795; the number immediately following the designation indicates the year of original adoption 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 intrinsic viscosity of purified celluloses such as bleached wood pulps, cotton linters, and regenerated cellulose. It is applicable to all cellulose samples with an intrinsic viscosity of 15 dl/g or less. Most native (unpurified) celluloses have intrinsic viscosity values too high for measurement by this test method.

NOTE 1—The use of cuprammonium hydroxide solution for regular viscosity determination is described in Test Methods D 539, Method T 206 m-55 of the Technical Association of Pulp and Paper Industry on "Cuprammonium Disperse Viscosity of Pulp," and Joint Army-Navy Specifications JAN-C-206.

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:

- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²
- D 539 Test Methods for Apparent Fluidity of Dispersions of Cellulose Fibers³
- D 629 Test Methods for Quantitative Analysis of Textiles⁴
- E 1 Specification for ASTM Thermometers⁵

3. Summary of Test Method

3.1 A weighed sample of the material is dissolved in a 0.5 M cupriethylenediamine hydroxide solution. The viscosity of this solution, and also that of the solvent, is determined at 25°C by means of a calibrated glass capillary-type viscometer. The relative viscosity is calculated and the corresponding intrinsic viscosity is read from a table.

4. Significance and Use

4.1 This test is a sensitive measure of the degradation of cellulose resulting from the action of heat, light, acids, alkalis, oxidizing and reducing agents, and the like, used in its

processing or purification. The intrinsic viscosity value may be converted to degree of polymerization (DP) or to intrinsic fluidity, if desired.

4.2 Solutions of cellulose are not Newtonian liquids; that is, their viscosity depends upon the rate-of-shear or velocity gradient during measurement. This effect is smaller for samples of low molecular mass (DP) and at low concentrations than for high- DP samples and at high concentrations. For the celluloses and concentrations included within the limits set forth in this test method, the effect of rate-of-shear is assumed to be negligible for referee purposes. For other conditions and for research purposes this assumption may be invalid, but to discuss ways of accounting for this effect is beyond the scope of the present test method.

5. Apparatus

5.1 *Viscometer, Glass, Capillary Type*—The Cannon-Fenske, Ubbelohde, or similar capillary type instrument as described in Test Method D 445 is recommended. Viscometers described in Test Methods D 539 are also suitable. In order to avoid correction for the kinetic energy effect, choose a viscometer with a small enough capillary to give an outflow time of 80 s or more for the Cannon-Fenske type. (A size 100 viscometer is normally used for the sample solution and a size 50 for the solvent.)

5.2 *Thermometer*—ASTM Kinematic Viscosity Thermometer for use at 25°C, having a range from 19 to 27°C and conforming to the requirement for Thermometer 17C as prescribed in Specification E 1.

5.3 *Bath*—A constant-temperature bath at 25°C suitable for immersion of the viscometer so that the reservoir or the top of the capillary, whichever is uppermost, is immersed at least 50 mm, and with provision for visibility of the instrument and the thermometer. Firm supports for the viscometer shall be provided; or the viscometer may be sealed in as an integral part of the bath. Either a liquid bath with thermostatic regulation and a stirrer or a vapor bath with pressure regulation is permissible. The efficiency of the stirring and the balance between heat losses and heat input must be such that the temperature of the bath medium does not vary by more than $\pm 0.1^\circ\text{C}$ over the length of the viscometer, or from viscometer to viscometer in the various bath positions. If a vapor bath is used, there must be no temperature gradient over the length of the viscometer greater than that permitted in a liquid bath.

5.4 *Timer*—A stop watch or other spring-activated timing device or electrical timing device shall be used, graduated in divisions of 0.2 s or less, and accurate to within 0.05 % when tested over not less than a 10-min period. Such electrical timing devices shall be used only on electrical circuits of continuously controlled frequency. Frequency-controlled de-

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 D01.36 on Celluloses.

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² Annual Book of ASTM Standards, Vols 10.03 and 05.01.

³ Discontinued, see 1973 Annual Book of ASTM Standards, Part 24.

⁴ Annual Book of ASTM Standards, Vol 07.01.

⁵ Annual Book of ASTM Standards, Vol 14.03.

viscosity of suitable capacity for laboratory purposes, accurate to within 1 part in 10 000 should be used. Errors exceeding 0.05 % of a 10-min interval may occur in timing devices actuated by electrical synchronous motors driven by most public power systems, which are intermittently and not continuously controlled.

6. Reagent

6.1 *Cupriethylenediamine Hydroxide Solution* (1.00 $\pm$ 0.01 *M*), in copper, with the molar ratio of ethylenediamine to copper of 2 $\pm$ 0.1 to 1. This solvent may be prepared in the laboratory as described in Test Methods D 539. It is also commercially available.⁶

7. Reference Materials

7.1 *Viscosity Oil Standards*—Calibrating oils in the specified ranges of viscosity.⁶ Aqueous solutions of glycerol may be used instead of standardized oils; the compositions for various viscosities are given in chemical handbooks. The applicable viscosity oil standards (Note 2) are listed in Table 1.

NOTE 2—The viscosity oil standards are available only as 1-pt (4.7-m³) samples. More than 1 pt of any given oil (for example, duplicate samples) are supplied only when it is established that 1 pt is inadequate.⁶

8. Calibration of Viscometer

8.1 The following directions apply to the Cannon-Fenske viscometer (Note 3). They should be modified according to the operating instructions for other types of viscometers. The viscometers shall be calibrated (Note 4) by means of liquids having known viscosities approximately equal to those of the solvent and cellulose solutions respectively (1.2 and 12 cP, approximately).

NOTE 3—Detailed specifications and directions for filling, calibrating, and measurement with types of capillary viscometers most used are given in Test Method D 445.

NOTE 4—Calibration of the viscometers may be avoided if both solvent and solution are measured in the same instrument. Then the relative viscosity is nearly the ratio of outflow times for solution and solvent, respectively. This simplification involves two assumptions. The first, that the densities of solution and solvent are equal, holds very well for the dilute solutions used in these tests. The second, that the kinetic energy correction is zero, depends upon the choice of viscometer. If the one used gives convenient outflow times for the solution of less than 150 s, then it will be too fast for the solvent. The kinetic energy correction is zero, depending upon flow. On the other hand, if one is chosen so that the outflow time for the solvent is large enough (80 s or more), then the times for the solutions will in most cases be inconveniently long. For some work, however, it may be desirable to make some sacrifice in

accuracy or in convenience during measurement in order to avoid calibration and using two sizes of viscometers.

8.2 By means of a pipet, add 7.0 mL of the calibrating liquid to the viscometer, in a constant-temperature bath at 25 $\pm$ 0.1°C (or fill as described in Test Method D 445, Appendix A).

8.3 When the liquid has reached temperature equilibrium with the bath (in about 5 min), determine the outflow time *t* by drawing the top meniscus of the liquid above the mark between the two bulbs and measuring the time required for the meniscus to pass from this mark to the mark below the lower bulb. Take the average of two or more observations, which should differ by not more than 0.2 s.

8.4 Determine the viscometer constant *C* by the equation:

$$C = \eta / dt \quad (1)$$

where:

η = viscosity of the calibrating liquid, cP,

d = density, g/mL, and

t = time, s.

9. Preparation of Sample

9.1 To avoid undesirable effects from long heating at high temperature, samples should be air-dried and the moisture content determined on a portion that is not used for measurement of viscosity. The mass of air-dried samples is then corrected for moisture to obtain the mass of oven-dried cellulose used to calculate concentration.

9.2 Soft, sheeted pulp should be picked apart with tweezers or scraped with a dull knife. Hard-pressed or harsh pulp should be slurried in water, formed into thin sheets on a Büchner funnel, and dried at a temperature below 100°C (preferably room temperature). Loose pulp should be picked apart by hand to break up any lumps. Slurried or slush pulps should be formed into thin sheets and dried. Yarn and staple should be washed in warm water containing a little detergent to remove the finish, rinsed thoroughly, dried (at low temperature), and fluffed. (It will be found helpful to cut yarn and long staple into short lengths, say ½ in. (13 mm), before washing.) Fabrics should be cut into small pieces, desized (see Test Methods D 629, Section 8),⁷ thoroughly washed, and dried. Raveling will be helpful before dissolving samples that tend to get in the solvent. Materials containing a considerable amount of non-cellulosic matter must first be purified; such treatments lie outside the scope of this test method.

10. Preparation of Solution

10.1 The sample size is dependent upon the nature of the material, smaller masses of high-viscosity celluloses and larger masses of low-viscosity celluloses being used in order to keep the viscosity of the solutions within rather narrow limits. (Working at nearly constant viscosity reduces the effect of rate of shear upon the measurements.) The concentration for each sample is chosen according to the rule:

$$[\eta]c = 3.09 \pm 0.5 \quad (2)$$

where:

⁷ This solution may be purchased in 1-gal (3.8-L) lots from the Ecusta Paper Div., Olin-Mathieson Chemical Corp., Pisgah Forest, NC or in 5-lb (2-kg) lots from Baker and Adamson, General Chemical Div., Allied Chemical Corp., 40 Rector St., New York, NY 10006.

TABLE 1 Viscosity Oil Standards

Viscometer		Viscosity Oil Standard	
Size	Absolute Viscosity Range, cP ^a	Designation	Approximate Absolute Viscosity at 77°F (25°C), cP
50	0.9 to 3.5	S-3	3.3
100	3.3 to 13.3	S-6	7.7

^a For solution with density of 0.9.

⁶ Viscosity oil standards are available from the Cannon Instrument Co., P.O. Box 16, State College, PA 16801.

$[\eta]$ = intrinsic viscosity, dL/g, and
 c = cellulose concentration, g/dL.

Obviously, use of this rule requires knowledge of the approximate intrinsic viscosity of the sample before the concentration can be estimated. In routine control work, such information is available. If it is not, Table 2 will serve as an approximate guide.

10.2 Make up a preliminary solution of about the indicated concentration, and determine the viscosity as described in Sections 11 and 12. From the relative viscosity thus obtained, find the approximate value of the intrinsic viscosity by means of Table 3. From this determine the concentration needed to give: $[\eta]c = 3.0$. If this preliminary solution does not give a value of $[\eta]c$ of 3.0 ± 0.5 , prepare a second solution of the indicated concentration for the final viscosity measurement.

10.3 Alkaline solutions of cellulose are known to be sensitive to oxidation. Hence it is usually necessary to exclude air during solution of the sample; several ways of doing this are described in the literature, but the following is simple and adequate: Weigh out the calculated amount of air-dried cellulose (corrected to oven-dry mass) and transfer quantitatively to a suitable glass or polyethylene container (capacity somewhat more than 50 mL) that can be tightly closed by a stopper or screw cap. Add 25.0 mL of distilled water from a pipet or buret, insert the stopper or cap, and shake in order to wet out and disperse the sample. Sweep the air from the vessel with a stream of nitrogen and, with the nitrogen still flowing, add 25.0 mL of solvent. Stopper or cap tightly and shake vigorously by hand or in a mechanical shaker until the cellulose is completely dissolved.

NOTE 5—Most work pulp and regenerated celluloses dissolve within 5 min. Mercerized celluloses do not dissolve as easily in cupriethylenediamine as in cuprammonium solvent and require longer times (up to 1 h). Some operators report difficulty in dissolving native celluloses including cotton linters with DP as low as that of wood pulps. The difficulty shows up by much poorer precision of results than the 1 to 2 % that is normally obtained between measurements made with two or more solutions of the same sample. Even solutions that leave no residue on fritted-glass filters have been observed to give erratic results. Inadequate dispersion of the sample is apparently the cause, and two modifications in procedure have been recommended in such cases. One is to add about 0.04 % wetting agent to the water used to wet out the sample.⁸ The other requires use of cupriethylenediamine solutions of

⁸ Lindsley, C. H., "Rapid Dispersion of Cellulose in Cupriethylenediamine," *Textile Research Journal*, Vol 21, 1951, p. 286.

TABLE 2 Intrinsic Viscosities of Typical Samples

Type of Material	Intrinsic Viscosity, dL/g	Approximate Concentration, g/dL
Regenerated cellulose (rayons)	2 to 3	1
Dissolving pulps:		
Low viscosity	3 to 4	1
Regular viscosity	4 to 7	0.5
High viscosity	7 to 10	0.3
Cotton linters, for rayon and acetate	6 to 9	0.4
Paper (wood) pulps	3 to 8	0.4
Native celluloses ^a	15 to 30	0.1 to 0.2

^a Serious error may be introduced when this test method, which neglects effects of rate of shear upon viscosity, is used for native celluloses of high intrinsic viscosity.

two concentrations: The sample is wetted out with one solution that is 0.167 M in copper and dispersion is completed by adding the second solution, 1.000 M in copper, in such volume as to make the final copper concentration 0.500 M (see Section 14 of Test Methods D 539).

11. Measurement of Viscosity

11.1 Transfer 7.0 mL of the solution by means of a syringe or pipet to a viscometer previously placed in the bath at 25°C and flushed with nitrogen (or fill as described in Test Method D 445, Appendix A). Allow at least 5 min for the solution to reach bath temperature.

11.2 By applying either pressure (with nitrogen) or suction, draw the solution into the lower bulb of the viscometer until the top meniscus is a little above the mark between the two bulbs. Measure the time t required for the meniscus to pass from this mark to the mark below the lower bulb. Repeat at least twice and average the observations, which should not differ by more than 0.3 %.

11.3 In the same way, measure the outflow time t_0 for the solvent. This of course must be determined not for the 1.00 M solvent as prepared or purchased, but for this solvent diluted with an equal volume of water.

12. Calculations

12.1 Calculate the viscosity, η , in centipoises, as follows:

$$\eta = Ctd \quad (3)$$

where:

C = viscometer constant (Section 8),

t = outflow time, s, and

d = density, g/mL.

Calculate the relative viscosity, η_{rel} , as follows:

$$\eta_{rel} = \eta/\eta_0$$

where η_0 is the viscosity of the solvent. Since the densities of solvent and solution are practically the same, they cancel in determining relative viscosity. Hence a little work may be saved by determining the kinematic viscosity, ϵ , in centistokes, for solution and solvent:

$$\epsilon = Ct \quad (4)$$

and then

$$\eta_{rel} = \epsilon/\epsilon_0 \quad (5)$$

If both solution and solvent are measured in the same viscometer (see Note 4), relative viscosity may be obtained directly from the ratio of outflow times:

$$\eta_{rel} = t/t_0 \quad (6)$$

12.2 By means of Table 3, determine the product $[\eta]c$ corresponding to the value of the relative viscosity. From this value and the concentration, calculate the intrinsic viscosity in decilitres per gram.

NOTE 6—Table 3 gives values of $[\eta]c$ for the indicated values of η_{rel} determined by the Martin equation:

$$\log[(\eta_{rel} - 1)/c] = \log[\eta] + k[\eta]c \quad (7)$$

where $k = 0.13$. More precise values of $[\eta]$ can be obtained by finding η_{rel} for three or more concentrations, plotting $\log[(\eta_{rel} - 1)/c]$ against c and extrapolating the straight line through the points to $c = 0$. The intercept gives $\log[\eta]$.

12.3 For some purposes it is desirable to express results as degree of polymerization, since this concept is easily understood by nontechnical people who must interpret technical

TABLE 3 Intrinsic Viscosity, $[\eta]c$, at Different Values of Relative Viscosity, $\eta_{rel}^{A,B}$

η_{rel}	$[\eta]c$									
	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
1.1	0.098	0.106	0.115	0.125	0.134	0.143	0.152	0.161	0.170	0.180
1.2	0.189	0.198	0.207	0.216	0.225	0.233	0.242	0.250	0.259	0.268
1.3	0.276	0.285	0.293	0.302	0.310	0.318	0.326	0.334	0.342	0.350
1.4	0.358	0.367	0.375	0.383	0.391	0.399	0.407	0.414	0.422	0.430
1.5	0.437	0.445	0.453	0.460	0.468	0.476	0.484	0.491	0.499	0.507
1.6	0.515	0.522	0.529	0.536	0.544	0.551	0.558	0.566	0.573	0.580
1.7	0.587	0.595	0.602	0.608	0.615	0.622	0.629	0.636	0.642	0.649
1.8	0.656	0.663	0.670	0.677	0.683	0.690	0.697	0.704	0.710	0.717
1.9	0.723	0.730	0.736	0.743	0.749	0.756	0.762	0.769	0.775	0.782
2.0	0.788	0.795	0.802	0.809	0.815	0.821	0.827	0.833	0.840	0.846
2.1	0.852	0.858	0.864	0.870	0.876	0.882	0.888	0.894	0.900	0.906
2.2	0.912	0.918	0.924	0.929	0.935	0.941	0.948	0.953	0.959	0.965
2.3	0.971	0.976	0.983	0.988	0.994	1.000	1.006	1.011	1.017	1.022
2.4	1.028	1.033	1.039	1.044	1.050	1.056	1.061	1.067	1.072	1.078
2.5	1.083	1.089	1.094	1.100	1.105	1.111	1.116	1.121	1.126	1.131
2.6	1.137	1.142	1.147	1.153	1.158	1.163	1.169	1.174	1.179	1.184
2.7	1.190	1.195	1.200	1.205	1.210	1.215	1.220	1.225	1.230	1.235
2.8	1.240	1.245	1.250	1.255	1.260	1.265	1.270	1.275	1.280	1.285
2.9	1.290	1.295	1.300	1.305	1.310	1.314	1.319	1.324	1.329	1.333
3.0	1.338	1.343	1.348	1.352	1.357	1.362	1.367	1.371	1.376	1.381
3.1	1.385	1.390	1.395	1.400	1.405	1.409	1.414	1.418	1.423	1.427
3.2	1.432	1.436	1.441	1.446	1.450	1.455	1.459	1.464	1.468	1.473
3.3	1.477	1.482	1.486	1.491	1.496	1.500	1.504	1.508	1.513	1.517
3.4	1.521	1.525	1.529	1.533	1.537	1.542	1.546	1.550	1.554	1.558
3.5	1.562	1.566	1.570	1.575	1.579	1.583	1.587	1.591	1.595	1.600
3.6	1.604	1.608	1.612	1.617	1.621	1.625	1.629	1.633	1.637	1.642
3.7	1.646	1.650	1.654	1.658	1.662	1.666	1.671	1.675	1.679	1.683
3.8	1.687	1.691	1.695	1.700	1.704	1.708	1.712	1.715	1.719	1.723
3.9	1.727	1.731	1.735	1.739	1.742	1.746	1.750	1.754	1.758	1.762
4.0	1.765	1.769	1.773	1.777	1.781	1.785	1.789	1.792	1.796	1.800
4.1	1.804	1.808	1.811	1.815	1.819	1.822	1.826	1.830	1.833	1.837
4.2	1.841	1.845	1.848	1.852	1.856	1.859	1.863	1.867	1.870	1.874
4.3	1.878	1.882	1.885	1.889	1.893	1.896	1.900	1.904	1.907	1.911
4.4	1.914	1.918	1.921	1.925	1.929	1.932	1.936	1.939	1.943	1.946
4.5	1.950	1.954	1.957	1.961	1.964	1.968	1.971	1.975	1.979	1.982
4.6	1.986	1.989	1.993	1.996	2.000	2.003	2.007	2.010	2.013	2.017
4.7	2.020	2.023	2.027	2.030	2.033	2.037	2.040	2.043	2.047	2.050
4.8	2.053	2.057	2.060	2.063	2.067	2.070	2.073	2.077	2.080	2.083
4.9	2.087	2.090	2.093	2.097	2.100	2.103	2.107	2.110	2.113	2.116
5.0	2.119	2.122	2.125	2.129	2.132	2.135	2.139	2.142	2.145	2.148
5.1	2.151	2.154	2.158	2.160	2.164	2.167	2.170	2.173	2.176	2.180
5.2	2.183	2.186	2.190	2.192	2.195	2.197	2.200	2.203	2.206	2.209
5.3	2.212	2.215	2.218	2.221	2.224	2.227	2.230	2.233	2.236	2.240
5.4	2.243	2.246	2.249	2.252	2.255	2.258	2.261	2.264	2.267	2.270
5.5	2.273	2.276	2.279	2.282	2.285	2.288	2.291	2.294	2.297	2.300
5.6	2.303	2.306	2.309	2.312	2.315	2.318	2.320	2.324	2.326	2.329
5.7	2.332	2.335	2.338	2.341	2.344	2.347	2.350	2.353	2.355	2.358
5.8	2.361	2.364	2.367	2.370	2.373	2.376	2.379	2.382	2.384	2.387
5.9	2.390	2.393	2.396	2.400	2.403	2.405	2.408	2.411	2.414	2.417
6.0	2.419	2.422	2.425	2.428	2.431	2.433	2.436	2.439	2.442	2.444
6.1	2.447	2.450	2.453	2.456	2.458	2.461	2.464	2.467	2.470	2.472
6.2	2.475	2.478	2.481	2.483	2.486	2.489	2.492	2.494	2.497	2.500
6.3	2.503	2.505	2.508	2.511	2.513	2.516	2.518	2.521	2.524	2.526
6.4	2.529	2.532	2.534	2.537	2.540	2.542	2.545	2.547	2.550	2.553

data. A reasonably good approximation to degree of polymerization is obtained by multiplying intrinsic viscosity by 190, but values so obtained have relative not absolute significance.

12.4 The concept of fluidity is used by large numbers of operators in place of viscosity. These two quantities are reciprocals of one another. Intrinsic fluidity, the reciprocal of intrinsic viscosity, is useful because it is a nearly linear

function of yarn or fiber strength; its unit is grams per deciliter.

13. Precision and Bias

13.1 *Precision*—Based on interlaboratory testing, the reproducibility is within 3 % of each other.

13.2 *Bias*—No statement of bias can be made, as no suitable reference material exists for determining bias.

TABLE 3 Continued

η_{rel}	$[\eta]c$									
	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
6.5	2.555	2.558	2.561	2.563	2.566	2.568	2.571	2.574	2.576	2.579
6.6	2.561	2.564	2.567	2.590	2.592	2.595	2.597	2.600	2.603	2.605
6.7	2.608	2.610	2.613	2.615	2.618	2.620	2.623	2.625	2.627	2.630
6.8	2.633	2.635	2.637	2.640	2.643	2.645	2.648	2.650	2.653	2.655
6.9	2.658	2.660	2.663	2.665	2.668	2.670	2.673	2.675	2.678	2.680
7.0	2.683	2.685	2.687	2.690	2.693	2.695	2.698	2.700	2.702	2.705
7.1	2.707	2.710	2.712	2.714	2.717	2.719	2.721	2.724	2.726	2.729
7.2	2.731	2.733	2.736	2.738	2.740	2.743	2.745	2.748	2.750	2.752
7.3	2.755	2.757	2.760	2.762	2.764	2.767	2.769	2.771	2.774	2.776
7.4	2.779	2.781	2.783	2.786	2.788	2.790	2.793	2.795	2.798	2.800
7.5	2.802	2.805	2.807	2.809	2.812	2.814	2.816	2.819	2.821	2.823
7.6	2.826	2.828	2.830	2.833	2.835	2.837	2.840	2.842	2.844	2.847
7.7	2.849	2.851	2.854	2.856	2.858	2.860	2.863	2.865	2.868	2.870
7.8	2.873	2.875	2.877	2.879	2.881	2.884	2.887	2.889	2.891	2.893
7.9	2.895	2.898	2.900	2.902	2.905	2.907	2.909	2.911	2.913	2.915
8.0	2.918	2.920	2.922	2.924	2.926	2.928	2.931	2.933	2.935	2.937
8.1	2.939	2.942	2.944	2.946	2.948	2.950	2.952	2.955	2.957	2.959
8.2	2.961	2.963	2.966	2.968	2.970	2.972	2.974	2.976	2.979	2.981
8.3	2.983	2.985	2.987	2.990	2.992	2.994	2.996	2.998	3.000	3.002
8.4	3.004	3.006	3.008	3.010	3.012	3.015	3.017	3.019	3.021	3.023
8.5	3.025	3.027	3.029	3.031	3.033	3.035	3.037	3.040	3.042	3.044
8.6	3.046	3.048	3.050	3.052	3.054	3.056	3.058	3.060	3.062	3.064
8.7	3.067	3.069	3.071	3.073	3.075	3.077	3.079	3.081	3.083	3.085
8.8	3.087	3.089	3.092	3.094	3.096	3.098	3.100	3.102	3.104	3.106
8.9	3.108	3.110	3.112	3.114	3.116	3.118	3.120	3.122	3.124	3.126
9.0	3.128	3.130	3.132	3.134	3.136	3.138	3.140	3.142	3.144	3.146
9.1	3.148	3.150	3.152	3.154	3.156	3.158	3.160	3.162	3.164	3.166
9.2	3.168	3.170	3.172	3.174	3.176	3.178	3.180	3.182	3.184	3.186
9.3	3.188	3.190	3.192	3.194	3.196	3.198	3.200	3.202	3.204	3.206
9.4	3.208	3.210	3.212	3.214	3.215	3.217	3.219	3.221	3.223	3.225
9.5	3.227	3.229	3.231	3.233	3.235	3.237	3.239	3.241	3.242	3.244
9.6	3.246	3.248	3.250	3.252	3.254	3.256	3.258	3.260	3.262	3.264
9.7	3.266	3.268	3.269	3.271	3.273	3.275	3.277	3.279	3.281	3.283
9.8	3.285	3.287	3.289	3.291	3.293	3.295	3.297	3.298	3.300	3.302
9.9	3.304	3.305	3.307	3.309	3.311	3.313	3.316	3.318	3.320	3.321
10	3.32	3.34	3.36	3.37	3.39	3.41	3.43	3.45	3.46	3.48
11	3.50	3.52	3.53	3.55	3.56	3.58	3.60	3.61	3.63	3.64
12	3.66	3.68	3.69	3.71	3.72	3.74	3.76	3.77	3.79	3.80
13	3.80	3.83	3.85	3.86	3.88	3.89	3.90	3.92	3.93	3.95
14	3.96	3.97	3.99	4.00	4.02	4.03	4.04	4.06	4.07	4.09
15	4.10	4.11	4.13	4.14	4.15	4.17	4.18	4.19	4.20	4.22
16	4.23	4.24	4.25	4.27	4.28	4.29	4.30	4.31	4.33	4.34
17	4.35	4.36	4.37	4.38	4.39	4.41	4.42	4.43	4.44	4.45
18	4.46	4.47	4.48	4.49	4.50	4.52	4.53	4.54	4.55	4.56
19	4.57	4.58	4.59	4.60	4.61	4.62	4.63	4.64	4.65	4.66

^a Swedish Method CCA 27-57, Karin Wilson, *Svensk Papperstidning*, Vol 60, 1957, pp. 513 to 521.

^b Derived from the equation:

$$\eta_{rel} - 1 = \eta_{sp} = [\eta]c^{k'/(n)c}$$

where $k' = 0.30$.

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Standard Test Methods for Chemical Analysis of Basic Lead Silicochromate¹

This standard is issued under the fixed designation D 1844; the number immediately following the designation indicates the year of original adoption 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 April 1991.

1. Scope

1.1 These test methods cover the chemical analysis of the pigment commercially known as basic lead silicochromate and are applicable to pigment supplied by the manufacturer and to pigment, but not mixed pigments, separated from liquid coatings. The presence of basic lead silicochromate species shall be confirmed by X-ray diffraction analysis (see Specification D 1648²).

1.2 For liquid coatings the pigment must first be separated from the vehicle before conducting the analysis.

1.3 The analytical procedures appear in the following order:

	Sections
Lead oxide	6 to 14
Chromium trioxide	15 to 23
Silica	24 to 27
Moisture and other volatile matter	28
Coarse particles	29
Oil absorption	30
Mass color and tinting strength	31

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1193 Specification for Reagent Water⁴
- D 1648 Specification for Basic Lead Silicochromate Pigment²

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.

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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, Vols 06.03 and 11.01.

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

3. Significance and Use

3.1 These test methods may be used to confirm the stated lead oxide, chromium trioxide and silica content of basic lead silicochromate and is useful for quality control.

4. Purity of Reagents

4.1 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.

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

5. Preparation of Sample

5.1 Thoroughly mix liquid coatings and separate in accordance with Test Method D 2371 sufficient pigment to enable the required analyses to be carried out.

5.2 Thoroughly mix pigment supplied as such and grind separated pigment to a fine powder in a mortar and pestle before taking portions for analysis.

TOTAL LEAD AS LEAD OXIDE-GRAVIMETRIC METHOD

6. Apparatus

6.1 *Glass Filtering Crucible* (medium-porosity fritted disk), dried to constant weight before use.

6.2 *Platinum Dish*.

7. Reagents

7.1 *Acetic Acid (Glacial)*.

7.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

7.3 *Ammonium Acetate, Acid Solution*—To 300 mL of water add an equal volume of NH₄OH. Neutralize with

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ "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."

glacial acetic acid and add 20 mL in excess.

7.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

7.5 *Hydrofluoric Acid* (47 %)—Concentrated hydrofluoric acid (HF).

7.6 *Hydrogen Sulfide* (H₂S)—Handle and use H₂S in hood.

7.7 *Isopropyl Alcohol* (50 and 98 %).

7.8 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃) (see 7.4.1).

7.9 *Potassium Dichromate Solution* (saturated)—Prepare a saturated solution of potassium dichromate (K₂Cr₂O₇) in water.

7.10 *Sulfuric Acid* (1+1)—Carefully mix 1 volume of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) with 1 volume of water (see 7.4.1).

8. Procedure

8.1 Weigh accurately (to 0.1 mg) 1 g of the sample into a platinum dish. Add 5 mL of HNO₃ and 10 mL of HF. Cautiously evaporate to dryness on a steam bath. Repeat the addition of HNO₃ and HF and again cautiously evaporate to dryness. Wash the sides of the dish with a little water and evaporate to dryness. Wet the residue with 5 mL of HNO₃, warm gently, and transfer the residue to a 400-mL beaker using a policeman. Neutralize with NH₄OH, and then make the solution just acid with HCl, adding 5 mL in excess. Dilute to 200 mL and heat to just below the boiling point until solution is complete.

8.2 Pass H₂S through the solution for about 20 min. Filter, using paper pulp. Wash the precipitate five to six times with water just acid with HCl and saturated with H₂S. Transfer the paper and precipitate to the original beaker, add 25 mL of HNO₃, boil until the residue is white, then add 10 mL of H₂SO₄ (1+1). Destroy the organic material by evaporating the solution to dense white fumes, making further additions of HNO₃ until there is no charring.

8.3 Cool the solution, add 10 mL of water, and evaporate to fumes. Repeat the addition of water and the evaporation. Cool the solution, add 40 mL of water, and bring the solution to boiling. Cool the solution and add 50 mL of isopropyl alcohol (98 %), stir, and allow the solution to stand cold for at least 4 h. Filter, using paper pulp, and wash once with cold isopropyl alcohol (50 %) containing 10 mL of H₂SO₄ (sp gr 1.84)/100 mL.

8.4 Transfer the precipitate to the original beaker with 150 mL of water, add 50 mL of the ammonium acetate solution, and boil the solution until the lead sulfate dissolves. Filter while hot through the original paper and wash well (6 to 8 washes) with hot water. To the filtrate add 5 mL of glacial acetic acid, heat the solution to boiling, and add 20 mL of saturated K₂Cr₂O₇ solution. Boil the solution until the precipitate turns orange, and allow to stand on a warm plate for at least 2 h. Filter through a glass filtering crucible (medium-porosity fritted disk), wash three times with hot water, and finally once with alcohol. Dry in an oven at 105°C for 2 h, cool, and weigh.

9. Calculation

9.1 Calculate the percent of total lead as lead oxide (PbO) as follows:

$$\text{PbO, \%} = (P \times 69.06)/S$$

where:

P = lead chromate (PbCrO₄) precipitate, g,
S = sample used, g, and

$$\frac{\text{molecular weight (PbO)}}{\text{molecular weight (PbCrO}_4\text{)}} =$$

$$\frac{223.21}{323.21} = 0.6906 \times 100 \text{ (for percent)} = 69.06$$

69.06 represents the gravimetric factor to convert grams of PbCrO₄ to grams of PbO.

This gravimetric factor has led to high results due to the presence of mixed lead chromates and an empirical factor can be used to compensate:

$$\text{Empirical factor} = 69.06 \times 0.9944 = 68.67$$

ALTERNATIVE METHOD FOR TOTAL LEAD AS LEAD OXIDE—TITRIMETRIC METHOD

10. Apparatus

10.1 *Platinum Dish*.

10.2 *Filter Paper*, ashless, medium texture, or paper pulp.

11. Reagents

11.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

11.2 *Glacial Acetic Acid* (min 99.7 %)—Concentrated glacial acetic acid (CH₃COOH).

11.3 *Acid Ammonium Acetate Buffer*—Mix 400 mL of distilled water and 400 mL of ammonium hydroxide (sp gr 0.90). Add 375 mL of reagent grade glacial acetic acid slowly while stirring.

11.4 *Cupric Sulfate Solution* (0.1 M)—Dissolve 25 g of CuSO₄·5H₂O in distilled water and dilute to 1 L.

11.5 *Disodium Ethylenediaminetetracetate Dihydrate* (0.05 M) (EDTA solution)⁷—Dissolve 18.6 g of the salt in distilled water and dilute to 1 L. Standardize the solution as follows: Transfer 25 mL of lead standard (11.14) to a 400-mL beaker. Add concentrated ammonium hydroxide (11.1) dropwise until a permanent precipitate just forms. Add 25 mL of acid ammonium acetate (11.3), dilute to 200 mL, heat to boiling, add 4 drops of copper EDTA (11.7) and 12 drops of PAN (11.13) and titrate with the EDTA to a clear yellow.

$$1 \text{ mL Na}_2 \text{ EDTA} = 0.2790/V \text{ g PbO}$$

where:

V = EDTA required for titration, mL

g = lead oxide, g, and

0.05 M × 0.22321 = 0.01116 g PbO in 1 mL of lead standard (10.14) (25 mL) (0.05 M) (0.22321) = 0.2790 g PbO in 25 mL of lead standard (10.14).

11.6 *Murexide Indicator Tablets*—Ammonium salt of purpuric acid.

11.7 *Copper-EDTA Solution*—Mix equivalent amounts of cupric sulfate solution (11.4) and EDTA solution (11.5) and store in a dropping bottle. The cupric sulfate EDTA equivalence may be determined as follows: Pipet 10 mL of cupric

⁷ The standardized solution may also be purchased from Corco Chemical Corp. Catalog No.—Special.

into a beaker, add concentrated ammonium hydroxide dropwise until the precipitate which forms just dissolves. Dilute to 200 mL with water, add two Murexide indicator tablets, and titrate immediately with EDTA to a change from yellow to purple.

11.8 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl) (see 7.4.1).

11.9 *Hydrofluoric Acid* (47 %)—Concentrated hydrofluoric acid (HF).

11.10 *Hydrogen Sulfide* (H₂S)—Cylinder. Handle and use in hood.

11.11 *Hydrogen Sulfide Wash Solution*—Add 10 mL of (sp gr 1.19) to 1 L of water and saturate with H₂S.

11.12 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

11.13 *Pan Indicator* (1-(2-pyridylazo)-2-naphthol)—Dissolve 0.1 g in 100 mL of ethanol.

11.14 *Primary Standard* (0.05 M lead nitrate)—Dissolve 5615 g of reagent grade lead nitrate in distilled water and dilute to 1 L.

$$1 \text{ mL} = 0.01116 \text{ g PbO}$$

11.15 *Sulfuric Acid* (1+1)—Carefully mix 1 volume of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) with 1 volume of water.

12. Procedure

12.1 Dissolve 1 g of pigment in accordance with 8.1 and 8.2.

12.2 Cool, so that the addition of 25 mL of water does not cause excessive splattering of the sulfuric acid; the solution at this point should be water white. Add ammonium hydroxide (sp gr 0.90) until the pH of the solution is 5 to 5.5 (as indicated by pH paper). Add 50 mL of acid ammonium acetate (11.3), boil 5 min, dilute to 200 mL with water, heat to boiling, add 4 drops of copper-EDTA (11.7) and 12 drops of PAN (11.13) and titrate while hot with standard EDTA to a clear yellow.

13. Calculation

13.1 Calculate the percent lead oxide as follows:

$$\text{PbO, \%} = \frac{V \times L \times 100}{S}$$

where:

V = EDTA required for titration, mL,

L = lead equivalent of EDTA solution, and

S = specimen weight, g.

14. Precision and Bias

14.1 On the basis of an interlaboratory study of the method in which operators in three laboratories analyzed two paints containing basic lead silicochromate with iron oxide the following criteria should be used for judging the acceptability of the results at the 95 % confidence level:

14.1.1 *Repeatability*—Two results, each the mean of duplicate determinations obtained by the same operator should be considered suspect if they differ by more than the following:

Percent Absolute PbO	
28 %	0.20
45 %	0.30

14.1.2 *Reproducibility*—A realistic range could not be established for results between laboratories because of the limited number of participating laboratories.

14.1.3 *Bias*—A bias statement could not be established because of the limited number of participating laboratories.

CHROMIUM TRIOXIDE

15. Interference

15.1 Soluble Fe⁺³ will cause high results. See Section 20.

16. Reagents

16.1 *Potassium Dichromate, Standard Solution* (0.1 N)—Weigh 4.904 g of dried potassium dichromate (K₂Cr₂O₇), dissolve it in water, and dilute to 1 L with water in a volumetric flask.

16.2 *Potassium Iodide Solution* (150 g/L)—Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 L.

16.3 *Hydrochloric Acid Mixture*—Saturate water with NaCl (about 350 g/L). To each litre of this solution add 150 mL of water and 100 mL of concentrated HCl (sp gr 1.19).

16.4 *Sodium Thiosulfate Solution* (0.1 N)—Dissolve 24.8 g of reagent grade Na₂S₂O₃·5H₂O in recently boiled water and dilute to 1 L with additional recently boiled water. To standardize, pipet 25 mL of the standard potassium dichromate solution (exactly 0.1000 N) into a 250-mL Erlenmeyer flask. Add 100 mL of water, 15 mL of potassium iodide solution, and 15 mL of concentrated hydrochloric acid (sp gr 1.19). Titrate the liberated iodine with sodium thiosulfate solution until the reddish-brown color becomes quite faint. Add 5 mL of starch solution and continue the titration dropwise until the blue color changes to a pale green. Calculate the normality of the sodium thiosulfate solution as follows:

$$N = \frac{2.5000}{M}$$

where:

N = normality, and

M = sodium thiosulfate solution, mL.

16.5 *Starch Indicator Solution*—Make a homogeneous paste of 10 g of soluble starch in cold water. Add to this 1 L of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/L) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to 10°C (40 to 50°F). Prepare fresh indicator when the end point of the titration from blue to colorless or blue to light green fails to be sharp.

17. Procedure

17.1 Weigh to 0.1 mg about 1 g of sample into a 500-mL Erlenmeyer flask. Add 100 mL of NaCl-HCl solution and dissolve the sample without heat, keeping the solution cold. (Silica will not dissolve, but it does not interfere with the analysis.) Add 10 mL of KI solution and titrate with 0.1 N sodium thiosulfate solution until the reddish-brown color is almost gone. Add 5 mL of starch solution and titrate slowly, dropwise, until the blue color changes to a light green.

18. Calculation

18.1 Calculate the percent of chromium trioxide (CrO₃) as follows:

$$\text{CrO}_3, \% = [(V_1 N \times 0.03334)/S_2] \times 100$$

where:

V_1 = sodium thiosulfate solution required for titration of the specimen, mL

N = normality of the sodium thiosulfate solution,

S_1 = sample used, g, and

$$\begin{aligned} \text{CrO}_3 \rightarrow \text{Cr}^{+3} &= \frac{\text{molecular weight (CrO}_3\text{)}}{1000 \times 3 \text{ (valence)}} \\ &= \frac{99.99}{1000 \times 3} = 0.03334 \end{aligned}$$

ALTERNATIVE METHOD FOR CHROMIUM TRIOXIDE

19. Scope

19.1 This test method determines total chromium as chromium trioxide. Iron oxide interference is masked by the use of EDTA. This procedure is not applicable when chromium oxide green (Cr_2O_3) is present. This test method is limited to basic lead silicochromate pigments with or without iron oxide.

19.2 The perchloric acid is employed as an oxidizing agent to ensure complete oxidation of any reduced chromium. When perchloric acid is used, concentrated nitric acid must also be used. The use of a perchloric acid hood is optional since the specimen is covered with a watch glass and is being oxidized, not wet ashed.

20. Reagents

20.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

20.2 *Disodium Ethylenediaminetetracetate Dihydrate* (0.05 M) (EDTA)—Dissolve 18.6 g of the reagent grade salt in distilled water and dilute to 1 L. See 11.5 for standardization.

20.3 *Hydrochloric Acid Mixture*—See 16.3.

20.4 *Hydrofluoric Acid* (47 %)—Concentrated hydrofluoric acid (HF).

20.5 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

20.6 *Perchloric Acid* (sp gr 1.67)—Concentrated perchloric acid (HClO_4).

20.7 *Potassium Dichromate, Standard Solution* (0.1 N)—See 16.1.

20.8 *Potassium Iodide Solution* (150 g/Litre)—See 16.2.

20.9 *Sodium Thiosulfate Solution* (0.1 N)—See 16.4.

20.10 *Starch Indicator Solution*—See 16.5.

21. Procedure

21.1 Transfer 0.5 g of pigment to a 500-mL high-silica Erlenmeyer flask.⁸ Add 10 mL of nitric acid (sp gr 1.42), and 10 mL of perchloric acid (sp gr 1.67) and cover with a watch glass. Evaporate to fumes (flask will fill with white vapor) of perchloric acid, cool, add 15 mL of water, and evaporate to fumes. Continue fuming for 15–20 min (the specimen should be red-orange color). Cool immediately by placing the flask in a cold water bath.

21.2 Wash down the cover glass and sides of the flask with

water. Add 50 mL of HCl mixture and 50 mL of 0.05 M EDTA solution. Adjust to pH 8 (determined with pH paper) with ammonium hydroxide (sp gr 0.90) and add 50 mL of HCl mixture (16.3). Cool the flask to 20 to 25°C, add 10 mL of potassium iodide solution, 10 mL of starch solution, and titrate immediately with standard sodium thiosulfate solution to a sharp change in the starch indicator. (The final solution color may be lavender or green depending on the composition of the sample).

21.3 Add 2 to 3 mL of HF, (20.4) stir for ½ min and if the indicator changes color titrate to the endpoint.

22. Calculation

22.1 Calculate the percent chromium trioxide (CrO_3) as follows:

$$\text{CrO}_3, \% = [(V_2 N_1 \times 0.03334)/S_3] \times 100$$

where:

V_2 = sodium thiosulfate solution required for titration of the specimen, g,

N_1 = normality of the sodium thiosulfate solution, and

S_3 = sample used, g.

23. Precision and Bias

23.1 On the basis of an interlaboratory study of this test method in which operators in three laboratories analyzed two paints containing basic lead silicochromate with iron oxide the following criteria should be used for judging the acceptability of the results at the 95 % confidence level.

23.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator should be considered suspect if they differ by more than the following:

Percent Absolute CrO_3	
3 % CrO_3	0.15
5 % CrO_3	0.40

23.1.2 *Reproducibility*—A realistic range could not be established for results between laboratories because of the limited number of participating laboratories.

23.1.3 *Bias*—A bias statement could not be established because of the limited number of participating laboratories.

SILICA

24. Apparatus

24.1 *Platinum Crucible*.

25. Reagents

25.1 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

25.2 *Hydrofluoric Acid* (47 %)—Concentrated hydrofluoric acid (HF).

25.3 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

25.4 *Nitric Acid* (1+19)—Mix 1 volume of concentrated HNO_3 (sp gr 1.42) with 19 volumes of water.

25.5 *Perchloric Acid* (sp gr 1.67)—Concentrated perchloric acid (HClO_4).

25.6 *Sulfuric Acid* (1+4)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84)⁶ with 4 volumes of water.

⁸ Vycor has been found suitable for this purpose.

26. Procedure

26.1 Weigh 0.5 g of pigment and transfer to a 400-mL beaker. Add 10 mL each of HCl (sp gr 1.19) and HNO₃ (sp gr 1.42). Warm slightly, if necessary, to dissolve the chromate. Add 50 mL of HClO₄ and fume for 15 to 20 min. Wash the sides of the beaker down with 30 to 40 mL of water, evaporate, and fume for 10 min. Cool and dilute to 200 mL with water. Filter through a double acid-washed quantitative paper and wash eight times with hot water, once with HNO₃ (1+19) and finally again with hot water.

26.2 Dry and ignite the paper and precipitate in a platinum crucible. Cool in a desiccator and weigh the crucible and contents. To the crucible add 2 drops of H₂SO₄ (1+4) and 15 mL of HF. Evaporate cautiously on a hot plate and ignite at 1000°C for approximately 5 min. Cool in a desiccator and weigh to constant weight.

27. Calculation

27.1 Calculate the percent of silica (SiO₂) as follows:

$$\text{SiO}_2, \% = [(W_1 - W_2)/S_4] \times 100$$

where:

W_1 = weight of crucible and contents before HF treatment, g,

W_2 = weight of crucible and contents after HF treatment, g, and

S_4 = weight of sample used, g.

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MOISTURE AND OTHER VOLATILE MATTER

28. Procedure

28.1 Determine moisture and other volatile matter in accordance with Method A of Test Method D 280.

COARSE PARTICLES

29. Procedure

29.1 Determine the percent coarse particles in the pigment as received in accordance with Test Methods D 185.

OIL ABSORPTION

30. Procedure

30.1 Determine the oil absorption of the pigment in accordance with Test Method D 281.

MASS COLOR AND TINTING STRENGTH

31. Procedure

31.1 Determine the mass color and tinting strength in accordance with Test Method D 387.

32. Precision and Bias

32.1 Precision and bias have not been determined.

33. Keywords

33.1 basic lead silicochromate; chromium in basic lead silicochromate; lead in basic lead silicochromate; lead oxide; pigment; silica in basic lead silicochromate



Standard Test Methods for Chemical Analysis of Strontium Chromate Pigment¹

This standard is issued under the fixed designation D 1845; the number immediately following the designation indicates the year of original adoption 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—Keywords were added editorially in January 1991.

1. Scope

1.1 These test methods cover the chemical analysis of strontium chromate pigment.

1.2 The analytical procedures appear in the following order:

	Sections
Strontium by the Strontium Sulfate Method	7 to 10
Chromium by the Thiosulfate Method	11 to 14
Chloride Content	15
Sulfate Content	16
Moisture and Other Volatile Matter	17
Coarse Particles	18
Mass Color and Tinting Strength	19

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³

D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller³

D 444 Test Methods for Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow)³

D 1193 Specification for Reagent Water⁴

3. Significance and Use

3.1 These test methods may be used to confirm the stated strontium oxide and chromium oxide content of strontium chromate.

4. Purity of Reagents

4.1 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.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 Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

5. Apparatus

5.1 *Gooch crucible.*

5.2 *Electric Furnace, capable of 800°C.*

6. Preparation of Sample

6.1 Mix the sample thoroughly. Take a sufficient quantity for chemical analysis and pass it through a No. 325 (4- μ m) sieve.

STRONTIUM BY THE STRONTIUM SULFATE METHOD

7. Reagents

7.1 *Acetic Acid (glacial).*

7.2 *Ammonium Hydroxide (1+3)*—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH , sp gr 0.90) with 3 volumes of water.

7.3 *Ethyl Alcohol*, conforming to Formula No. 2B or No. 30 of the U.S. Bureau of Alcohol, Tobacco, and Firearms.

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

7.5 *Sulfuric Acid (1+19)*—Mix 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) with 19 volumes of water.

8. Procedure

8.1 Weigh to 0.1 mg about 0.5 g of the sample into a 400-mL beaker. Add 40 mL of HCl (1+1) and heat the solution to dissolve the pigment. Add 40 mL of ethyl alcohol until the chromium is reduced, as this is indicated by a dark coloration of the solution. Add to the solution 100 mL of water and sufficient NH_4OH (1+3) to form a slight persistent precipitate.

8.2 Add HCl dropwise until the precipitate just redissolves (Note 1). Heat the solution to just under boiling and add 20 mL of H_2SO_4 (1+19). Add 100 mL of ethyl alcohol and

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and are under the jurisdiction of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

³ *Annual Book of ASTM Standards*, Vol 06.02.

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."

allow the precipitate to settle for several hours or overnight.

NOTE 1—For a cleaner precipitate, add HCl until a pH of 2.0 is reached, instead of adding HCl dropwise until the precipitate is just dissolved. Then add 25 mL of acetic acid to minimize the tendency of the sulfate to form a complex with trivalent chromium. Finally, heat the solution, and add H_2SO_4 (1+19).

8.3 Filter the precipitate through a Gooch crucible that has been previously dried to constant weight. Wash the precipitate several times with a solution of equal volumes of ethyl alcohol, water, and H_2SO_4 (1+19). Dry the crucible in an oven and then ignite for $\frac{1}{2}$ h at 800°C or until constant weight is attained (weight loss less than 0.1 mg). Cool and weigh the crucible.

9. Calculation

9.1 Calculate the percent of strontium oxide (SrO) as follows:

$$\text{SrO, \%} = [(P \times 0.56416)/S] \times 100$$

where:

P = SrSO_4 precipitate, g, and

S = pigment specimen, g.

$$0.56416 = \frac{\text{molecular weight of SrO}}{\text{molecular weight of SrSO}_4} = \frac{103.63}{163.70}$$

10. Precision

10.1 *Within Laboratory*—The usual difference between duplicate runs performed by the same analyst is approximately 0.2 % of the SrO content of the pigment tested.

10.2 *Between Laboratories*—The average difference between two determinations performed by different analysts in different laboratories is approximately 0.5 % of the SrO content of the pigment tested.

CHROMIUM BY THE THIOSULFATE METHOD

11. Reagents

11.1 *Hydrochloric Acid* (1+5)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 5 volumes of water.

11.2 *Potassium Iodide Solution* (150 g per L)—Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 L.

11.3 *Sodium Thiosulfate Standard Solution* (0.1 N)—Dissolve 24.8 g of reagent grade $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in recently boiled water and dilute to 1 L with additional recently boiled water. To standardize, pipet 25 mL of the standard potassium dichromate solution (exactly 0.1000 N) into a 250-mL Erlenmeyer flask. Add 100 mL of water, 15 mL of potassium iodide solution, and 15 mL of concentrated hydrochloric acid (sp gr 1.19). Titrate the liberated iodine with sodium thiosulfate solution until the reddish-brown color becomes quite faint. Add 5 mL of starch solution and continue the titration dropwise until the blue color changes to a pale green. Calculate the normality of the sodium thiosulfate solution as follows:

$$N = \frac{2.5000}{M}$$

where:

N = normality, and

M = sodium thiosulfate solution, g.

11.4 *Starch Indicator Solution*—Make a homogenous paste of 10 g of soluble starch in cold water. Add to this 1 L of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/L) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to 10°C (40 to 50°F). Prepare fresh indicator when the end point of the titration from blue to colorless or blue to light green fails to be sharp.

12. Procedure

12.1 Weigh accurately approximately 0.2 g of the sample into a 250-mL iodine flask. Add 50 mL of water and 50 mL of HCl (1+5); swirl the flask to complete solution. Add 20 mL of KI solution and allow the solution to stand for several minutes.

12.2 Titrate the liberated iodine with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution until the reddish brown iodine color becomes faint. Add 1 mL of starch solution and continue the titration cautiously to the end point which is easily discernible when the color changes from blue to a light green with no blue tinge.

13. Calculation

13.1 Calculate the percent of chromium as chromic oxide (CrO_3) as follows:

$$\text{CrO}_3, \% = [(VN \times 0.03334)/S] \times 100$$

where:

V = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of specimen, mL,

N = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, and

S = sample used, g.

14. Precision and Bias

14.1 *Within Laboratories*—The usual difference between duplicate runs performed by the same analyst is approximately 0.3 % of the CrO_3 content of the pigment tested.

14.2 *Between Laboratories*—The average difference between two determinations performed by different analysts in different laboratories is approximately 0.5 % of the CrO_3 content of the pigment tested.

$$0.0334 = \frac{\text{molecular weight CrO}_3}{1000 \times 3 (\text{volume})} = \frac{99.99}{1000 \times 3}$$

= milliequivalent weight

CHLORIDE CONTENT

15. Procedure

15.1 Determine the chloride content in accordance with Test Methods D 444.

SULFATE CONTENT

16. Procedure

16.1 Determine the sulfate content in accordance with the Test Methods D 444.

MOISTURE AND OTHER VOLATILE MATTER

MASS COLOR AND TINTING STRENGTH

17. Procedure

17.1 Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

COARSE PARTICLES

18. Procedure

18.1 Determine the percentage of coarse particles in the pigment as received in accordance with Test Methods D 185.

19. Procedure

19.1 Determine mass color and tinting strength in accordance with Test Method D 387.

20. Keywords

20.1 chromium, thiosulfate method; strontium chromate pigment

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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 Total Chlorine Content of Epoxy Resins¹

This standard is issued under the fixed designation D 1847; the number immediately following the designation indicates the year of original adoption 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 total chlorine content, in concentrations below 1 weight percent, of epoxy resins. Both organic and inorganic chlorine compounds contained in the resin are determined. Epoxy resin is defined as the reaction product of a chlorohydrin and a di- or polyfunctional phenolic compound.

1.2 Two alternative test methods for determining chloride ion concentration resulting from combustion of the sample are included as follows:

	Sections
Test Method A—Potentiometric Titration	6 to 10
Test Method B—Gravimetric Determination	11 to 15

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. Specific hazards are given in Section 12.*

2. Referenced Document

- 2.1 *ASTM Standard:*
D 1193 Specification for Reagent Water²

3. Summary of Test Methods

3.1 The resin is oxidized by combustion in a bomb containing oxygen under pressure. The chlorine compounds thus liberated are absorbed in a sodium carbonate solution. In Test Method A the amount of chlorine present is determined by potentiometric titration with standard silver nitrate solution. In Test Method B the amount of chlorine present is determined gravimetrically by precipitation as silver chloride.

4. Significance and Use

4.1 The total chlorine content of epoxy resins is an important variable in determining reactivity of epoxy resins and performance of coatings prepared from them. These test methods may be used to determine the total chlorine content of manufactured epoxy resins to confirm specification limits.

5. Purity of Reagents

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

TEST METHOD A—POTENTIOMETRIC TITRATION

6. Apparatus

6.1 *Combustion Apparatus*—An oxygen bomb⁴ apparatus for combustion of the sample.

6.2 *Titration Apparatus*⁵—A suitable pH meter equipped with a glass-silver electrode system and titration stand, stirrer, beakers, and 10-mL buret.

7. Reagents

7.1 *Methyl Red Indicator* (0.2 % alcohol solution)—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol or isopropanol.

7.2 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

7.3 *Oxygen*, free of combustible materials and halogen compounds, available at a pressure of 40 atm.

7.4 *Silver Nitrate, Standard Solution* (0.01 N)—Dissolve in water 1.70 ± 0.01 g of crystalline silver nitrate (AgNO₃) which previously has been pulverized, dried at 150°C for 1 h, and stored in a glass-stoppered bottle, and dilute to 1 L. Standardize against an accurately weighed amount of sodium chloride (NaCl) dissolved in 75 to 100 mL of water.

7.5 *Sodium Carbonate Solution* (20 g/L)—Dissolve 20 g of sodium carbonate (Na₂CO₃) in water and dilute to 1 L.

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 The Parr double-valve, self-sealing oxygen bomb equipped with a safety-relief-type oxygen filling connection, ignition circuit, fuse wire, sample cups, and water bath, available from Parr Instrument Co., 209 53rd St., Moline, IL 61265, has been found satisfactory for this purpose.

⁵ The Dual Titrimeter manufactured by the Precision Scientific Co., 3737 W. Cortland St., Chicago, IL, and the Beckman pH meter (Model M or G) manufactured by Beckman Instruments, Inc., 2500-T Harbor Blvd., Fullerton, CA, have been found satisfactory for this purpose.

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 D 01.33 on Resins and Varnishes, Including Shellac.

Current edition approved Nov. 27, 1987. Published January 1988. Originally published as D 1847 - 61 T. Last previous edition D 1847 - 73 (1980) ^{ϵ} .

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

8. Procedure

8.1 Combustion of Specimen:

8.1.1 Place 25 mL of Na_2CO_3 solution in the bottom of the bomb. Incline the bomb and rotate it in such a manner that the interior surface is moistened by the solution.

8.1.2 Weigh to 1 mg 0.8 to 1.2 g of sample into the metal sample cup. Grind solid resins to pass a No. 60 (250- μm) sieve and moisten with a few drops of acetone before combustion.

8.1.3 Insert the cup containing the sample in the loop electrode and attach the fuse wire so that it hangs slightly above the specimen. Assemble the bomb and tighten the cover securely; however, do not use auxiliary tools to tighten the cover.

8.1.4 Attach the filling connection to the oxygen-filling valve and admit oxygen slowly (to avoid blowing the sample from the cup) until a pressure of 35 atm is indicated on the gage; then close the control valve. Open the relief valve to reduce the pressure in the tube and in the connection to atmospheric pressure. Detach the filling connection from the bomb and replace with the bomb thumb nut. Tighten the thumb nut with finger pressure.

8.1.5 Immerse the bomb in the water bath, then connect the terminals to the electrical circuit. Ignite the charge within the bomb by closing the switch for not more than 5 s, or until the pilot light goes out. Allow the bomb to stand in the water bath for 10 min while cooling.

8.1.6 Remove the bomb from the water bath, and invert and rotate it so that maximum washing of the inside surface is achieved. Release the pressure at a uniform rate over a period of not less than 1 min.

8.2 Titration:

8.2.1 Transfer the absorbing solution from the combustion of the sample to a 250-mL tall-form titration beaker. Wash all the inside surfaces of the bomb with a fine stream of water and transfer the washings to the beaker. Adjust the volume of the solution to 150 to 200 mL with water. Add three drops of methyl red indicator solution and neutralize with HNO_3 ; then add 6 drops in excess.

8.2.2 Place the beaker on the titration stand and adjust its position so that the electrodes are about half immersed. Fill the buret with 0.01 N AgNO_3 solution, and place the buret in position on the titration assembly so that the tip extends approximately 25 mm below the surface of the liquid in the beaker. Adjust the speed of the stirrer to give vigorous stirring without splattering. Record the initial buret and meter (cell potential) readings.

8.2.3 Add small portions of 0.01 N AgNO_3 solution and, after waiting until a constant potential has been established, record the buret and meter readings. In regions between inflections where the potential change is small for each increment of AgNO_3 solution, add volumes as large as 0.5 mL. When the rate of change of cell potential becomes greater than 5 mV per 0.1 mL, use 0.1-mL increments of 0.01 N AgNO_3 solution.

8.2.4 Continue the titration until the rate of change of cell potential is less than 2 mV per 0.1 mL of AgNO_3 solution. Remove the titrated solution, rinse the electrodes well with water, wipe with a dry cloth, and burnish the silver electrode lightly with a fine emery cloth. Between determinations, immerse the electrodes in water.

8.2.5 Plot the cumulative volumes of 0.1 N AgNO_3 solution added against the corresponding cell potentials. Select as the end point the midway point on the steepest portion of the inflection curve.

8.2.6 Make a blank determination in accordance with 8.2.1 through 8.2.4, but omit the specimen. This procedure will give a measure of the chlorine in the reagents used.

9. Calculation

9.1 Calculate the chlorine content of the sample as follows:

$$\text{Chlorine, \%} = [(V - B)N \times 0.0355/S] \times 100$$

where:

V = AgNO_3 solution required for titration of the specimen, mL

B = AgNO_3 solution required for titration of the blank, mL

N = normality of the AgNO_3 solution, and

S = specimen weight used, g.

10. Precision

10.1 The following criteria should be used for judging the acceptability of 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 0.02 weight %.

10.1.2 *Reproducibility*—The results, each the mean of two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.05 weight %.

TEST METHOD B—GRAVIMETRIC DETERMINATION

11. Apparatus

11.1 *Combustion Apparatus*—See 6.1.

11.2 *Fritted-Glass*, medium porosity.

12. Reagents

12.1 *Warning*—*Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

12.2 *Caution*—*Nitric Acid* (1+99)—Mix 1 volume of HNO_3 (sp-gr 1.42) with 99 volumes of water.

12.3 *Oxygen*—See 7.3.

12.4 *Potassium Iodide Solution* (300-g/litre)—Dissolve 300 g of potassium iodide (KI) in water and dilute to 1 L.

12.5 *Silver Nitrate Solution* (100 g/L)—Dissolve 100 g of silver nitrate (AgNO_3) in water and dilute to 1 L.

12.6 *Silver Nitrate, Standard Solution* (0.01 N)—See 7.4.

12.7 *Sodium Carbonate Solution* (20 g/L)—See 7.5.

13. Procedure

13.1 Oxidize the specimen by combustion in accordance with 8.1.1 to 8.1.6.

13.2 Transfer the absorbing solution from the combustion of the specimen to a 250-mL beaker. Wash all the inside surfaces of the bomb with a fine stream of water and transfer the washings to the beaker. Add 1 mL of HNO_3 (sp gr 1.42) to the solution; then, with stirring, add 1 mL of AgNO_3 solution (100 g/L). Heat the solution to boiling as rapidly as possible. Allow the mixture to cool in a dark cabinet for at least 1 h.

13.3 Filter the precipitate by suction on a clean, fritted-glass filter funnel. Wash the silver chloride (AgCl) precipitate thoroughly with HNO_3 (1+99). Dry the filter and precipitate at 110°C for 1 h. Cool in a desiccator and weigh accurately to 0.1 mg.

13.4 Dissolve the AgCl from the filter, using KI solution. Wash the filter thoroughly with HNO_3 (1+99). Dry the filter at 110°C for 1 h, cool in a desiccator, and reweigh. The weight of AgCl obtained from the sample is the difference between the weight of the filter funnel containing the precipitate and the weight of the filter funnel obtained at this time.

13.5 Make a blank determination in accordance with 13.2 and 13.4, but omit the specimen. This procedure will give a measure of the chlorine in the reagents used.

14. Calculation

14.1 Calculate the chlorine content of the specimen as

follows:

$$\text{Chlorine, \%} = [(P - B) \times 0.2474/S] \times 100$$

where:

P = AgCl precipitate from the specimen, g,

B = AgCl found in the blank, g, and

S = specimen used, g.

15. Precision

15.1 The following criteria should be used for judging the acceptability of results at the 95% confidence level.

15.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.05 weight percent.

15.1.2 *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 0.05 weight percent.

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 Chromatographic Analysis of Chemically Refined Cellulose¹

This standard is issued under the fixed designation D 1915; the number immediately following the designation indicates the year of original adoption 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—Editorial changes were made throughout, including the title, in October 1989.

1. Scope

1.1 This test method covers the determination of the composition of chemically refined cellulose by chromatographic analysis. This test method is suitable for rapid and routine testing of large numbers of samples with high accuracy and precision.

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 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 Quantitative paper chromatographic analysis of polysaccharides by the chemical method involves the following operations: (1) total hydrolysis, (2) neutralization, (3) concentration, (4) chromatographic separation, (5) elution, (6) analysis of separated sugars, and (7) calibration and calculation.

4. Significance and Use

4.1 While this test method involves a chemical determination of separated sugars, it is recognized that direct photometry of the chromatogram can also be capable of good precision. All of the steps involved, up to the actual determination, are the same for both techniques. Direct chemical measurement is of more general applicability in a chromatographic laboratory, however, since it involves no special instrumentation and it is adaptable to the determination of a variety of materials.

4.2 The carbohydrate composition of a cellulosic material may be expressed either on the basis of the total initial sample or on the basis of the carbohydrate portion of the sample. The former establishes composition on an absolute basis. It requires precise information concerning all components present in the starting material, or lacking this, it

requires quantitative handling of the carbohydrate portion throughout the entire sequence of chromatographic operations. In the case of purified celluloses, where the carbohydrate fraction comprises all but a minor part of a given sample, it is convenient to express composition on the basis of the ratios of the carbohydrate constituents; that is, the quantity of each isolated sugar is expressed as a percent of the combined sugar content of the sample. Quantitative handling is thus not required until application of the hydrolyzate to the paper. Proper correction for conversion of polysaccharide to monosaccharide and for decomposition are of course required in any method of analysis. These may conveniently be accounted for by calibration.

5. Purity of Reagents

5.1 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.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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

6. Sampling

6.1 Take samples from not less than 10 % of each lot in the shipment. Then composite these samples to represent each lot or batch.

TOTAL HYDROLYSIS

7. Summary of Procedure

7.1 The total hydrolysis of a cellulose requires a primary hydrolysis with strong mineral acid followed by a secondary hydrolysis in dilute acid. The primary hydrolysis results in the formation of a mixture of oligosaccharides. It is the function of the secondary hydrolysis to complete the conversion to monomeric sugars (1).⁴

¹ This test method is under the jurisdiction of ASTM Committee D-23 on Cellulose and Cellulose Derivatives and is the direct responsibility of Subcommittee D23.20 on Cellulose.

Current edition approved Nov. 19, 1963. Published January 1963. Originally published as D 1915 - 61. Last previous edition D 1915 - 61T.

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 The boldface numbers in parentheses refer to the list of references at the end of this test method.

Apparatus

1. *Shell Vials*, 25 by 90-mm.
2. *Stirring Rods*, 5 by 150-mm.
3. *Water Bath*, controlled at $30 \pm 0.1^\circ\text{C}$.
4. *Pipet*, $3 \pm 0.005\text{-mL}$ (preferably syringe pipet for safe, precise pipetting of the sulfuric acid; see Fig. 1).
5. *Laboratory Sterilizing Autoclave*, preferably with boiler to provide clean steam, and fitted with time clock-actuated steam and water valves.
6. *High-Speed Mixer* to disintegrate tough pulp sheets.
7. *Analytical Balance*, with a capacity of 200 g and a sensitivity of 0.1 mg

Reagent

1. *Sulfuric Acid* (72.0 ± 0.1 weight %)—To 1 volume of water add slowly while stirring vigorously 2 volumes of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84). When cool, standardize against a standard alkaline solution. Adjust the acid strength to 72.0 ± 0.1 weight %.

10. Procedure

10.1 *Primary Hydrolysis*—Weigh 0.30 g of loose pulp (Note 1) into a 25 by 90-mm shell vial and add 3.0 mL of H_2SO_4 (72 %) (Note 2). Using a 5 by 150-mm rod, stir at intervals as required to dissolve the pulp rapidly and completely (Notes 3 and 4), leaving the stirring rod in the vial at all times. Maintain the mixture at $30.0 \pm 0.1^\circ\text{C}$ for a total of 1 h. Then dilute by washing the contents of the vial into a 250-mL, tared, wide-mouth bottle with 84 mL of water measured in a 100-mL graduate. The bottles should be of such size as to fit the cups of a laboratory centrifuge.

NOTE 1—Precise weighing is necessary if the determination is to be on an absolute basis; it is not necessary if the final determination is based on the ratios of the component sugars.

NOTE 2—The use of a hypodermic syringe pipet, equipped with suitable stops, greatly facilitates the precise pipetting of replicate 3-mL portions of H_2SO_4 (72 %) with an accuracy of ± 0.005 mL. One design of such a pipet is shown in Fig. 1. An aluminum button on the end of the plunger and a brass band around the barrel are bonded to the glass with epoxy resin. The projection on the brass band is slotted so as to

serve as a guide as well as a stop for the volume adjusting rod. Accurate dispensing of acid facilitates neutralization on a routine basis.

NOTE 3—Sheeted specimens may prove difficult to dissolve. They should be dispersed in a water slurry by means of a high-speed stirrer or blender, then filtered on a Büchner funnel, washed with acetone, and air-dried.

NOTE 4—When many specimens are being handled, primary hydrolysis of successive specimens should begin and end at 2-min intervals.

10.2 *Secondary Hydrolysis*—Cover the bottles containing the diluted hydrolyzates from the primary hydrolysis with 50-mm watch glasses and place them in a preheated autoclave. Admit live steam to the autoclave and continue heating for 4 h (Note 5) at the boiling point of water (saturated steam at atmospheric pressure) (Note 6), following which cool the autoclave to room temperature. A total of 50 specimens can be processed simultaneously in a standard 20 by 30-in. (508 by 762-mm) laboratory sterilizer.

NOTE 5—For routine use, it is convenient to control the duration of the incoming steam by means of a solenoid valve actuated by a time clock. At the end of the steaming cycle, a second clock-operated solenoid valve can be used to admit cooling water to a coil of copper tubing in contact with the metal rack supporting the sample bottles. In this way, the 4-h secondary hydrolysis can be completed overnight and the specimens are ready for neutralization next morning.

NOTE 6—It is possible to reduce the time required for secondary hydrolysis by going to higher steam pressures in the autoclave (2). Because of increased rate of sugar decomposition, however, it then becomes necessary to exercise rigid control over each step in the autoclaving operation so as to maintain a high level of accuracy. On this basis, the use of atmospheric steam pressure in an autoclave fitted out as described above would seem to present fewer problems. In addition, recent work has shown that complete hydrolysis can be accomplished with less net destruction at the lower temperature.

NEUTRALIZATION

11. Apparatus

- 11.1 *pH Meter*.
- 11.2 *Titration Assembly*, with stirrer, as shown in Fig. 2.
- 11.3 *Balance*, sensitive to 0.1 g.
- 11.4 *Centrifuge*.

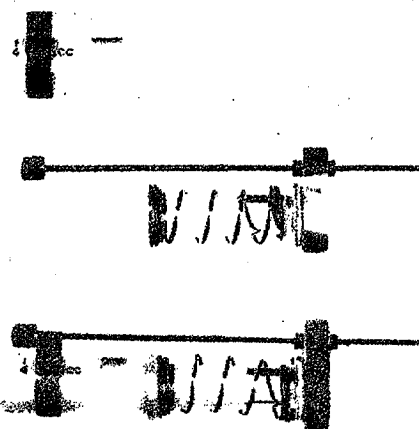


FIG. 1 Adjustable Syringe Pipet

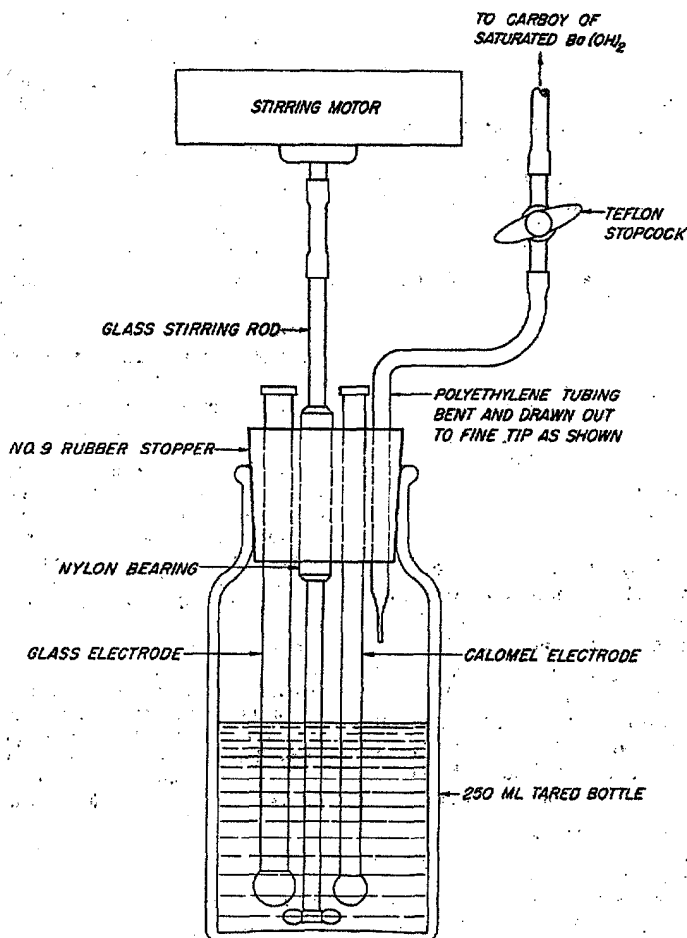


FIG. 2 Barium Hydroxide Titration Assembly

11.5 *Calibrated Pipets*, 200-mL, for quantitative transfer of hydrolyzate to the evaporator. These pipets are required only when the sugar ratio method is not adequate.

12. Reagents

12.1 *Barium Hydroxide Solution* (70 g/L)—Dissolve 70 g of barium hydroxide ($\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$) in water, dilute to 1 L, and protect suitably from atmospheric carbon dioxide.

12.2 *Ion-Exchange Resin*, anion exchanger, weakly basic, granular form, 20 to 50-mesh.⁵

13. Procedure

13.1 Neutralize the hydrolyzates by direct titration with a saturated barium hydroxide ($\text{Ba}(\text{OH})_2$) solution, as follows (Note 7): Affix an individual specimen in its tared bottle to the titration assembly shown in Fig. 2. Admit the $\text{Ba}(\text{OH})_2$ solution directly from the stock carboy, while the bottle contents are under vigorous agitation, to a pH of approxi-

mately 5.5. Put the bottle aside until all other specimens in the series are neutralized. The barium sulfate (BaSO_4) formed in these relatively concentrated solutions occludes H_2SO_4 , which is slowly released, resulting in a decreasing pH. By allowing the solution to stand for about 1 h and then readjusting the pH to the desired 5.3 with additional $\text{Ba}(\text{OH})_2$ solution, the problem of drift is eliminated.

NOTE 7—Many laboratories prefer to neutralize sugar hydrolyzates with ion-exchange resins, using both anion and cation-exchange columns. When handling large numbers of specimens, however, columns are inconvenient, and simple batch neutralization with anion resin only has been found adequate (2). Neutralization is accomplished by mixing the cooled hydrolyzates with approximately 25 g of the freshly washed ion-exchange resin. This quantity of resin is such as to raise the pH to 3.8 to 4.0 in the convenient time of 25 to 30 min, following which the solution is recovered by filtration and washing. Here again, quantitative sugar recovery from the resin is necessary only if the determination is to be made on an absolute basis. The need for prewashing the resin arises from the fact that relatively large amounts of water-soluble materials are released by the resin during storage.

13.2 Adjust the bottles containing the neutralized hydrolyzates to a net weight of 258.4 g by the addition of

⁵ Amberlite IR-4B, manufactured by Rohm & Haas Co., Philadelphia, PA, has been found satisfactory for this purpose.

water. Each bottle now contains 250 mL of solution and 8.4 g of BaSO_4 .

13.3 Centrifuge the bottles to settle the BaSO_4 and take a 200-mL aliquot of the clear supernatant liquid from each for evaporation.

NOTE 8—To avoid sugar losses by yeast or microbial action, storage of neutralized hydrolyzates should be done under refrigeration. The addition of a few drops of toluene affords added protection.

NOTE 9—If, as in the usual case, it is sufficient to measure ratios of the carbohydrate constituents, the 200-mL aliquot and the prior

adjustment to 250-mL need not be done accurately. If, however, it is desired to determine composition on an absolute basis, exact volumes must be used.

CONCENTRATION

14. Apparatus

14.1 *Evaporator and Condenser*—A simple, inexpensive evaporator that is rapid in operation, permits high recovery of product, and causes a minimum of sugar decomposition is

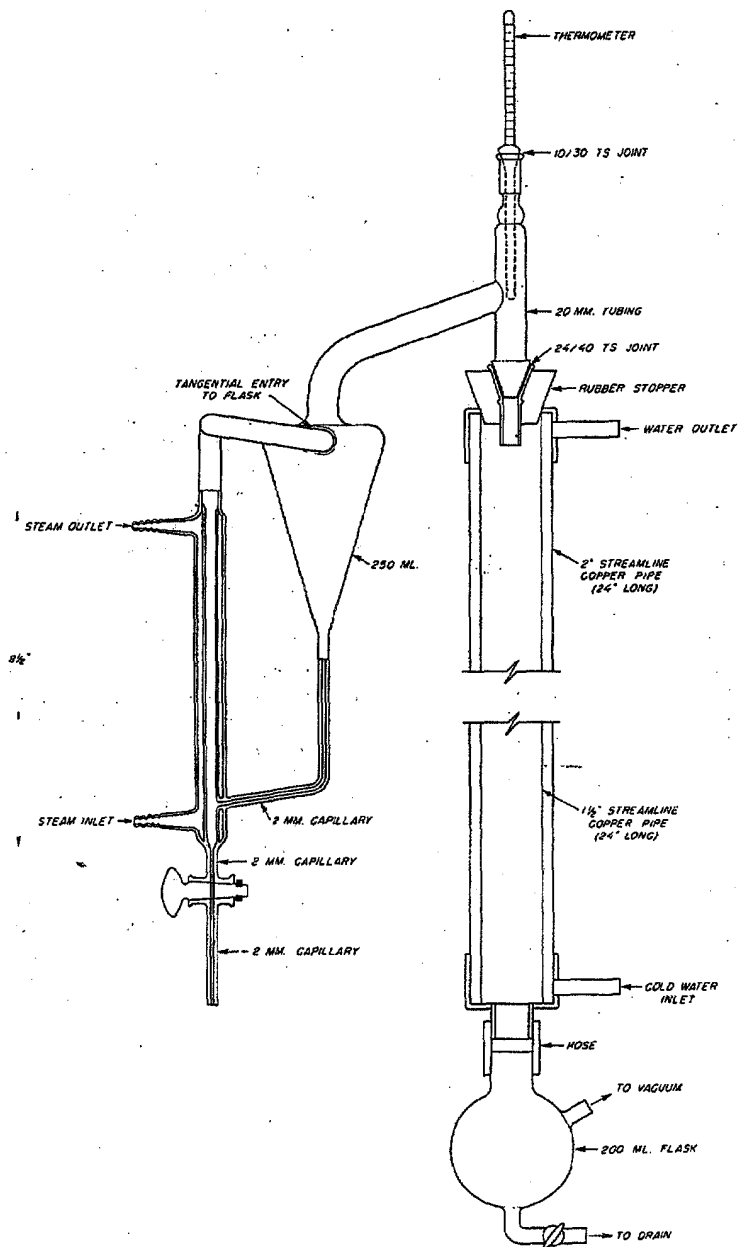


FIG. 3 Evaporator Unit for Concentrating Sugar Solutions

shown in Fig. 3 (2) (Note 10). With a bank of three such units, one operator can conveniently evaporate 50 specimens a day from 200 mL of 3 mL (Note 11).

NOTE 10—The evaporators shown in Fig. 3 can be made by a glass blower from the indicated design. The main critical feature of the evaporator is that of providing a smooth tangential entry from the boiler section to the liquid reservoir.

NOTE 11—In unbuffered systems, a 100-fold concentration of solutions containing a nonvolatile strong acid results in a drop of 2 units in pH. On complete solvent removal, much greater pH changes result. Because of the buffering action of materials dissolved from the resin, resin-neutralized solutions show much less pH change on evaporation than do those neutralized with barium hydroxide. Regardless of the neutralization or evaporation technique employed, it must be kept in mind that excessive pH changes can lead to serious error in the sugar content of hydrolyzates. Too low a pH causes reversion, while too high a pH results in epimerization. Adjustments in technique should be made as necessary to ensure that the pH of the concentrate will be maintained in the 3.5 to 5.0 region.

14.2 *Vapor Washing Unit*, consisting of a 3-L flask with ground joint fitting the evaporator and arranged for boiling water rapidly. This unit is required only when the sugar ratio method is not adequate.

14.3 *Pressure Controllers* for the water and steam supply to the evaporators.

14.4 *Aspirator*, one for each evaporator.

15. Procedure

15.1 With the aspirator turned on and the condenser in operation, draw the entire 200 mL of hydrolyzate into the evaporator through the capillary stopcock, taking care to admit the last portion slowly and to close the cock just as air enters the bore. The solution should have been previously cooled to a temperature below the boiling point at the pressure used for evaporation (Note 8). Then adjust the stopcock to provide a very slow stream of air bubbles into the evaporator. This promotes smooth ebullition. Turn on steam into the boiler jacket.

NOTE 12—The addition of pressure controllers to the steam and water supply greatly facilitates the operation of the evaporators. With pressure control, no individual adjustment of steam to the evaporator or water to the condenser is necessary. Pressures of 5 psi (30 kPa) for steam and 15 psi (100 kPa) for water have proven satisfactory.

15.2 Carry out the last stages of evaporation cautiously; in fact, this can be accomplished with the small amount of warm condensate left in the heater jacket as the steam is turned off. The concentration required at this point depends on the composition of the pulp, but a final volume of 2 to 4 mL is common. This final volume can be read with sufficient accuracy by using calibration marks on the evaporator. The concentrate is recovered in high yield by draining it from the stopcock into a small plastic-capped vial (Note 13). The solutions are now ready for chromatography. They should be kept under refrigeration until so used.

NOTE 13—Quantitative removal of sugar concentrate from the evaporator may be effected by removing the evaporator from the condenser and fitting it to a flask containing rapidly boiling water. The vapors condensing on the cold inside surfaces wash out the sugars with a minimum of dilution.

CHROMATOGRAPHIC SEPARATION

16. Summary of Procedure

16.1 In this separation procedure the previously prepared

sugar concentrates are applied to filter paper by streaking, the paper is irrigated by descending chromatography, and the sugar-containing areas are cut out for chemical analysis.

17. Apparatus

17.1 *Plywood Jig* for folding paper to conform with requirements of the paper holders in the chromatographic cabinets.

17.2 *Chromatographic Streaking Device*, as shown in Fig. 4.

17.3 *Chromatographic Cabinets* for 18¼ by 22½-in. (463.6 by 571.5 mm) paper.

17.4 *Separate Chromatographic Room* held at $30 \pm 0.1^\circ\text{C}$ (Note 15).

17.5 *Steel Template*, 7.5 ± 0.1 by 60 cm (any convenient thickness).

17.6 *Paper-Cutting Knife*.

17.7 *Circulating Oven* for heating guide strips at 105°C .

17.8 *Filter Paper*—Special filter paper for paper chromatography, in 18¼ by 22½-in. sheets. Two grades are required, a normal high grade, rapid paper for general work, and a thick, strong paper of medium filtering speed with smooth surface.

18. Reagents

18.1 *Irrigation Solvent*—Mix 10 volumes of butanol with 3 volumes of pyridine and 3 volumes of water. Also needed when arabinose is present is a solution of 9 volumes of ethyl acetate, 2 volumes of acetic acid, and 2 volumes of water.

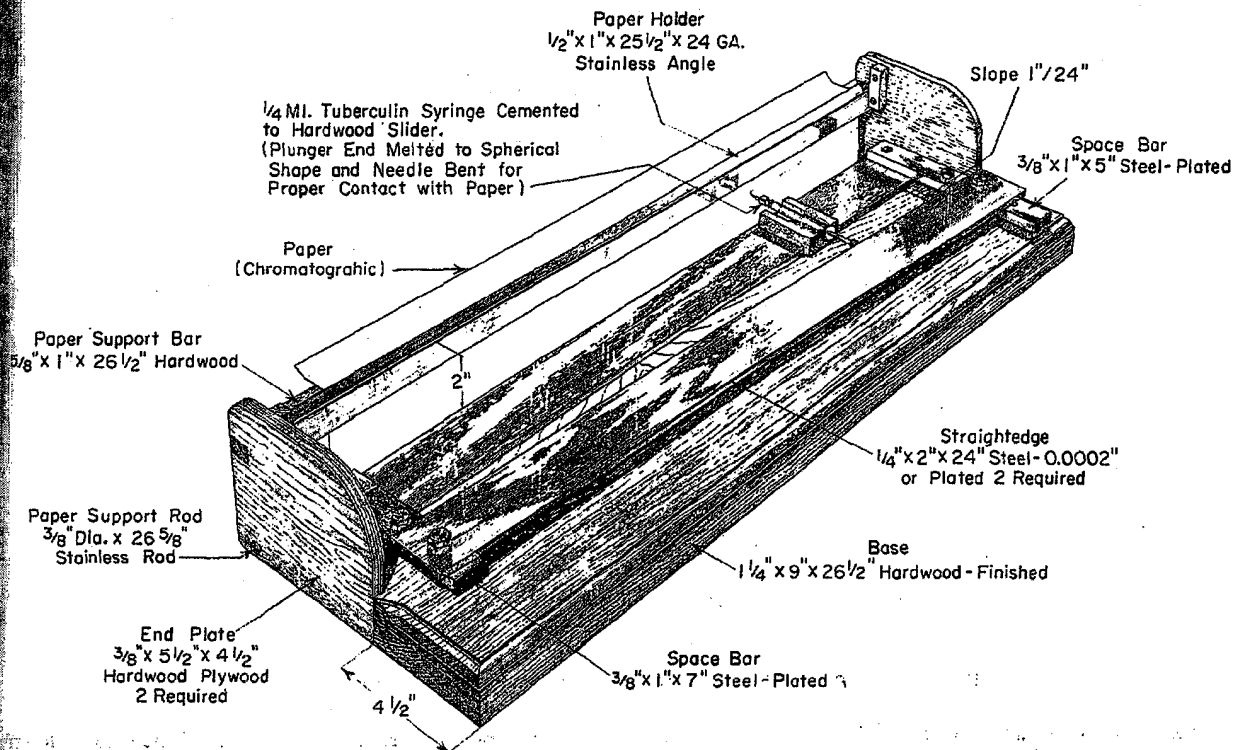
18.2 *Indicating Solution*—Mix 800 mL of butanol, 40 mL of water, 8 mL of aniline, and 14.7 g of phthalic acid. This may be used as a spray. For use as a dip, mix equal volumes of this solution and ethyl ether.

19. Procedure

19.1 *Sample Application*—Carry out chromatographic separations on 18¼ by 22½-in. (463.6 by 571.5-mm) sheets of chromatographic paper, normal, rapid filtering type,⁶ having folds made on a jig in positions to suit the requirements of the paper holders in the chromatographic cabinet. The long dimension of the paper is the machine direction and is the direction used for solvent flow. Hang the folded paper over the supporting bar of the streaking device illustrated in Fig. 4 (Note 14). Move the wooden holder bearing the syringe loaded with sugar concentrate down the steel bar to apply a uniform streak of specimen to the starting line. With the normal chromatographic paper, the maximum loading is limited to about 6 µL of solution per centimetre of streak, or about 400 µg of total sugar per centimeter of streak. Loading on heavier papers can be increased in proportion to paper weight. Air-dry the streaked papers.

NOTE 14—The application of specimen to the paper as a continuous streak rather than as spots has the advantage, in direct photometry, of providing a uniform band of specimen; and when chemical methods are used, it has the advantage of loading the paper to the limit of its capacity, thereby providing the most favorable ratio of applied sugar to paper blank. The streaking device shown in Fig. 4 is simple to construct, and its precision and utility have been proven in extensive use. The only

⁶ A Beckman Model DU spectrophotometer, manufactured by Beckman Instruments, Inc., 2500 Harbour Blvd., Mail Station E 31D, Fullerton, CA 92634, has been found satisfactory for this purpose.



NOTE—1 in. = 25.4 mm.

FIG. 4 Device for the Precise Linear Application of Pulp Hydrolyzates to Chromatographic Paper

critical details of construction are the two straightedges whose angular separation governs the loading of sugar concentrate on the paper. The indicated slope and syringe size are suitable for the normal chromatographic paper.

19.2 Solvent Irrigation and Isolation of Sugars—For irrigation, hang the streaked papers from the supporting rods in the chromatographic cabinet (Note 15), then add approximately 500 mL of solvent directly to the bottom of the cabinet or to a shallow tray resting on the bottom. Close the cabinet for about 1 h to permit vapor equilibration. Add solvent to the troughs into which the starting edges of the papers project. Continue irrigation for a period of time sufficient to move xylose nearly to the bottom of the sheets. When using the butanol-pyridine-water solution (Note 16), this time period is approximately 55 h for the normal chromatographic paper (Note 17) at 30°C.

19.3 After completion of the irrigation period, dry the papers in a suitably ventilated space. Cut four strips spaced about equally apart vertically from the starting line of each chromatogram, using a steel template 75 ± 1 mm wide by 600 mm long and a paper knife. (With a little experience, this cutting can be done accurately to a fraction of a millimetre.)

19.4 Next dip the guide strips remaining into a solution made by mixing equal volumes of ethyl ether and indicating solution. Allow the strips to drip dry, then heat in a circulating oven at 105°C for 15 min to color the sugar-containing areas. Match the previously cut out vertical strips

with the guide strips, and remove the areas containing the glucose, mannose, and xylose. At this point, individual sugar components of the initial pulp hydrolyzate are present on four replicate tabs of paper, each 75 mm wide by approximately 2 in. (50 mm) long in the initial-length direction of the sheet.

NOTE 15—Temperature differences across a cabinet can have a serious effect on the parallelism of the separating sugar zones. For this reason, it is desirable to locate the cabinets in a small thermostatted room. By means of a mercury regulator, operating one or more household circulating fan heaters, room temperature can be controlled to approximately 0.1°C. Auxiliary fans directed at the cabinets enhance air circulation. Cabinets should be supported several inches off the floor on wooden blocks. Because of the obnoxious odor of pyridine, adequate ventilation facilities should be provided to remove solvent vapors when papers are being removed from the cabinets and air-dried.

NOTE 16—Purified wood pulps generally contain only glucan, mannan, and xylan as their carbohydrate components. The butanol-pyridine-water system has proved entirely adequate for such separations. This system fails to resolve mannose and arabinose, however, and in the presence of large amounts of glucose, galactose cannot be cleanly separated from glucose. In pulps yielding these other sugars, the hydrolyzates are streaked on narrower sheets of paper which are first irrigated in chromatographic jars with an ethyl acetate-acetic acid-water system (9 + 2 + 2) for 28 h then dried and irrigated in the cabinets with a butanol-pyridine-water system (10 + 3 + 3) for 32 h. This double irrigation separates mannose, arabinose, and xylose into well defined bands. Galactose may be isolated by extending the irrigation with butanol-pyridine-water to the point where glucose is at the bottom of the sheet, all the other sugars being off. Galactose then occupies a separated

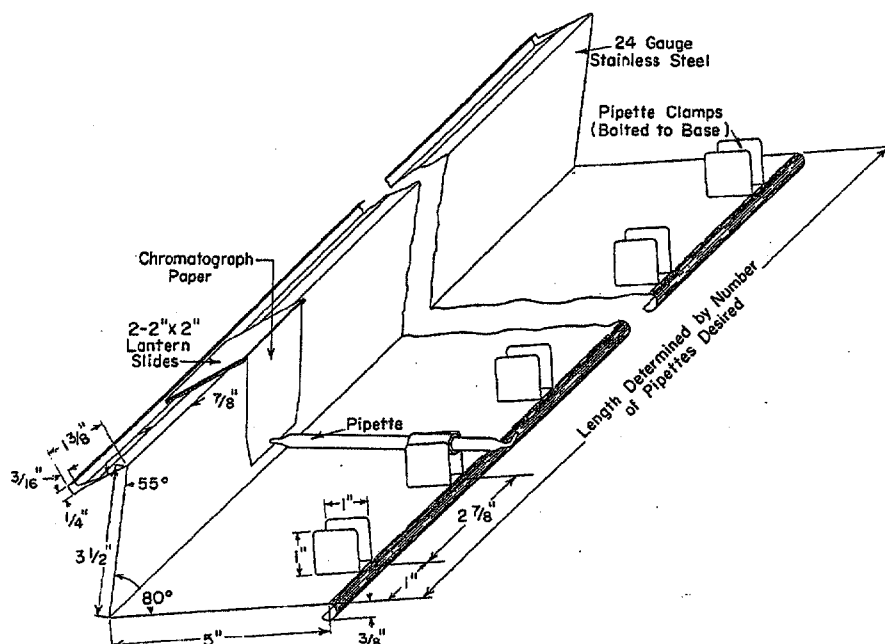


FIG. 5 Elution Rack + Calibrated Pipet for Recovering Sugars from Chromatogram

band between glucose and the starting line. The ethyl acetate-acetic acid-water system provides rather clean separation of all five sugars in small jars, but its performance in cabinets has been disappointing.

ELUTION

20. Summary of Procedure

20.1 The determination of small quantities of sugar by chemical analysis requires that individual sugars be isolated from their respective 2 by 3-in. (51 by 76-mm) tabs of paper. This can be done quantitatively with little solvent and little manipulation by a combination of techniques (3, 4).

21. Apparatus

21.1 *Elution Rack*, as illustrated in Fig. 5.

21.2 *Cabinet* for elution rack (a simple glass-covered box lined with blotting paper).

21.3 *Elution Pipets*, 0.75-mL, constructed from 3-mm (inside diameter) tubing as described in Note 20.

21.4 *Glass Plates* (2 by 2-in. (51 by 51-mm) transparency cover glasses) for holding paper in the elution rack.

21.5 *Adapter Plate* (optional) for use in elution of heavier paper, constructed as described in Note 18.

22. Procedure

22.1 Barely catch the tabs of paper containing mannose and xylose between the edges of a pair of 2 by 2-in. (51 by 51-mm) glass plates and hang them in an elution rack as shown in Fig. 6, with the 3-in. (76-mm) dimension vertical (Note 18). Position the elution pipets so as to touch near the bottom edge of the tabs. To prevent excessive evaporation of water, the entire assembly should be contained in a glass-covered humidified box (Note 19).

22.2 Start elution by filling the trough with water, which

rises by capillarity between the plates and then flows down the paper and into the pipets. When a pipet is filled, capillary forces prevent flow through the pipet, and surplus water flows past the tip. The volume provided by a 0.75-mL pipet (Notes 20 and 21) is more than adequate to remove all of the sugar from the tab, so that the last water to enter is sugar-free. Quantitative transfer of sugar to a test tube for analysis is achieved by draining the pipet through the end not in contact with the paper.

22.3 Soak the paper tabs containing glucose in 25 mL of water for about 15 min with occasional swirling. Filter the eluates through glass wool to remove fibers, following which take replicate 0.75-mL aliquots of the supernatant solutions for analysis.

NOTE 17—Uniformity of performance and of paper blank are of critical importance in the selection of a chromatographic paper for quantitative work. The normal inexpensive, high grade, rapid paper used for general work⁶ has been widely used for this purpose and can be purchased in a grade especially selected for chromatographic work. For high-purity pulps and for cotton where minor constituents are present in very small quantities, the use of a thick, strong paper of medium filtering speed with smooth surface⁷ may be advantageous, since it can be loaded with three times as much solution.

NOTE 18—With thicker papers, the 2 by 2-in. (50.8 by 50.8-mm) glass plates may become separated to such an extent that water will not rise between them. A stainless steel adapter plate, 2 in. in width and of length equal to that of the elution rack, can be made to solve this problem. The thickness of one edge of this plate is reduced by milling a cut 1/4 in. (3.2 mm) wide by 0.008 in. (0.20 mm) deep the full length of the plate. The paper tabs can now be caught in the space between this milled depression and a covering 2 by 2-in. glass slide. Elution is carried out in the usual way.

⁷ Whatman No. 3MM paper, available from, Whatman Inc., 9 Bridewell Place, Clifton, NJ 07014, has been found satisfactory for this purpose.

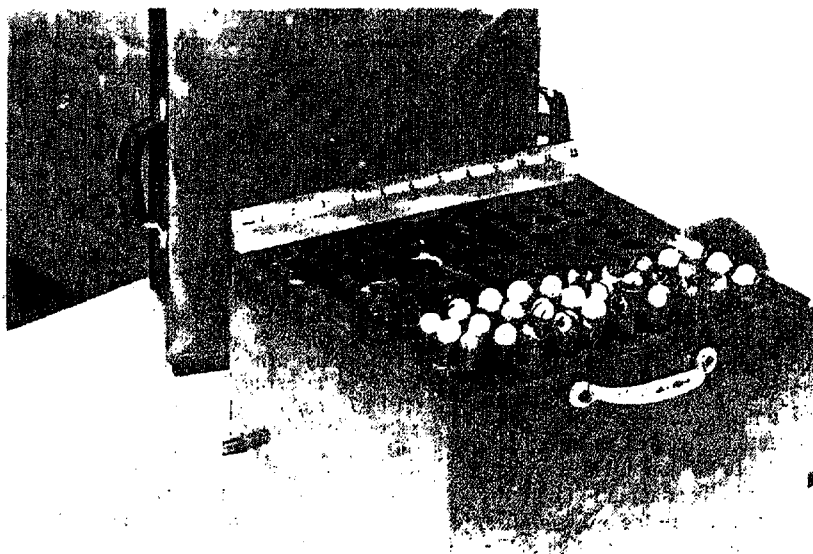


FIG. 6 Combination Steam Bath-Test Tube Rack for Sugar Analysis

NOTE 19—Humidification of the elution cabinet is easily accomplished by lining the walls with strips of wet blotting paper.

NOTE 20—The elution pipets are made from 3-mm (inside diameter) borosilicate glass tubing and calibrated to deliver 0.75 ± 0.005 mL of water. This volume is suitable for use with either paper described in Note 17. Draw down the ends of the pipets to an inside diameter of approximately 0.020 in. (0.51 mm), bending one end up at about a 60° angle as illustrated in Fig. 5. Grind the tips flat and then give them a slight bevel to reduce chipping. Capillary forces are now such that eluate is readily drawn into the straight end of the pipet but does not flow out of the bent end.

NOTE 21—After every use, wash the pipets by drawing through them, in succession, dilute sodium hydroxide, acid dichromate, and water. Dry by leaving them for $\frac{1}{2}$ h in a bottle evacuated with an aspirator. Clean the 2 by 2-in. slide cover glasses in dichromate and keep wet until use.

ANALYSIS OF SEPARATED SUGARS

23. Significance and Use

23.1 The chemical method of analysis is well adapted to work in which the major emphasis is on the accurate determination of minimal quantities of "extraneous" carbohydrates present in a cellulose. The best reagents are those which provide a maximum discrimination against the soluble polysaccharides of the paper. The copper reagent (5) and the arsenomolybdate chromogenic reagent (6) have proven quite suitable from this standpoint.

24. Apparatus

24.1 *Combination Steam Bath—Test Tube Rack* (Fig. 6).

24.2 *Glass Marbles*, 15 to 16 mm in diameter.

24.3 *Calibrated Syringe Pipets*, 1.00 ± 0.001 mL and 2.25 ± 0.002 mL (Fig. 1 and Note 2).

24.4 *Spectrophotometer*⁵ using 3-mL borosilicate glass cuvettes with a 1-cm light path.

25. Reagents

25.1 *Copper Reagent (5)*—Dissolve 24 g of anhydrous sodium carbonate (Na_2CO_3) and 12 g of Rochelle salt ($\text{NaKC}_4\text{H}_4\text{O}_6$) in about 250 mL of boiled and cooled water. Add to this solution 4 g of hydrated copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) dissolved in 40 mL of boiled water and mix. Add 16 g of sodium bicarbonate (NaHCO_3), and when dissolved, pour the combined solution into a 1000-mL graduated cylinder. Dissolve 180 g of anhydrous sodium sulfate (Na_2SO_4) in about 500 mL of hot water, and boil the solution to expel air. When cool, add this solution to the cylinder. Dilute the combined mixture to the mark and mix thoroughly.

NOTE 22—It is preferable to prepare a 10-L stock of the copper reagent several weeks in advance of its use. This allows impurities and a slight amount of cuprous oxide (Cu_2O) to settle. The supernatant solution may then be siphoned into a clean bottle.

25.2 *Arsenomolybdate Chromogenic Reagent (6)*—Dissolve 25 g of ammonium molybdate ($(\text{NH}_4)_2\text{MoO}_4$) in 450 mL of water, add 21 mL of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) mix, and add 3 g of monobasic sodium orthoarsenate ($\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$) dissolved in 25 mL of water. Place this solution in an incubator at 37°C for 24 to 48 h before use and store in brown bottles.

NOTE 23—For routine use, this solution should also be made up in 10-L quantities.

26. Preparation of Calibration Curves

26.1 *Glucose Calibration Curve*—As will be seen later, it is convenient to express all sugars in terms of glucose equivalent. Prepare a calibration curve for this purpose by pipetting 0.75 mL of glucose solutions, containing from 0 to 210 μg of sugar, into $\frac{3}{8}$ by 6-in. (15.9 by 152-mm) test tubes and processing as described in Section 27 (Note 24). This is

most accurately done by dissolving 0.2800 g of glucose in 1 L of water (210 μ g/0.75 mL) and diluting this to give the desired number of calibration points. A plot of absorbance versus glucose concentration provides the desired calibration curve.

NOTE 24—The use of individually calibrated syringe pipets greatly facilitates the multiple pipettings encountered in this sugar analysis procedure.

26.2 All sugars undergo measurable destruction when subjected to the acid conditions employed for saccharification. Sugar degradation products can migrate at the same speed as the sugars. Each sugar exhibits a characteristic reducing powder toward the copper reagent. Sugar values must be converted to sugar anhydride. Appropriate individual corrections for these matters are troublesome. It is possible, however, to account in one operation for all conversion and correction factors involved in paper chromatographic analysis by the use of empirical calibration curves. Such a correction is especially adapted to high-purity pulps where mannan and xylan represent the only significant nonglucose carbohydrates present. It is advised that calibration curves be established specifically for the system under study. This is illustrated by the procedure described in 26.3, which is adapted to purified pulps with mannan and xylan contents up to 5 %.

26.3 *Empirical Calibration Curves for Mannan and Xylan in Purified Pulps*—Prepare 0.300-g quantities of mixtures of glucose, mannose, and xylose containing, respectively, 0.00, 1.00, 2.00, 3.00, 4.00, and 5.00 % of both mannose and xylose. These specimens will have respective mannan equivalents of 0.00, 1.00, 2.00, 3.00, 4.00, and 5.00 % and respective xylan equivalents of 0.00, 0.98, 1.96, 2.94, 3.92, and 4.90 %. To avoid weighing such small quantities of mannose and xylose, solutions of the desired mixtures may be prepared and suitable aliquots (containing 0.300 g of total sugar) pipetted into the vials used for primary hydrolysis and dried in a vacuum desiccator.

26.3.1 Hydrolyze these sugar mixtures in duplicate, neutralize, concentrate, chromatograph, and analyze exactly in accordance with the procedures described for cellulose pulp analysis. This yields the glucose, mannose, and xylose content of each mixture in terms of micrograms of glucose equivalent. From these values subtract the amount, in micrograms, of apparent reducing sugar due to the paper blank (Note 25). Then total the corrected quantities, in micrograms, of glucose, mannose, and xylose. By dividing the corrected quantities of xylose (as glucose) by this total (all sugars as glucose), obtain the apparent percent of xylose in each mixture. Calculate the apparent percent of mannose similarly.

26.3.2 Construct two curves, one in which the apparent percent of xylose is plotted against the calculated xylose content of the original mixtures as listed above, and second, in which the apparent percent of mannose is plotted against the calculated mannan content of the original mixtures (Note 26). These are the master calibration curves, and should serve as long as the defined technique is followed.

NOTE 25—All chromatographic papers contain traces of soluble reducing substances which constitute a source of error in the sugar determination. Since the absolute amount of these materials varies widely with the different papers and may also vary appreciably between different lots of the same paper, it is not feasible to account for them in the master calibration curves. For a given lot of paper, the correction for apparent reducing sugar may be obtained by irrigating several randomly selected sheets in the usual way with spots of mannose and xylose applied to the starting line in the guide-strip regions. Two by three-in. (51 by 76-mm) tabs of paper are then cut from the sugar-free regions adjacent to the mannose and xylose areas, and eluted. The eluates are analyzed for their apparent reducing sugar content. The mean values so obtained (in micrograms) provide the desired corrections for paper blank. It has been found that there is a small but significant difference in the blank correction for the mannose and xylose regions. The uncertainty in the blank between successive tabs of a given lot of paper is on the order of 1 μ g.

NOTE 26—The described calibration procedure was designed to accommodate pulps having a mannan and xylan range between 0 and 5 %. By strict adherence to the prescribed techniques and with adequate replication, errors can be held to a level of about 0.1 % of a minor constituent, based on the total carbohydrate. For highly purified pulps with mannan and xylan contents in the 1 to 1.5 % range, the master calibration curves can be made more sensitive by selecting a series of five or more calibration points over the range of 0 to 1.5 % mannan and xylan. This illustrates the principle of calibrating specifically for the system under study.

27. Procedure

27.1 To each of the 0.75-mL sugar eluates (Section 22) contained in $\frac{1}{2}$ -in. (15.9 by 152-mm) test tubes add 1.00 mL of copper reagent. Cover the tubes with the glass marbles and heat them by direct steam for 20 min in the combination steam bath and test tube rack illustrated in Fig. 6. Connect the steam bath to a water line to provide rapid cooling. Next, add 1.00 mL of the chromogenic reagent to each tube and shake the contents to expel carbon dioxide. Finally, add 2.25 mL of water to each tube and thoroughly mix the contents (Note 24).

27.2 Determine absorbances at 520 nm with 3-mL cuvettes and a spectrophotometer.⁶ Use a reagent blank in the reference cell. In cases where absorbance exceeds 1.0, dilute both sample and blank with an additional 5.00 mL of water.

27.3 From the glucose calibration curve obtain the glucose equivalent (see 26.1), and from the master calibration curves (see 26.3) determine the composition of the pulp.

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Standard Test Methods for Carboxyl Content of Cellulose¹

This standard is issued under the fixed designation D 1926; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. 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 carboxyl content, or ion-exchange capacity, of cellulose from any source. Two test methods are described, the sodium chloride-sodium bicarbonate method (1)² and the methylene blue method (2). The test methods must be used within their limitations, and it must be recognized that there is no way of determining the accuracy of any method for the determination of carboxyl. The precision of the sodium chloride-sodium bicarbonate method is low in the lower range of carboxyl values. The methylene blue method can be used over the whole range of carboxyl values; it is especially useful in the low range. It is not applicable to the determination of carboxyl in soluble carbohydrate material. Although these test methods may be used to determine the ion-exchange capacity of unbleached pulps, the residual lignin will cause an undetermined error, especially the sulfonic acid groups in unbleached sulfite pulps (3).

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 1193 Specification for Reagent Water³

3. Significance and Use

3.1 These test methods measure the amount of carboxyl groups present in wood or cotton linter pulp. Carboxyl groups are indicative of the surface charge of the pulp which is a very important quantity for use in the papermaking industry.

4. Purity of Reagents

4.1 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.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.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

SODIUM CHLORIDE-SODIUM BICARBONATE METHOD

5. Summary of Test Method

5.1 In the sodium chloride-sodium bicarbonate method the specimen is deashed with hydrochloric acid, washed, soaked in sodium chloride-sodium bicarbonate solution, filtered, and an aliquot of the filtrate titrated with 0.01 *N* hydrochloric acid to a methyl red end point. The difference between the concentration of the filtrate and of the sodium chloride-sodium bicarbonate solution is a measure of the ion-exchange capacity of the cellulose.

6. Reagents

6.1 *Hydrochloric Acid, Standard (0.01 N)*—Prepare and standardize a 0.01 *N* solution of hydrochloric acid (HCl).

6.2 *Hydrochloric Acid (1 + 99)*—Dilute 1 volume of concentrated HCl (sp gr 1.19) with 99 volumes of water.

6.3 *Methyl Red Indicator Solution.*

6.4 *Sodium Chloride-Sodium Bicarbonate Solution*—Dissolve 5.85 g of sodium chloride (NaCl) and 0.84 g of sodium bicarbonate (NaHCO₃) in water and dilute to 1 L.

6.5 *Sodium Hydroxide Solution (0.4 g/L)*—Dissolve 0.4 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

7. Procedure

7.1 Condition the specimen in the atmosphere near the balance for at least 20 min before weighing duplicate portions of 2.5 ± 0.01 g. At the same time, weigh specimens for the determination of moisture. Disintegrate the specimen in water, filter through fritted glass, and disperse to about 1 % consistency in HCl (1 + 99) at room temperature. After 2 h collect the specimen on a fritted-glass filter funnel and wash with water saturated with carbon dioxide (CO₂). Continue the washing until the filtrate, after boiling, does not require more than 1 or 2 drops of NaOH solution to give an alkaline color with methyl red.

7.2 Weigh the wet pulp pad, transfer it immediately to a 250-mL glass-stoppered Erlenmeyer flask, add 50 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 D01.36 on Cellulosics.

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² The boldface numbers in parentheses refer to the list of references at the end of these test methods.

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."

NaCl-NaHCO₃ solution with a pipet, and shake to obtain a homogeneous slurry (Note 1). Allow the mixture to stand for 1 h at room temperature. Filter through a clean, dry, fritted-glass funnel, pipet a 25-mL aliquot of the filtrate into an Erlenmeyer flask, and titrate with 0.01 N HCl, using methyl red solution as an indicator. When the first change in color occurs, boil the solution for about 1 min to expel the carbon dioxide and continue the titration to a sharp end point.

NOTE 1—If the cation-exchange capacity is very low, use a solution containing about 5.85 g of NaCl and 0.42 g of NaHCO₃ per litre. It is important that the excess of NaHCO₃ be large enough that the pH does not fall below 7.0.

7.3 Pipet 25 mL of the NaCl-NaHCO₃ solution into an Erlenmeyer flask and titrate as described in 7.2.

8. Calculation

8.1 Calculate the cation-exchange capacity, c , of the specimen in milliequivalents per 100 g as follows:

$$c = \left(b - a - \frac{av}{50} \right) \frac{2}{G} \quad (1)$$

where:

G = weight of oven-dry specimen, g,

v = weight of water in the wet pulp pad, g,

a = millilitres of 0.01 N HCl consumed by 25 mL of filtrate, and

b = millilitres of 0.01 N HCl consumed by 25 mL of the NaCl-NaHCO₃ solution.

9. Report

9.1 Until more data are obtained on the precision of this test method, it is suggested that the ion-exchange capacity be reported to 0.01 milliequivalent/100 g of pulp.

10. Precision and Bias

10.1 The precision of this test method varies with the carboxyl content and from laboratory to laboratory. Precision data is available in Ref (4).

10.2 No statement on bias can be made as no suitable reference material exists for determining bias.

METHYLENE BLUE METHOD

11. Summary of Test Method

11.1 In the methylene blue method the specimen is treated with 0.0002 M methylene blue solution buffered to a pH of 8 with diethylbarbituric acid (barbital). The decrease in methylene blue concentration, measured photometrically, is a function of the ion-exchange capacity of the cellulose.

12. Apparatus

12.1 *Spectrophotometer or Filter Photometer*, capable of measuring absorbance near 620 nm.

12.2 *Shaker or Mixer* for agitating the specimens in the methylene blue solution. A wheel or rod, to which the specimen vials can be attached, that rotates at about 15 r/min, has proven satisfactory.

12.3 *Centrifuge*, capable of settling the cellulose from the methylene blue solution.

13. Reagents

13.1 *Buffer, Stock Solution*—Dissolve 1.151 g of dieth-

ylbarbituric acid (barbital) in water, add the equivalent of 0.16 g of sodium hydroxide using a standard solution and buret, and dilute with water to 1 L in a volumetric flask.

13.2 *Hydrochloric Acid (1 + 99)*—Dilute 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 99 volumes of water.

13.3 *Methylene Blue, Stock Solution (0.002 M)*—Dissolve 0.640 g of methylene blue in water, making allowance for moisture, and dilute to 1 L in a volumetric flask.

NOTE 2—Information on the determination of the purity of methylene blue is given in the literature (5).

13.4 *Methylene Blue—Buffer Solution (0.0002 M)*—Mix 1 volume of methylene blue stock solution with 1 volume of buffer stock solution and dilute to a total of 10 volumes in a volumetric flask. The volume of solution to be prepared will vary with the requirements. For example, pipet 10 mL of each solution into a 100-mL volumetric flask, dilute to the mark with water, and mix thoroughly. Prepare a fresh solution for each determination.

14. Preparation of Calibration Curve for Ordinary Size Specimens

14.1 In order to prepare a calibration curve, make up a series of methylene blue buffer solutions containing the same amount of buffer but different amounts of methylene blue, to cover the desired range. Add 50 mL of the stock solution of buffer to each of nine 500-mL volumetric flasks. Add to these flasks 10, 15, 20, 25, 30, 35, 40, 45, and 50 mL, respectively, of the 0.002 M stock solution of methylene blue. Dilute each solution to the mark with water and mix thoroughly.

NOTE 3—The concentrations suggested for preparing calibration curves need not be followed exactly as long as enough points are obtained to allow construction of an acceptable calibration curve.

14.2 Pipet 10 mL of each solution into 100-mL volumetric flasks, add 10 mL of HCl (1 + 99), dilute to the mark with water, and mix (Note 4). Measure the absorbance of the solutions and prepare a plot of absorbance at 620 nm against concentration (Note 5).

NOTE 4—The procedure described for the colorimetric determination of methylene blue is based on the use of the Beckman DU spectrophotometer⁵ with 1-cm-absorption cells. The dilution procedure may have to be modified for use with filter photometers or for cells with a longer light path.

NOTE 5—It has been reported (5) that Beer's law is obeyed at 620 nm, and it is recommended that measurements be made at this wavelength. Measurements may also be made at 675 nm, which is close to the absorption peak, but Beer's law is not obeyed at this wavelength.

15. Preparation of Calibration Curve for Small Specimens

15.1 In order to prepare a calibration curve, pipet 1 mL of each of the nine solutions mentioned in Section 14 into 10-mL volumetric flasks, add 1 mL of HCl (1 + 99), dilute to the mark, mix, and measure the absorbance at 620 nm. If the volumetric apparatus is sufficiently precise, this calibration curve should be identical with the one described in Section 14 (Note 6). Obviously, any specific procedure that gives solutions in the right concentration range for the colorimetric measurements should be satisfactory.

⁵ Manufactured by Beckman Instruments, Inc., 2500 Harbour Blvd., Mail Station 31 D, Fullerton, CA 92634.

NOTE 6—Since methylene blue solutions have a tendency to form droplets on glassware, this can seriously impair the accuracy in transferring small volumes of solution. For this reason the 1-mL automatic pipet should be coated with silicone to make it nonwetting. The point should be completely emptied during use, and should be calibrated in the same way in which it is used. An automatic pipet is highly desirable for the precise delivery of 1 mL of solution.

The pipet should be thoroughly cleaned before coating with silicone. If electrolytes are likely to be on the surface, the pipet should be boiled in water for 30 min, dried, and then degreased by heating to 400°C for 1 h or more, or by the use of some solvent, such as carbon tetrachloride or methylene chloride. The pipet is coated with a fresh 2 weight % solution of silicone⁶ in methylene chloride, carbon tetrachloride, or perchloroethylene, allowed to drain, and air-dried or heated for 1 h at 100°C to remove the solvent. The film must then be cured by heating for 1 h at 300°C or for 2 h at 275°C.

16. Procedure for Ordinary Size Specimens

16.1 Determine the approximate carboxyl content in a preliminary experiment. Weigh out three specimens, one estimated to give 50 % exhaustion of the dye solution, one 10 to 15 % smaller, and one 10 to 15 % larger, making allowance for the moisture content. Weigh the specimens into 125-mL glass-stoppered flasks (any other convenient size flask may be used), and add 50 mL of 0.0002 *M* methylene blue-buffer solution from a pipet. Lubricate the stoppers with a little petroleum jelly and secure them with rubber bands. Place the flasks on a device that will turn them end over end or otherwise agitate the solutions.

16.2 After overnight agitation, centrifuge the solutions and pipet a 10-mL aliquot of the supernatant liquid into a 100-mL volumetric flask. Add 10 mL of HCl (1 + 99), and fill the flask to the mark with water. Measure the absorbance of the solutions at 620 nm.

16.3 Using the observed absorbances, refer to the calibration curve and read the concentration of methylene blue present for each of the three portions of specimen.

16.4 Plot the specimen size against the concentration of methylene blue in the supernatant liquid, and read from the plot the specimen size that gives 50 % exhaustion of the dye solution.

NOTE 7—It is not absolutely necessary to plot the specimen size against methylene blue concentration in order to calculate dye absorp-

⁶ Dow Corning 200 silicone solution manufactured by Dow Corning, Midland, MI, has been found satisfactory for this purpose.

tion. The dye absorption may be calculated from two slightly different weights of cellulose that will give approximately 50 % exhaustion, and the mean of the two results taken.

17. Procedure for Small Specimens

17.1 The general procedure is the same as for ordinary size specimens (Section 16). Weigh the specimens into glass-stoppered weighing bottles of about 10-mL capacity and add 5 mL of 0.0002 *M* methylene blue-buffer solution. After overnight agitation, centrifuge the solutions, remove a 1-mL aliquot with an automatic pipet, and transfer to a 10-mL volumetric flask. Add about 1 mL of HCl (1 + 99), dilute to the mark, and measure the absorbance at 620 nm. Determine the specimen size that gives 50 % exhaustion of the methylene blue solution as described in 16.4.

18. Calculations

18.1 *Ordinary Size Specimens*—The size specimen that gives 50 % exhaustion of 50 mL of 0.0002 *M* methylene blue solution has used 0.005 millimole of methylene blue in ion exchange with carboxyl groups. Therefore the millimoles of carboxyl per 100 g of cellulose, M_1 , is calculated as follows:

$$M_1 = (0.005/W) \times 100 \quad (2)$$

where W = specimen to give 50 % exhaustion of 50 mL of 0.0002 *M* methylene blue solution, g.

18.2 *Small Specimens*—The size specimen that gives 50 % exhaustion of 5 mL of 0.0002 *M* methylene blue solution has used 0.0005 millimole of methylene blue in ion exchange with carboxyl groups. Therefore the millimoles of carboxyl per 100 g of cellulose, M_2 , is calculated as follows:

$$M_2 = (0.0005/W) \times 100 \quad (3)$$

where W = specimen to give 50 % exhaustion of 5 mL of 0.0002 *M* methylene blue solution, g.

19. Report

19.1 Until more data are obtained on the precision of this test method, it is suggested that the ion-exchange capacity be reported to 0.01 meq/100 g of pulp.

20. Precision and Bias

20.1 The precision of this test method varies with the carboxyl content and from laboratory to laboratory. Precision data is available in Ref (4).

20.2 No statement of bias can be made as no suitable reference material exists for determining bias.

REFERENCES

- (1) Wilson, K., "Bestämning av Karboxylgrupper i Cellulosa," *Svensk Papperstidn*, Vol 51, 1948, p. 45.
- (2) Davidson, G. F., "The Absorption of Methylene Blue," *Journal, Textile Institute*, Vol 39, 1948, p. T65.
- (3) Jaymé, G., and Neuschäffer, K., "Über die Bestimmung des Carboxylgruppengehaltes von Zellstoffen," *Das Papier*, Vol 9, 1955, p. 143.
- (4) Wilson, W. K., and Mandel, J., "Determination of Carboxyl in Cellulose; Comparison of Various Methods, Report of TAPPI-ACSI-ICCA Subcommittee on Carboxyl," *Tappi*, Vol 44, 1961, p. 131.
- (5) Davidson, G. F., "The Determination of Methylene Blue," *Journal, Textile Institute*, Vol 38, 1947, p. T408.
- (6) Anti-Wuorinen, O., and Visapää, A., "Application of an Automatic Titration Method in the Study of the Behavior of Cellulose Towards Different Salt Solutions," *Paper and Timber*, Vol 38, 1956, p. 327.
- (7) Davidson, G. F., and Nevell, T. P., "A Comparison of Various Methods Proposed for the Determination of Carboxyl Content," *Journal, Textile Institute*, Vol 39, 1948, p. T102.

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Standard Test Method for Free Formaldehyde Content of Amino Resins¹

This standard is issued under the fixed designation D 1979; the number immediately following the designation indicates the year of original adoption 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 free formaldehyde in amino resins and their aqueous and non-aqueous solutions. Amino resin-free formaldehyde levels from about 0.02 to 5.0 % can be determined by this test method. The applicability of this test method to other matrices is unknown.

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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

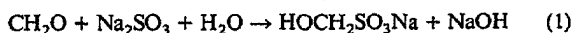
2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids³

3. Summary of Test Method

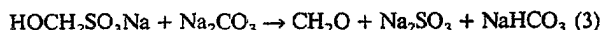
3.1 Specimens are mixed with borate buffer solution and ice water, then kept cool in an ice bath. Excess sodium sulfite is added to consume free formaldehyde as follows:



3.2 The excess sodium sulfite is removed by titration with iodine using starch as the indicator as follows:



3.3 The sodium sulfite-formaldehyde complex is then decomposed with sodium carbonate to quantitatively regenerate sodium sulfite and formaldehyde as follows:



3.4 The liberated sodium sulfite is titrated with iodine (Eq 2) and free formaldehyde⁴ is calculated from this second iodine titration.

4. Significance and Use

4.1 The amount of free formaldehyde in amino resins may be of concern to both producer and user, as its presence in air above threshold amounts may produce objectionable odors and irritant effects. This test method can be useful for evaluating suppliers' products and for quality control.

5. Apparatus

5.1 *Analytical Balance*, ± 0.1 mg.

5.2 *Magnetic Stirrer and Stir Bar*—A "heavy duty" magnetic stirrer is necessary.

5.3 *Buret*, 50 mL manual or electronic. Manual burets should be of the type designed to minimize the exposure of reagent to air. Electronic burets are preferred.

5.4 *Beakers*, glass, 100 mL, 600 mL, and 1500 mL.

5.5 *Stopwatch or Timer*.

5.6 *Graduated Cylinders*, glass, 50 mL and 250 mL.

5.7 *Glass Pipet*, 2 mL.

5.8 *Thermometer*, subdivision 1°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 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 sufficient 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 II of Specification D 1193.

6.3 *Acetic Acid Solution (1.0 M)*—Dissolve 60 mL of glacial acetic acid ($\text{CH}_3\text{CO}_2\text{H}$) in water and dilute to 1 L.

6.4 *Boric Acid Buffer Solution*—Prepare a sodium hydroxide solution (1.0 M) by dissolving 40 g of sodium hydroxide (NaOH) in water and diluting to 1 L. Dissolve 12.39 g of boric acid (H_3BO_3) in 100 mL of 1.0 M NaOH, then dilute to 1 L. Maintain temperature at 0°C in an ice bath. The pH at 0°C should be 9.4 (glass electrode).

6.5 *Ice*, finely crushed.

6.6 *Iodine Solution (0.1 N)*—Dissolve 40 g of iodate free potassium iodide in 25 mL of water in a 100-mL beaker. Weigh out 12.6905 g of dry re-sublimed iodine and add to the beaker. Stir until the iodine is dissolved and dilute to 1 L with water. Store iodine titrant in the dark. This reagent can be purchased as a standardized solution or must be standardized against sodium thiosulfate.

6.7 *Methylene Chloride (CH_2Cl_2)*—Cool to 0°C in an ice bath.

6.8 *Sodium Carbonate Solution (1.0 M)*—Dissolve 106 g of sodium carbonate (Na_2CO_3) in water 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 D01.33 on Polymers and Resins.

Current edition approved Feb. 22, 1991. Published April 1991.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

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."

6.9 *Sodium Sulfite Solution* (1.0 M)—Dissolve 126 g of sodium sulfite (Na_2SO_3) in water and dilute to 1 L. This reagent is unstable and must be prepared fresh daily.

6.10 *Starch*—0.2 % aqueous (weight/volume) or 1.0 % aqueous (weight/volume). This indicator can be purchased as stabilized solution or prepared in accordance with Test Method D 1959. Store in a cool, dark place.

6.11 *Water* (H_2O)—Cool Type II water to 0°C in an ice bath.

Procedure

7.1 A blank determination is run following this procedure, omitting 7.2 (addition of sample).

7.2 Taking the appropriate specimen size from Table 1, weigh the specimen to the nearest mg into a 600-mL beaker.

7.3 Place the beaker into the ice bath that rests upon a heavy-duty magnetic stirrer.

7.4 Place a stirring bar in the beaker and immediately dissolve the specimen as described in 7.4.1 (for aqueous specimens) or 7.4.2 (for non-aqueous specimens). Maintain the temperature of the specimen solution at 0°C by adding ice. The temperature must not rise above 2°C during subsequent steps. Note that boric acid buffer (6.4), methylene chloride (6.7) and water (6.11) should be pre-cooled to 0°C.

7.4.1 *Aqueous Specimens*—Rapidly mix the specimen with 150 mL of ice water and 25 mL of boric acid buffer solution.

7.4.2 *Non-aqueous Specimens*—Rapidly mix the specimen with 50 mL of methylene chloride cooled to 0°C. Stir to dissolve specimen. Add 150 mL of ice water and 25 mL of boric acid buffer. Continue extraction for 5 min. Uninterrupted stirring must be maintained through 7.5 to 7.10.

7.5 While constantly stirring the specimen solution, pipet 2 mL of 1.0 M sodium sulfite solution into the beaker.

7.6 Continue stirring for 15 min.

7.7 Add 10 mL of 1.0 M acetic acid solution and 10 mL of the starch solution.

7.8 Titrate the excess sodium sulfite with 0.1 N iodine solution to a green-blue color that persists for 10 s. Re-zero the buret.

7.9 Add 30 mL of 1.0 M sodium carbonate solution.

7.10 Titrate the liberated sodium sulfite with 0.1 N iodine solution to a constant blue color that persists for 1 min. The shade of blue varies with the sample under test and may appear slightly different in emulsions when compared to the blank. Record the volume of the iodine solution used in this step for use in the calculation, Section 8.

8. Calculation

8.1 Calculate the weight percent free formaldehyde in the sample as follows:

$$\text{Formaldehyde, \%} = \frac{(A - B) \times 3}{W \times 20}$$

where:

A = volume of iodine solution for specimen titration (7.10), mL,

B = volume of iodine solution for blank titration (7.10), mL, and

W = sample weight, g.

9. Precision and Bias

9.1 *Precision*—The precision statements are based upon an interlaboratory study in which one analyst in each of five different laboratories analyzed three samples of commercial melamine resins with free formaldehyde contents between 0.2 and 2.7 % in duplicate on two different days. The within-laboratory coefficient of variation was found to be 4.07 % at 12 df, and the between-laboratories coefficient of variation was found to be 13.69 % at 9 df. Based on these coefficients of variation, the following criteria should be used in judging the acceptability of results at the 95 % confidence level.

9.1.1 *Repeatability*—Two results, each the means of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 12.5 % relative.

9.1.2 *Reproducibility*—Two results, each the means of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 43.8 % relative.

9.2 *Bias*—Bias has not been established for this test method.

10. Keywords

10.1 amino resins; formaldehyde; sodium sulfite

TABLE 1 Specimen Size According to Expected Formaldehyde Content

Free Formaldehyde, weight, %	Specimen Weight, g
0.02-0.05	3.0
0.5-1.0	1.5
1.0-2.0	1.0
2.0-3.0	0.5
3.0-5.0	0.25

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Standard Test Method for Clarity and Cleanness of Paint and Ink Liquids¹

This standard is issued under the fixed designation D 2090; the number immediately following the designation indicates the year of original adoption 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 the visual examination of any unpigmented liquid for use in paints and inks, including fatty oils and acids, drier solutions, solvents, miscellaneous chemicals, varnishes, resin solutions, clear lacquers, and other clear coatings for the presence or absence of undesirable components.

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 1003 Test Method for Haze and Luminous Transmittance of Transparent Plastics²

D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems³

D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁴

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 There are various terms for clarity or cleanness of liquids, which are established as trade vernacular in describing undesirable components of a liquid. The following seven are preferred over the other terms (in bold face) related to them:

3.1.2 *foreign matter*—any visible material unrelated to the true origin of the liquid specified.

3.1.3 *sediment*—any solid which can settle or be centrifuged from the main portion of the liquid, for example, foots, meal, grain, gum.

3.1.4 *skins*—partial solid layers of material which may form, from the material itself or otherwise.

3.1.5 *turbid*—a relatively great amount of nonsettling floc, gels, suspended matter, particles, droplets, or other insoluble or separated matter, even though the liquid is translucent and transmits at least a little light.

3.1.6 *hazy*—a relatively small amount of nonsettling, finely dispersed matter which is not visibly homogeneous with the mass of the liquid specified, even though the liquid is transparent and transmits most of the light incident upon it.

3.1.7 *clear*—a complete lack of any visible nonuniformity when viewed in mass, in bottles or test tubes, by strong transmitted light.

3.1.8 *clean*—a complete lack of any visible nonuniformity sometimes referred to as seeds, when viewed in thin films by any macroscopic or microscopic use of visible light.

4. Summary of Test Method

4.1 The sample is visually examined in its original container, in the specified sample containers, and then in a film thin enough to show any nonuniformity.

5. Significance and Use

5.1 The results of the clarity and cleanness examinations are used as controls in production, and for specification acceptance of any nonpigmented liquid used in paints and inks.

6. Sampling

6.1 Sampling of one or more containers of a liquid is especially important for the validity of a clarity or cleanness test, and each type of container, such as tank car, tanktruck, drum, carboy, etc., requires its own detailed sampling procedure. Temperature conditions and periods affect amounts of solidified matter which may form, or volatilized matter lost, such as phosphatides, waxes, or high melting acids solidified from fatty oils or acids, or low boiling solvents volatilized from varnishes, resin solutions, etc. Therefore, the precise mechanics, the date, time and temperature of sampling, the type of sample container and the temperature of the container, light, and any other critical sample storage conditions shall be specified.

7. Conditioning Sample

7.1 Because limits may be desired on the amounts of gums or other solids which will separate from a liquid very slowly at various temperatures, specify limits of a time and temperature schedule for conditioning the specimen and for making the examination. When not stated otherwise, the sample is presumed to have been stable for any period of time and observed at 77°F (25°C).

8. Procedure

8.1 Examine all parts of the sample and its container under at least 50 ft-candles (53.8 lx) of light for any nonuniformity.

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 01.33 on Polymers and Resins.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 2090 - 62 T. Last previous edition D 2090 - 70(1981)¹. Discontinued under 3-year tentative rule 1969. Reinstated and adopted as standard in 1970.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 06.01.

⁴ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

8.2 Transfer some of the sample to fill a clean Gardner-Ridd tube as specified in Test Method D 1545 to leave an air bubble under a clean stopper. Tilt the tube at a small angle from the horizontal so that the air bubble will move slowly and permit observation in the moving liquid of any particles that may produce a haze in the specimen.

8.3 Drain the tube of 80 to 90 % of its contents, replace the stopper, let stand vertically for 15 min or other time specified to allow a highly viscous specimen to complete its flow to the bottom, while leaving a very thin film of the specimen over the upper walls of the tube, and while still protected from extraneous dust and from evaporation. Examine the drained, vertical tube by both transmitted and reflected strong light to detect particles of any sort in the thin film. A liquid may appear *clear* in mass, yet not *clean* in a thin film.

NOTE—Many variables influence the choice of details of specimen conditioning and examination, such as:

Highly colored bitumen solutions, driers, varnishes, etc., that nevertheless may be judged to be clear and clean when examined in a very thin film.

Relative pure fatty acids may have narrow melting ranges of temperature, and their cleanness is easily observed at a temperature of 77°F (27.8°C) above their melting point, whereas gross or dark mixtures of fatty acids may have such a wide range of melting temperature that specification of time and temperature for specimen conditioning and for passing a clean test may constitute a convenient, proximate method of limiting their composition.

Glyceride oils may contain small amounts of fatty acids, phosphatides, waxes, and high melting glycerides, for example, stearin in fish oil; specification of time, temperature, and air and moisture exposure for specimen conditioning and for passing a clean test may

constitute a convenient, proximate method of limiting their composition.

Solutions of varnishes, resins, driers, soaps or polymerized oils may contain tiny gel particles which spoil the appearance of high gloss paints and enamels made from them. The drained tube technique is particularly useful in detecting these particles when their index of refraction makes them difficult to detect in mass.

Test Method D 1210, may also be used for semiquantitative determination of particle size and frequency in unpigmented liquids. However, the films prepared by either Test Method D 1210 or by simple drainage on clean, vertical glass plates are more susceptible to contamination by extraneous matter during the course of the test.

9. Report

9.1 Report the following information:

9.1.1 Name of original or specimen container examined and any significant details of sampling procedure,

9.1.2 Temperature range and time period of specimen conditioning.

9.1.3 Temperature of specimen at time of observation, and

9.1.4 Term or terms, selected from Section 3, which describe the clarity or cleanness of the specimen.

10. Precision

10.1 No numerical statement of precision is possible in this qualitative method.

11. Index Terms

11.1 This test method is indexed under the following terms:

foreign matter content; sediment; skins content.

APPENDIX

(Nonmandatory Information)

X1. INSTRUMENTAL DETERMINATION

This instrumental test method is offered for comment, and without cooperative testing, because it utilizes an existing ASTM test method to fill a recognized need. It is published as information only and offered without prejudice against other test methods.

X1.1 Summary of Test Method

X1.1.1 A specimen of liquid is placed in a curette and a parallel beam of light shining through it is measured at the normal (sere) angle of emergence and also at an angle 10° from normal. The ratio of intensities of the emergent beams is a measure of clarity.

X1.2 Procedure

X1.2.1 Test the specimen in accordance with Procedure A of Test Method D 1003. Use the Hazemeter, but in place of

the solid specimen of plastic specified therein, pour the thoroughly mixed specimen into a 10-mm glass cell with parallel walls and place it in the specified positions on the integration sphere.

X1.3 Calculation

X1.3.1 Calculate percent haze H as follows:

$$H = (T_d/T_t) \times 100$$

where:

T_d = diffuse transmittance, and
 T_t = total transmittance.

REFERENCES

- (1) Nimeroff, I., "Status of ASTM Methods and Standards for Appearance Valuation," Symposium on Visual Aids for Standardizing and Communicating Product Appearance, STP 258, Am. Soc. Testing Mats. 1959.
- (2) Billymer, Jr., F. W., "Measurement of Optical Clarity by Low-Angle Light Scattering," *Journal, Optical Soc. Am.*, Vol. 49, April 1959, pp. 368-371, a method for measuring scattered light photoelectrically at a 10° angle, with an angular resolution of 1°. It is suitable for plant control applications.
- (3) Rieger, C. J., and Carpenter, F. G., "Light Scattering by Commercial Sugar Solution," *Journal of Research, Nat. Bureau Standards* Vol 63A, November 1959, pp. 205-211.
- (4) Method D 871, sections on determination of haze by comparison with arbitrary standards based on aqueous suspensions of fuller's earth; when balloted in Committee D-23 on Cellulose and Cellulose Derivatives, and adopted by the Society.
- (5) Coleman Nephelometer method used by L. V. Anderson in ASTM Committee D-1, Subcommittee II, Group II, on Gum Determinations in Linseed Oil.

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Standard Specification for Molybdate Orange Pigments¹

This standard is issued under the fixed designation D 2218; the number immediately following the designation indicates the year of original adoption 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 7133 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 This specification covers the pigment known as molybdate orange.

1.2 The following hazard caveat applies to the test method portion of this specification only. *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 126 Test Methods for Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate and Chromium Oxide Green²

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints^{2,3}

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)⁴

D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²

D 523 Test Method for Specular Gloss³

D 600 Specification for Liquid Paint Driers⁴

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 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems³

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵

2.2 Federal Specification:⁶

TT-R-266 Resin, Alkyd; Solutions

3. Composition and Properties

3.1 *Dry Pigment*—The pigment shall be a product made by the chemical coprecipitation of lead chromate and lead molybdate, with or without admixtures of other insoluble compounds of lead or other materials used in manufacture to control certain properties. The pigment shall conform to the requirements for chemical composition as prescribed in Table 1.

3.2 The mass color and character of the tint formed by a mixture with a white pigment shall be the same as, and the strength shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller.

3.3 When mutually agreed upon between the purchaser and the seller as being essential to the end use of the pigment, resistance to loss of gloss, chalking, and color change shall be tested as specified in 5.1.6 The exposed panel shall show no chalking, a loss of not more than 10 % of the original gloss, and a color change difference of not more than three units.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb (4540 kg), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or samples from the same production unit may be blended in equal quantities to form a composite sample.

TABLE 1 Requirements for Chemical Composition

	%	
	Min	Max
Chromium (calculated as PbCrO ₄)	70	...
Molybdenum (calculated as PbMoO ₄)	8	...
Total of all substances (including moisture and water soluble compounds) other than insoluble compounds of lead	...	12
Moisture and other volatile matter	...	1.5
Coarse particles (total residue retained on a 45- μ m (No. 325) sieve	...	1.0
Matter soluble in water	...	1.0

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 D01.31 on Pigment Specifications.

Current edition accepted Sept. 8, 1967. Originally issued 1963. Replaces D 2218 - 63 T.

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, Vols 06.01 and 14.02.

⁶ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods:

5.1.1 *Chemical Analysis of Lead Chromate*—Proceed in accordance with the Lead Chromate section of Test Methods D 126. The alternative procedure is not applicable.

5.1.2 *Chemical Analysis of Lead Molybdate and Total Molybdenum*—Weigh to 1 mg about 1-g sample of pigment into a 250-mL beaker. Add 20 mL of sulfuric acid (H_2SO_4 , 1+1) and heat to light fumes. Cool the solution, dilute to 150 mL, filter, and wash with water. To reduce chromate, add freshly prepared sulfurous acid (H_2SO_3) until the solution turns green, and then add several millilitres in excess. Boil the solution until excess H_2SO_3 is removed. Cool the solution to 5 to 10°C, and add 30 mL of a 2 % solution of alphasbenzoinoxime in alcohol slowly, with stirring. Add sufficient bromine water to tint the solution a pale yellow, and then add a few more millilitres of the alphasbenzoinoxime reagent. After 15 min at 5 to 10°C, filter the solution through very thin, medium paper. Then wash the solution with cold H_2SO_4 (1+100) containing 25 to 50 mL of prepared alphasbenzoinoxime reagent per litre. Place the precipitate in a platinum crucible, char cautiously, then ignite to constant weight at 500°C, and weigh as molybdenum oxide (MoO_3). Calculate weight percent lead molybdate ($PbMoO_4$), M , as follows:

$$M = (A \times 255)/S$$

where:

A = weight of MoO_3 obtained, g, and
 S = specimen weight, g.

5.1.3 *Coarse Particles*—Test Methods D 185.

5.1.4 *Mass Color and Tinting Strength*—Test Method D 387.

5.1.5 *Moisture and Other Volatile Matter*—Test Methods D 126.

5.1.6 *Resistance to Loss of Gloss, Chalking, and Color Change*.

5.1.6.1 Prepare a test enamel consisting of ingredients conforming to the applicable specifications in the following proportions:

Ingredient	Weight %
Molybdate orange sample	29.51
Alkyd resin solution ^a	61.72
Mineral spirits ^b	8.37
Lead naphthenate ^c	0.25
Cobalt naphthenate ^c	0.15
Total	100.0

^a Conforming to Type III of U.S. Federal Specification TT-R-266.

^b See Specification D 235.

^c See Class B of Specification D 600.

5.1.6.2 Give the enamel three passes through a moderately tight roller mill setting to give a fineness of grind, as determined following Test Method D 1210, of 1.5 mils (40 μ m) or less. Apply the enamel to duplicate flat metal panels by spray or applicator to complete hiding, and allow to dry 72 h. Measure the gloss at 60° in accordance with Test Method D 523. Measure the directional reflectance of the coating in accordance with Test Method E 97. Subject the coated panels for 168 h to accelerated weathering under the conditions prescribed in Practice D 822. Examine the exposed coating for chalking. Wash the exposed panel under running water with a thoroughly degreased lamb's wool pad to remove scum or dirt. Wipe off the water with clean cheesecloth and dry the panel for 2 h. Calculate the loss of gloss from gloss measurements made before and after exposure. After exposure, determine the directional reflectance for each panel as described in Test Method E 97. Estimate the color change or lightness-difference estimate (ΔL) as follows:

$$\Delta L = K(Y_2^{1/2} - Y_1^{1/2})$$

where:

Y_1 = luminous directional reflectance of the panel measured before exposure,

Y_2 = reflectance measured after exposure, and

K = 100 when the reflectances are expressed in decimal fractions or 10 if reflectances are expressed in percent.

The color change for the coating shall be the mean obtained for the two panels.⁷

⁷ The method of determining the lightness-difference estimate is described in detail in example 4, "Photoelectric Tristimulus Colorimetry with Three Filters," Circular C429, NBSA, Nat. Bureau Standards.

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Standard Test Method for Antimony Oxide in White Pigment Separated From Solvent-Reducible Paints¹

This standard is issued under the fixed designation D 2350; the number immediately following the designation indicates the year of original adoption 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 7016 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been approved by the Department of Defense.

Scope

1.1 This test method covers the determination of the total antimony oxide in white pigment separated from solvent-reducible paints.

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 1193 Specification for Reagent Water²

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints³

3. Summary of test Method

3.1 The pigment is dissolved in hydrochloric acid (HCl). Sulfuric acid (H_2SO_4) is added. The mixture is titrated with potassium permanganate ($KMnO_4$) and calculated to antimony oxide (Sb_2O_3) which gives antimony in the *ous* condition.

3.2 The pigment is dissolved in H_2SO_4 with potassium sulfate (K_2SO_4) and reduced. Sodium sulfite (Na_2SO_3) is added and sulfur dioxide gas (SO_2) is expelled. The solution is diluted and HCl added after which the solution is titrated with $KMnO_4$, which gives total antimony calculated to Sb_2O_3 .

3.3 Sb_2O_3 from *ous* condition is subtracted from total Sb_2O_3 and residual Sb_2O_3 is calculated to Sb_2O_5 .

3.4 The procedure is also described for antimony oxide in presence of large amounts of iron.

4. Significance and Use

4.1 Antimony trioxide is often used in fire-retardant paints, so it is useful to formulators and users to be able to monitor the amount of this compound in whole paints.

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.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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent grade water conforming to Specification D 1193.

5.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated HCl.

5.4 *Hydrogen Sulfide* (H_2S).

5.5 *Potassium Permanganate, Standard Solution* (0.1 N)—Dissolve 3.2 g of pure potassium permanganate ($KMnO_4$) in 1 L of water, let stand 8 to 14 days, and siphon off the clear solution (or filter through a glass filter). For use in determining antimony, the $KMnO_4$ solution is best standardized as follows: To 0.25 g of pure metallic antimony in a 500-mL resistant-glass Erlenmeyer flask, add 12 to 15 mL of H_2SO_4 (sp gr 1.84) and 10 to 12 g of K_2SO_4 . Heat until all the antimony is dissolved, cool, dilute to 100 mL with water, add 1 to 2 g of sodium sulfite (Na_2SO_3), and boil until all the SO_2 is expelled. This expulsion is evident when no blue color is obtained with starch-iodate paper (5.7). The volume will be reduced about one half. Dilute to 250 mL with water, add 20 mL of HCl (sp gr 1.19) and titrate to a faint pink tint with 0.1 N $KMnO_4$ solution.

NOTE 1—For normal routine control work, the reduction with sodium sulfite may be eliminated. The material may be diluted to 250 mL, 20 mL of HCl added, and titrated immediately after cooling the sulfuric acid-potassium sulfate digestion.

5.6 *Potassium Sulfate* (K_2SO_4).

5.7 *Starch-Iodate Paper*—Impregnate filter paper with a solution obtained by heating 2 g of starch with 100 mL of water, and, after solution, adding 0.2 g of potassium iodate (KIO_3) dissolved in 5 mL of water.

5.8 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

5.9 *Tartaric Acid*.

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.

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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. Preparation of Sample

6.1 Separate and prepare the pigment in accordance with Test Method D 2371.

7. Procedure

7.1 Transfer 0.3 g of a straight Sb_2O_3 pigment, or 0.5 g of a mixed pigment, to a 500-mL resistant-glass Erlenmeyer flask, add 15 mL of water and 25 mL of HCl (sp gr 1.19). Cover with a watch glass, warm on the steam bath for 10 to 15 min to dissolve the antimony oxide, wash off the cover, add 250 mL of water, and 15 mL of H_2SO_4 (sp gr 1.84). Boil 2 min, cool to 10 to 15°C, and titrate to a faint pink tint with 0.1 N KMnO_4 solution. Calculate to Sb_2O_3 .

7.2 The procedure described in 7.1 gives only the antimony in the *ous* conditions. The following method gives the total antimony (*ous* and *ic* forms): Transfer 0.3 g of a straight Sb_2O_3 pigment, or 0.5 g of a mixed pigment, to a 500-mL resistant-glass Erlenmeyer flask, and add 15 mL of H_2SO_4 (sp gr 1.84), 10 g of K_2SO_4 , and a 9-cm filter paper (to furnish carbon to act as a reducing agent). Place a funnel in the neck of the flask and heat until the solution becomes colorless. Cool, wash off the funnel, dilute to 100 mL with water, add 1 to 2 g of Na_2SO_3 , and boil until all the SO_2 is expelled. This expulsion is evident when no blue color is obtained with starch-iodate paper (5.7). The volume will be reduced about one half. Dilute to 250 mL with water, add 20 mL of HCl (sp gr 1.19), and titrate to a faint pink tint with 0.1 N KMnO_4 solution (Note 1).

7.2.1 Calculate total antimony to Sb_2O_3 . Subtract the Sb_2O_3 found using the procedure given in 7.1 from the total Sb_2O_3 and calculate the residual Sb_2O_3 to Sb_2O_5 (multiply by the factor 1.1098).

7.3 Procedure in Presence of Appreciable Amounts of Iron:

7.3.1 Treat 1 g of the mixed pigment, or 0.3 g of a tinted Sb_2O_3 pigment, in a covered 250-mL beaker with 5 mL of water and 20 mL of HCl (sp gr 1.19). Heat on the steam bath

for 15 min, cool, wash off the cover, add 3 g of tartaric acid and 100 mL of hot water, and digest a few minutes. Filter, catching the filtrate in a 500-mL resistant-glass Erlenmeyer flask. Wash thoroughly with hot water, dilute to 300 mL with hot water, and pass in H_2S until the precipitation is complete. (If the sample contains no insoluble matter, dissolve directly in a 500-mL resistant-glass Erlenmeyer flask, add tartaric acid, dilute, and pass in H_2S .)

7.3.2 Filter, wash with water containing H_2S until free from HCl , return paper and precipitate to the Erlenmeyer flask, add 15 mL of H_2SO_4 (sp gr 1.84) and 10 g of K_2SO_4 , place a funnel in the neck of the flask, and heat until the solution is colorless. Cool, wash off the funnel, dilute to 100 mL with water, add 1 to 2 g of sodium sulfite (Na_2SO_3), and boil until all the SO_2 is expelled. This expulsion is evident when no blue color is obtained with starch-iodate paper (5.7). The volume will be reduced about one-half. Dilute to 250 mL with water, add 20 mL of HCl (sp gr 1.19), and titrate to a faint pink tint with 0.1 N KMnO_4 solution (Note 1).

8. Calculation

8.1 Calculate the percent antimony, B , as Sb_2O_3 as follows:

$$B = \frac{AV}{S} \times 100$$

where:

A = Sb_2O_3 equivalent of the KMnO_4 solution, g/mL,

V = KMnO_4 solution required, mL, and

S = sample used, g.

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.

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Standard Test Method for Sulfide in White Pigment Separated from Solvent-Reducible Paints¹

This standard is issued under the fixed designation D 2351; the number immediately following the designation indicates the year of original adoption 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 sulfide sulfur in white pigment separated from solvent-reducible paints.
- 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.*

Referenced Documents

- 2.1 *ASTM Standards:*
 - D 215 Test Methods for Chemical Analysis of White Linseed Oil Paints²
 - D 1193 Specification for Reagent Water³
 - D 2371 Test Method for Pigment Content of Solvent-Reducible Paints²

3. Summary of Test Method

3.1 The extracted pigment is placed in a flask with mossy zinc and the sulfide generated by addition of HCl reacts with lead nitrate in an absorption flask forming lead sulfide. The lead sulfide is dissolved with nitric acid (HNO_3) and the lead determined as lead sulfate in accordance with Test Methods D 215.

3.2 A rapid method is also described.

4. Significance and Use

4.1 Sulfide containing pigments such as lithopone have been used in paints in varying degrees in the past years. As such it is useful to formulators and users to be able to monitor the amount of this compound in whole paints.

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 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 grade water conforming to Type II of Specification D 1193.

5.3 *Ammoniacal Cadmium Chloride or Zinc Sulfate Solution*—Dissolve 8 g of cadmium chloride ($\text{CdCl}_2 \cdot 2\text{H}_2\text{O}$) in 200 mL of water and add 200 mL of ammonium hydroxide (NH_4OH , sp gr 0.90); or, dissolve 200 g of zinc sulfate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in 1080 mL of water and 920 mL of NH_4OH (sp gr 0.90).

5.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated HCl.

5.5 *Lead Nitrate, Alkaline Solution*—Into 100 mL of potassium hydroxide (KOH) solution (56 g in 140 mL of water) pour a saturated solution of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) (250 g in 500 mL of water) until the precipitate ceases to redissolve, stirring constantly while mixing. Let settle, filter through a glass filter, and dilute the clear filtrate with an equal volume of water. About 3 volumes of the $\text{Pb}(\text{NO}_3)_2$ solution will be required for 1 volume of the KOH solution.

5.6 *Mossy Zinc*.

5.7 *Nitric Acid* (1+4)—Mix 1 volume of concentrated HNO_3 , (sp gr 1.42) with 4 volumes of water.

5.8 *Potassium Iodate, Standard Solution*—Dissolve 3.6 g of potassium iodate (KIO_3) and 39 g of potassium iodide (KI) in 1 L of water. For general work the theoretical sulfur titer of this solution should be used; for special work the solution may be standardized against similar material, such as a lithopone of known sulfide sulfur content. The theoretical titer is based on standard sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) and is obtained as follows: To 300 mL of water in a 600-mL flask, preferably glass-stoppered, add 10 mL of HCl (sp gr 1.19) and 1 g of KI. Cool and add 10 mL of 0.1 N potassium permanganate (KMnO_4) solution which has been standardized against $\text{Na}_2\text{C}_2\text{O}_4$. Swirl gently, stopper, and let stand for 5 min. Titrate the liberated iodine with standard sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution until the color fades. Then add 10 mL of starch solution and continue the titration until the blue color is destroyed. Repeat the titration, except substitute 10 mL of the KIO_3 for the KMnO_4 solution. Calculate the normality of the KIO_3 solution 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 D01.21 on Chemical Analysis of Paints and Paint Materials.

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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."

5.8.1 Standardization Calculation for Theoretical Sulfur Titer:

$$B = \frac{V_1 \times N}{V_2} \quad (1)$$

where:

B = normality of KIO_3 ,
 V_1 = standard $\text{Na}_2\text{S}_2\text{O}_3$ solution, mL, required to titrate 10 mL of KMnO_4 solution,
 N = normality of standardized KMnO_4 solution, and
 V_2 = standard $\text{Na}_2\text{S}_2\text{O}_3$ solution, mL, required to titrate 10 mL of KIO_3 solution.

5.8.2 Standardization Against Known Pigment:

$$C = (A \times S)/(V \times 100) \quad (2)$$

where:

A = sulfur in known pigment, %
 C = sulfide equivalent of the KIO_3 solution, g/mL,
 S = pigment, g, and
 V = KIO_3 solution required to titrate known pigment, mL.

5.9 Starch Indicator (for Sulfur Titration)—To 1 L of boiling water add a cold suspension of 6 g of starch in 100 mL of water and boil vigorously for 5 min. Cool the solution, add 6 g of zinc chloride (ZnCl_2) dissolved in 50 mL of cold water, thoroughly mix, and set aside for 24 h. Decant the clear supernatant liquid into a suitable container, add 3 g of KI, and mix thoroughly.

5.9.1 (Optional) Prepare an emulsion of 6 g of soluble starch in 25 mL of water, add a solution of 1 g of sodium hydroxide (NaOH) in 10 mL of water, and stir the solution until it gels. Dilute to 1 L with water, add 3 g of KI, and mix thoroughly.

6. Preparation of Sample

6.1 Separate and prepare the pigment for this determination in accordance with Test Method D 2371.

7. Procedure

7.1 Place 0.5 to 1 g of the pigment in a flask with about 10 g of "feathered" or mossy zinc and add 50 mL of water; insert a stopper carrying a separatory funnel and an exit tube. Run in 50 mL of HCl (sp gr 1.19) from the funnel, having previously connected the exit tube to two absorption flasks in

series; the first flask containing 100 mL of alkaline lead nitrate solution, the second flask, 50 mL of the same solution as a safety device. After all of the acid has run into the evolution flask, heat slowly, finally boiling until the first appearance of steam in the first absorption flask.

7.2 Disconnect, let the lead sulfide (PbS) settle, filter, and wash with cold water, then with hot water until neutral to litmus paper and until the washings give no test for lead. Dissolve the PbS precipitate in hot HNO_3 (1+4) and determine the lead as lead sulfate (PbSO_4) in accordance with Test Method D 215.

7.3 For very rapid work, the evolved hydrogen sulfide (H_2S) may be absorbed in an ammoniacal CdCl_2 or ZnSO_4 solution (5.3) contained in two flasks connected in series, the contents of the absorption flasks washed into a vessel with cold water and diluted to about 1 L, acidified with HCl (sp gr 1.19), and titrated with standard KIO_3 solution using starch indicator (5.9).

8. Calculation

8.1 Calculate the percent of sulfide sulfur, E , as follows:

NOTE—The percent of sulfide sulfur can be calculated from the percent of total zinc and zinc soluble in acetic acid (2 to 3 %), assuming the sulfide to be zinc sulfide (ZnS). See section on Total Zinc of Test Method D 215.

$$E = [(P \times 0.1054)/S] \times 10C \quad (3)$$

where:

P = PbSO_4 formed, g, and
 S = sample used, g.

$$E = (AV/S) \times 100 \quad (4)$$

where:

A = sulfide equivalent of the KIO_3 solution, g/mL,
 V = KIO_3 solution required for titration of specimen, mL, and
 S = sample used, g.

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.

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Standard Test Method for Sulfur Dioxide in White Pigment Separated from Solvent- Reducible Paints¹

This standard is issued under the fixed designation D 2352; the number immediately following the designation indicates the year of original adoption 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 10 was added editorially in May 1990.

1. Scope

1.1 This test method covers the determination of sulfur dioxide in white pigment separated from solvent-reducible paints.

1.2 This test method is not applicable in the presence of sulfides decomposable under the conditions given.

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 1193 Specification for Reagent Water²

D 2371 Test Method for Pigment Content of Solvent-Reducible Paints³

3. Summary of Test Method

3.1 The extracted pigment is placed in a flask and readily available sulfur dioxide (SO₂) is released by addition of hydrochloric acid (HCl) and bubbled through an absorption flask containing iodine solution and potassium iodide (KI). The solution is titrated with sodium thiosulfate (Na₂S₂O₃) using starch indicator.

3.2 A blank is run as directed in 3.1 with the pigment omitted. The blank is then subtracted from the figure obtained for the sample titration and SO₂ is calculated.

4. Significance and Use

4.1 Calcium sulfate pigments such as titanium-calcium have been used in paints in varying degrees in the past years. As such it is useful to the formulator and the user to be able to monitor the amount of this compound in whole paints.

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.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.

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

5.3 *Hydrochloric Acid (1+3)*—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 3 volumes of water.

5.4 *Iodine, Standard Solution (0.05 N) (for SO₂)*—Place 15 to 20 g of pure potassium iodide (KI) in a 1-L flask, dissolve in as little water as possible, and then add about 6.4 g of resublimed iodine. Shake until the iodine is all dissolved, dilute to the mark with water, and mix. Standardize the solution against 0.05 N Na₂S₂O₃ solution to obtain its true normality.

5.5 *Potassium Iodide Solution (100 g/L)*—Dissolve 100 g of potassium iodide (KI) in water and dilute to 1 L.

5.6 *Sodium Thiosulfate, Standard (0.05 N) Solution (for SO₂)*—Dissolve pure sodium thiosulfate Na₂S₂O₃ in water (that has been well boiled to free it from carbon dioxide) in the proportion of 12.42 g of Na₂S₂O₃ · 5H₂O to 1 L of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize⁵ with pure resublimed iodine, pure potassium biiodate, or pure potassium iodate. This solution will be approximately 0.05 N, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly 0.05 N. Preserve in a stock bottle provided with a guard tube filled with soda lime.

5.7 *Starch Indicator Solution*—Make a homogenous paste of 10 g of soluble starch in cold water. Add to this 1 L of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/L) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to 10°C (40 to 50°F). Prepare fresh indicator when the 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 D01.21 on Chemical Analysis of Paints and Paint Materials.

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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."

⁵ Buehrer, T. F., and Mason, C. M., "Thiosulfate, Standardization of," *Analytical Chemistry*, Vol 2, 1930, p. 138.

point of the titration from blue to colorless or blue to light green fails to be sharp.

6. Preparation of Sample

6.1 Separate and prepare the pigment for this determination in accordance with Test Method D 2371.

7. Procedure

7.1 Transfer 10 g of the pigment to a suitable flask, insert a stopper fitted with a separatory funnel and a spray trap delivery tube (Note 1), and attach the latter to a condenser. Place about 150 mL of HCl (1+3) in the funnel, the stopcock being closed (Note 2), and connect the other end of the condenser with a delivery tube that passes through a two-hole stopper and extends nearly to the bottom of an absorption flask; through the other hole of the stopper connect a tube or flask to serve as a safety device. Place 25 mL of 0.05 N iodine solution (5.4) in the absorption flask (dilute with water if necessary) and 20 mL of KI solution (100 g/L) in the safety tube; fit the stopper in the absorption flask. Open the stopcock and allow the acid to slowly enter the flask. Before all of the acid is admitted, force air (washed with sodium hydroxide (NaOH) solution) through the top of the separatory funnel (about 2 bubbles per second in the KI solution). Boil the solution 3 min with air passing through then remove the source of heat and pass air through for 30 min.

NOTE 1—A Knorr CO₂ apparatus is very convenient. In this case, the vertical condenser may be connected with an absorption tower containing the iodine solution, followed by the KI solution in a suitable tube.

NOTE 2—To minimize, if not eliminate, any possible oxidation by the air, add about 1 g (in one piece) of sodium bicarbonate (NaHCO₃) to

the evolution flask, then add the acid directly to the flask, omitting the separatory funnel and the current of air. Boil the solution until about 50 mL of distillate has passed over.

7.2 Disconnect the absorption vessels, wash the KI solution into the iodine solution, and titrate at once with 0.05 N Na₂S₂O₃ solution using starch indicator. Run a blank determination in exactly the same manner except for the omission of the pigment. Subtract the figure obtained for the blank determination from the figure obtained for titration of the sample and calculate the final result to SO₂ (1 mL 0.05 N iodine = 0.0016 g SO₂).

8. Calculation

8.1 Calculate the percent of sulfur dioxide, *A*, as follows:

$$A = [(V - B)I/S] \times 100$$

where:

V = 0.05 N iodine solution required for titration of specimen, mL,

B = 0.05 N iodine solution required for titration of the blank, mL,

I = SO₂ equivalent of the 0.05 N iodine solution, g/mL, and

S = sample used, g.

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 sulfate analysis; sulfur dioxide; white pigment

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 Minimum Film Formation Temperature (MFFT) of Emulsion Vehicles¹

This standard is issued under the fixed designation D 2354; the number immediately following the designation indicates the 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 minimum temperature at which emulsion vehicles coalesce to form continuous films. The term "emulsion" in this test method includes latex vehicles.

1.2 This test method is limited to emulsions having minimum film formation temperatures below 25°C.

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. Summary of Test Method

2.1 In this test method the minimum film formation temperature (MFFT) is determined by visual observation of cracking or whitening in films that have dried over a substrate having a controlled temperature gradient.

3. Significance and Use

3.1 The satisfactory film integrity of emulsion coatings requires that as the aqueous phase evaporates the resinous portion of the vehicle coalesces into a continuous film. Low temperature impairs the fluidity of the resin particle and thereby their ability to coalesce. Vehicles that can withstand low temperatures are therefore required, and MFFT is an important quality feature of emulsion vehicles.

4. Apparatus

4.1 **Minimum Film Formation Temperature Bar**—An MFFT unit² such as is illustrated in Fig. 1. The bar represented in Fig. 1 is designed to be used with thermocouples. The thermocouple leads used with the assembly are iron-constantan with an external resistance of 10 Ω . Also included is a pyrometer (not shown in Fig. 1).

NOTE 1—Thermometers can be used as an alternative way of measuring temperature.³ It is noted that larger holes ($\frac{1}{32}$ in.) (4 mm) will have to be drilled in the bar to accommodate the thermometers. If

thermometers are selected, all holes are drilled on the same side of the bar, as opposed to the system of alternate placement of holes when using thermocouples.

NOTE 2—A self-contained unit eliminating the need for external cooling and requiring only an electrical outlet, piped water and an air or nitrogen supply has been developed by ICI Paints Division.⁴

4.2 **Dry Air or Dry Nitrogen Supply**, with a regulating valve and flow meter.

4.3 **Film Caster**, having a 6-mil (150- μ m) clearance.

5. Reagents and Materials

5.1 **Dry Ice**.

5.2 **Isopropanol**, anhydrous.

6. Procedure

6.1 Set up the MFFT bar in a level position with the lower temperature leg of the bar immersed in a dry ice-isopropanol bath. It is important that the surface of the bar be smooth and plane to ensure uniform film thicknesses. Use a $\frac{1}{2}$ -gal (2-L) container for this purpose. (The container can be insulated with any suitable material to reduce heat loss.) Adjust the height of the dry ice-isopropanol bath as needed to bring the temperature of the bar to approximately 0°C at the lowest temperature-measuring stage of the unit. Lower or higher temperatures can be obtained, if needed, by adjusting the bath height. The bath height needed to obtain 0°C will vary depending on the heat conductivity of the metal used in the construction of the bar. Expose the higher temperature end of the bar at approximately 25°C by either conducting the test in a constant-temperature room at this temperature or by thermostating the leg.

6.2 Immediately after imposing the temperature gradient on the bar, place the cover over it and start the flow of dry air (or nitrogen) at a rate of 3000 cm³/min from the low-temperature end to the high-temperature end of the bar, controlling the rate by a simple flow meter. Allowing the air to retain moisture by warming up prevents water condensation at the cold temperature end that would interfere with film drying. Approximately 1 to 1½ h is needed for the temperature gradient to reach equilibrium once the bar is placed in operation. The air manifold holes must be clean and free of frost before starting a test.

NOTE 3—A mixture of ethylene glycol and surfactant applied to the air outlet alleviates the frosting condition temporarily.

6.3 Once a constant-temperature gradient is established,

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.

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² A unit manufactured by Custom Scientific Instruments Inc., 13 Wing Dr., Cedar Knolls, NJ 07927, has been found suitable for this purpose.

³ An example of such a thermometer is Prince No. A-011044, 90° angle, red-reading, range from -5 to +50°C, having an accuracy of $\pm 0.5^\circ\text{C}$ and manufactured by Prince Instruments Inc., 1020 Industrial Hwy., Southampton, PA 18966.

⁴ The unit identified as an MFFTBAR, Model SS-3000, manufactured by Sheen Instruments, Ltd., 8 Waldegrave Rd., Teddington, Middlesex TW11 8LD, England has been found suitable for this purpose.



FIG. 1 Minimum Film Formation Temperature Bar

remove the cover and cast the films using the 6-mil applicator (Note 3). Cast films from the low to the high-temperature end of the bar because the extension at the high-temperature end is designed to permit overflow to fall into a container below the bar. After the film is cast and the cover put in place, approximately 1 to 2 h are required for the film to dry. Check the air flow and isopropanol bath height before any tests are started and then maintain them at their respective levels until the test is completed.

NOTE 4—If desired, a 1/2-mil (13- μ m) polyethylene terephthalate sheet of 5-in. (125-mm) width can be placed on the bar surface before casting the film. Tests show that there are no differences between minimum filming temperatures determined with and without the sheet. The polyethylene terephthalate film permits not only easier clean-up of the apparatus, but also a semi-permanent record of the test. Special care must be exercised to ensure good contact of the sheet with the bar. Accomplish this by either of the following techniques:

- (1) Wipe the sheet with a dry cloth to develop a static charge before placement on the bar.
- (2) Coat the sheet with silicone oil before placement on the bar.

6.4 After the film has dried, observe for discontinuity as evidenced by whitening or cracking or both. Record the lowest temperature on the bar at which the film is still continuous as the minimum film formation temperature.

7. Report

- 7.1 Report the following information:
- 7.1.1 MFFT in degrees Celsius, and
- 7.1.2 Description of the discontinuity indicating degree of whitening and cracking.

8. Precision and Bias

8.1 *Precision*—Information for a formal precision statement has not yet been obtained, but it is reported that MFFT values obtained by this test method can be expected to fall within $\pm 2^{\circ}\text{C}$ of their mean.

8.2 Bias—Bias has not yet been determined.

9. Keywords

- 9.1 emulsion vehicles; film formation; minimum film formation temperature, MFFT

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Standard Test Methods for Hydroxypropyl Methylcellulose¹

This standard is issued under the fixed designation D 2363; the number immediately following the designation indicates the year of original adoption 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 1989.

Scope

1.1 These test methods cover the testing of hydroxypropyl methylcellulose.

1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 6
Ash (as Sulfate)	7 to 10
Chlorides (as NaCl)	11 to 14
Alkalinity (as Na_2CO_3)	15 to 18
Iron	19 to 24
Heavy Metals	25 to 29
Methoxyl Content	30 to 35
Hydroxypropoxyl Content	36 to 41
Viscosity	42 to 46
pH	47
Solids	48 to 51
Density	52 to 56

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 Note 1.

Referenced Documents

2.1 ASTM Standards:

D96 Test Methods for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)²

E 70 Test Method for pH of Aqueous Solutions With the Glass Electrode³

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.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.

3.2 Unless otherwise indicated, references to water shall be understood to mean distilled water.

MOISTURE

4. Scope

4.1 This test method covers the determination of the volatile content of hydroxypropyl methylcellulose and, by common usage, designated moisture.

5. Procedure

5.1 Transfer 2 to 5 g of the sample weighed to the nearest 0.01 g to a tared dish (fitted with a lid) and dry for 2 h in an oven at 100 to 105°C with lid removed. Remove the dish from the oven, cover with a lid, cool in a desiccator, and weigh.

6. Calculation

6.1 Calculate the percent of moisture as follows:

$$\text{Moisture, \%} = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, and

B = sample used, g.

ASH—AS SULFATE

7. Scope

7.1 This test method covers the determination of the amount of residue left from igniting a sample of hydroxypropyl methylcellulose after being moistened with sulfuric acid.

8. Reagents

8.1 Nitric Acid (sp gr 1.42)—Concentrated nitric acid (HNO_3).

8.2 Sulfuric Acid (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

9. Procedure

9.1 Weigh to the nearest 0.01 g about 2 g of the sample (previously dried for ½ h at 105°C) into a tared Coors No. 1, high-form, porcelain crucible. Add 5 drops of H_2SO_4 around the inside surface of the crucible. Place the crucible inside of a loosely fitting aluminum ring (approximately 1¼ in. (32 mm) high, with ¼-in. (6.4-mm) sidewall, and 1¾-in. (44-

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 D01.36 on Cellulosics.

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² Annual Book of ASTM Standards, Vol 05.01.

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."

mm) inside diameter, cut from a piece of aluminum pipe) on a hot plate. Loosely cover with a crucible cover. Carefully char the hydroxypropyl methylcellulose until all the volatiles are removed.

9.2 Cool the crucible, add 1 mL of H_2SO_4 and 2 mL of HNO_3 so that it completely wets the charred residue. Cautiously heat to dense white fumes on a hot plate. Place the uncovered crucible in a muffle furnace at 600°C and ignite until all the carbon is gone (for about 1 h). Transfer to a dessicator until cool, then weigh. (Save the residue for the Heavy Metals determination.)

10. Calculation

10.1 Calculate the percent of ash, C , as follows:

$$C = (A/B) \times 100 \quad (2)$$

where:

A = sulfated ash, g, and

B = sample used, g.

CHLORIDES—AS SODIUM CHLORIDE

11. Scope

11.1 This test method covers the determination of the total percent of chloride (bromide included if present) calculated as sodium chloride (NaCl) in hydroxypropyl methylcellulose. The sample is dispersed and the chloride titrated volumetrically with 0.100 N silver nitrate solution.

12. Reagents

12.1 *Ferric Alum Indicator Solution*—Add 100 g of ferric ammonium sulfate $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ to 250 mL of water. Heat to boiling and add NH_3 (sp gr 1.42) slowly until the red color is removed. This will usually require about 6 to 15 mL of HNO_3 . Filter the solution and store in a glass bottle.

12.2 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

12.3 *Potassium Thiocyanate Standard Solution* (0.1 N)—Dissolve 10 g of potassium thiocyanate (KCNS) in 1 L of water. By means of a pipet, measure 25 mL of 0.100 N silver nitrate (AgNO_3) solution into a 400-mL beaker. Add 100 mL of water, 10 mL of HNO_3 (sp gr 1.42), and 5 mL of ferric alum indicator solution. Titrate with the KCNS solution, while stirring, until a faint persistent red color is produced. Calculate the normality, N , of the KCNS solution as follows:

$$N = (A/B) \times 0.1 \quad (3)$$

where:

A = 0.100 N AgNO_3 solution added, mL, and

B = KCNS solution required for the titration, mL.

12.4 *Silver Nitrate Standard Solution* (0.100 N)—Grind silver nitrate (AgNO_3) crystals fine enough to pass through a No. 20 (850- μm) sieve and then dry for 2 h at 110°C . Prepare a 0.100 N solution by dissolving 16.989 g of dry AgNO_3 in chloride-free water and diluting to 1 L in a volumetric flask.

13. Procedure

13.1 Weigh to the nearest 0.01 g about 1.0 g of the sample (previously dried for $\frac{1}{2}$ h at 100 to 105°C) and transfer to a 500-mL, wide-mouth Erlenmeyer flask. Add 250 mL of hot

water and swirl for a few minutes; then cool to dissolve.

13.2 Add 5 mL of 0.100 N AgNO_3 solution and 5 mL of ferric alum indicator solution, and back-titrate with 0.1 N KCNS solution to the first appearance of a faint pink color.

14. Calculation

14.1 Calculate the percent of chlorides as NaCl as follows:

$$\text{Chlorides, \%} = [(AB - CD) \times 0.0585] / E \times 100 \quad (4)$$

where:

A = AgNO_3 solution added, mL,

B = normality of the AgNO_3 solution,

C = KCNS solution required to back-titrate the excess AgNO_3 , mL,

D = normality of the KCNS solution, and

E = sample used, g.

ALKALINITY—AS SODIUM CARBONATE

15. Scope

15.1 This test method covers the determination of the total alkalinity of hydroxypropyl methylcellulose expressed as sodium carbonate (Na_2CO_3).

16. Reagents

16.1 *Methyl Purple Indicator Solution*.

16.2 *Sulfuric Acid, Standard* (0.01 N)—Prepare and standardize a 0.01 N solution of sulfuric acid (H_2SO_4).

17. Procedure

17.1 Weigh to the nearest 0.01 g about 1.0 g of the sample (previously dried for $\frac{1}{2}$ h at 100 to 105°C) and transfer to a 500-mL, wide-mouth Erlenmeyer flask. Add 250 mL of hot water and swirl for a few minutes; then cool to dissolve.

17.2 Add 4 drops of methyl purple indicator solution and titrate to the first faint pink color with 0.01 N H_2SO_4 .

18. Calculation

18.1 Calculate the percent alkalinity as Na_2CO_3 , S , as follows:

$$S = [(AB \times 0.053) / C] \times 100 \quad (5)$$

where:

A = H_2SO_4 required for titration of the sample, mL,

B = normality of the H_2SO_4 , and

C = sample used, g.

IRON

19. Scope

19.1 This test method covers the determination of total iron content in samples of hydroxypropyl methylcellulose. The iron is converted to ferric sulfate which reacts with the indicator to form a pink color that can be quantitatively measured.

20. Apparatus

20.1 *Photometer*—Any photoelectric filter photometer or spectrophotometer suitable for measurements at 430 nm.

20.2 *Kjeldahl Flasks*—Calibrated to contain 50 mL, and made of heat- and chemical-resistant glass.

21. Reagents

21.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

21.2 *Buffer Solution*—Dissolve 20 g of sodium bicarbonate (NaHCO_3) and 10 g of sodium carbonate (Na_2CO_3) in water and dilute to 1 L.

21.3 *Disodium-1,2-Dihydroxybenzene-3,5-Disulfonate Solution*⁵—Prepare an aqueous solution containing 25 g/L.

21.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

21.5 *Hydrogen Peroxide* (30 %)—Concentrated hydrogen peroxide (H_2O_2).

21.6 *Iron Standard Solution* (0.0001 g Fe/ml)—Dissolve 0.01 g of iron powder containing not less than 99.9 % iron in HCl (sp gr 1.19). Oxidize the solution with bromine water and expel the excess by boiling. Dilute to 1 L in a volumetric flask.

21.7 *Phenolphthalein Indicator Solution* (1 g/100 mL)—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %).

21.8 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

21.9 *Sulfuric Acid* (1+4)—Carefully mix 1 volume of H_2SO_4 (sp gr 1.84) with 4 volumes of water, adding the H_2SO_4 gradually while mixing.

22. Preparation of Calibration Curve

22.1 Following the procedure given in Section 23, and using varied amounts of the standard iron solution prepared in accordance with 21.6, prepare a calibration curve showing iron content in parts per million and the corresponding photometer readings.

23. Procedure

23.1 Weigh to the nearest 0.01 g about 2 g of the sample (previously dried for $\frac{1}{2}$ h at 100 to 105°C). Transfer by means of a funnel to a Kjeldahl flask. Place the flask at a 20° angle in the furnace at 500°C and heat until some charring of the sample has taken place. (Care must be taken not to char too much.) Remove and allow to cool.

23.2 Add 3 mL of H_2SO_4 to the flask. Place on the digestion rack and digest. Cool and add H_2O_2 dropwise until the solution is clear. Heat over a Meker burner to a volume of 2 mL. Cool, and wash the sides of the flask with water. Add 3 drops of phenolphthalein indicator solution. Add NH_4OH to a red end point. Wash the neck of the flask. The solution should be clear and not greater than 20 mL in volume.

23.3 Add 2 mL of the color-forming solution described in 21.3, and mix. Adjust pH to 7.0 and then dilute to mark with buffer. Transfer a small portion to an absorption cell and determine the photometer reading at 480 nm.

23.4 *Blank*—Make a blank determination, using the same amount of reagents and the same procedure as for the sample.

24. Calculation

24.1 Read the iron content, in parts per million, directly

from the calibration curve (Section 22). Subtract the parts per million of iron due to iron in the blank.

HEAVY METALS

25. Scope

25.1 This test method covers the determination of whether or not the heavy metals content of hydroxypropyl methylcellulose is below a given level based on a lead standard.

26. Summary of Test Method

26.1 The ash residue from the sulfated ash test is digested with dilute hydrochloric acid. A standard containing a known amount of lead is prepared, and the heavy metals content is determined qualitatively by comparing the sample to the standard.

27. Apparatus

27.1 *Nessler Tubes*, 50-mL.

27.2 *Volumetric Flasks*, 50-mL.

28. Reagents

28.1 *Acetic Acid*—Glacial acetic acid.

28.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

28.3 *Ammonium Hydroxide* (2+3)—Dilute 400 mL of NH_4OH (sp gr 0.90) with sufficient water to make 1000 mL.

28.4 *Buffer Solution*—Dissolve 60 mL of acetic acid in about 500 mL of water, add 10 mL of NH_4OH , and dilute to 1 L.

28.5 *Hydrochloric Acid* (1+2)—Dilute 1 volume of concentrated hydrochloric acid (HCl , sp gr 1.19) with 2 volumes of water.

28.6 *Hydrogen Sulfide TS*—Saturate a convenient volume of water with hydrogen sulfide (H_2S) in a narrow-neck, glass-stoppered, amber bottle. This solution must be made fresh.

28.7 *Hydroxylamine Hydrochloride Solution* (200 g/L)—Dissolve 20 g of hydroxylamine hydrochloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$) in 100 mL of water.

28.8 *Lead Nitrate Stock Solution*—Dissolve 159.8 mg of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 100 mL of water containing 1 mL of HNO_3 (sp gr 1.42). Dilute with water to 1000.0 mL and mix. This solution should be prepared and stored in glass containers that are free from lead salts.

28.9 *Lead Standard Solution* (1 mL = 1 μg Pb)—Dilute 10 mL of the lead nitrate stock solution, accurately measured, with water to 100.0 mL. Each millilitre of the solution so prepared contains 10 μg of lead.

29. Procedure

29.1 Pipet into a 50-mL Nessler tube 0.15 mL of HCl (1+2), 2 mL of the buffer solution, and a volume of the standard lead solution containing the quantity of lead equivalent to the specified heavy metals limit. Add water to make 40 mL and label as Solution A. The final pH of these solutions should be between 3 and 4. This can be tested by pH indicator paper or pH meter. For visual comparison the optimum lead content should be between 20 and 40 μg .

29.2 To the crucible containing the sulfated ash residue

⁵ A suitable prepared solution of this reagent, known as "Tiferron" or "Tiron," is supplied by the LaMotte Chemical Products Co., Baltimore, MD.

add 4 mL of HCl (1+2). Cover and carefully digest on a steam bath for 10 min. Uncover and slowly evaporate to dryness. Moisten the residue with 0.15 mL of HCl (1+2), 0.5 mL of hydroxylamine hydrochloride solution, and 10 mL of hot water. Carefully digest for 2 min. Add 2 mL of the buffer solution. Filter if necessary. Thoroughly wash the crucible and filter with water into a 50-mL Nessler tube that matches the one used for Solution A. If necessary, adjust the pH to 3 to 4 with NH_4OH (2+3) or HCl (1+2) using pH indicator paper. Dilute to 40 mL and label this Solution B.

29.3 Add 10 mL of H_2S solution to each tube: Solution A and Solution B. Mix and view downward over a white surface. The color of Solution B shall be no darker than that of Solution A. Make the comparison within 10 min.

METHOXYL CONTENT

30. Scope

30.1 This test method covers the determination of the methoxyl content of hydroxypropyl methylcellulose. Total alkoxyl is first determined and the methoxyl content found by subtracting the hydroxypropyl content from the total alkoxyl figure.

31. Summary of Test Method

31.1 The hydroxypropyl methylcellulose is heated with a strong solution of hydriodic acid to form an alkyl iodide, which by means of a carbon dioxide stream is swept through a condenser and finally absorbed in a mixture of bromine, acetic acid, and sodium acetate. The alkyl iodide absorbed in the aforementioned mixture is oxidized to an alkyl iodate. The excess bromine is reduced with formic acid, and the iodate is determined iodometrically using potassium iodide and standard thiosulfate.

32. Apparatus

32.1 *Distillation Apparatus*, as illustrated in Fig. 1, consisting of a boiling flask with a side arm for admission of carbon dioxide or nitrogen, an air condenser with a trap, and a receiver.

32.2 *Oil Bath*, equipped with a heating device, preferably electrical, so that the bath can be maintained at 145 to 150°C.

33. Reagents

33.1 *Bromine Solution*—Dissolve 5 mL of bromine in 145 mL of the potassium acetate solution. Prepare the bromine solution fresh daily in a hood to remove bromine vapors.

33.2 *Carbon Dioxide*—This may be obtained by the interaction of marble and hydrochloric acid (HCl, 1+1) in a Kipp generator or preferably from a cylinder of the gas equipped with a suitable needle valve. The carbon dioxide (CO_2) shall be passed through a bubble counter and a dry trap, and then through a pressure regulator consisting of a glass tee whose vertical arm extends almost to the bottom of a 10-in. (254-mm) column of water. A screw clamp shall be attached to the thin-walled rubber tubing connecting the horizontal arm of the tee with the boiling flask. This arrangement permits regulation of the flow of gas and allows

any excess gas to escape. Nitrogen may be used in place of CO_2 .

33.3 *Formic Acid* (90 %).

33.4 *Gelatin Capsules*—Gelatin capsules of a suitable size to hold 50 to 60 mg of the dried sample.

33.5 *Hydriodic Acid* (57 %, sp gr 1.70)—Hydriodic acid (HI) forms with water a constant-boiling mixture (boiling point 126 to 127°C) that contains 57 % HI. The concentration of HI in the reagent used should be not less than 56.5 %. The blank determination, which is affected primarily by free iodine in the reagent should require not more than 0.5 mL of 0.1 N sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution.⁶ If necessary, the acid may be purified by adding to it a small amount of red phosphorus and boiling for 20 to 30 min in a hood, while passing a stream of CO_2 into the liquid. Distillation shall then be carried out behind a safety glass shield in a hood, using an all glass apparatus with a slow stream of CO_2 running through the receiver. (Warning—See Note 1) Put the purified HI in small, brown, glass-stoppered bottles, previously swept out with CO_2 , and seal the stoppers with molten paraffin. Store in a dark place. To minimize decomposition of HI due to contact with air, run CO_2 into the bottle while withdrawing portions of the acid for use.

NOTE 1: Warning— Under some conditions the poisonous gas phosphine (PH_3) is formed during distillation, and this may unite with molecular iodine to form phosphorus triiodide (PI_3), which may explode on contact with air. It is, therefore, advisable to keep the current of CO_2 going after the distillation is ended and until the apparatus has cooled.

33.6 *Phosphorus Slurry*—Add about 0.06 g of red phosphorus to 100 mL of water. Shake well before using.

33.7 *Potassium Acetate Solution* (100 g/L)—Dissolve 100 g of anhydrous potassium acetate crystals in 1 L of a solution containing 900 mL of glacial acetic acid and 100 mL of acetic anhydride.

33.8 *Potassium Iodide* (KI).

33.9 *Sodium Acetate Solution* (220 g/L)—Dissolve 220 g of anhydrous sodium acetate in water and dilute to 1 L.

33.10 *Sodium Thiosulfate Standard Solution* (0.1 N)—Dissolve 25 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 200 mL of water and dilute to 1 L. Use freshly boiled and cooled water. It is preferable to allow the solution to stand for a few days before standardization. Standardize the solution against 0.1000 N potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) prepared by dissolving exactly 4.9037 g of $\text{K}_2\text{Cr}_2\text{O}_7$ (National Institute of Standards and Technology Standard Sample No. 136) in water and diluting to a 1 L in a volumetric flask. By means of a buret, measure accurately 35 to 45 mL of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution into a 250-mL Erlenmeyer flask. Add 2 g of KI and 50 mL of H_2SO_4 (1+9) and allow to stand for about 5 min. The flask should be stoppered during the standing period to avoid loss of iodine. Titrate the liberated iodine with the $\text{Na}_2\text{S}_2\text{O}_3$ solution, using starch indicator solution near the end point. At the end point, the blue color of the starch indicator will be destroyed, leaving the pale green color of the chromate ion. The normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution should

⁶ Hydriodic acid suitable for methoxyl determination may be prepared by the method of Samsel, E. P., and McHard, J. A., "Determination of Alkoxyl Groups in Cellulose Ethers," *Industrial and Engineering Chemistry, Analytical Edition*, IECHA, Vol 14, 1942, p. 750. Hydriodic acid available from Merck and Co. has been found satisfactory for this purpose.

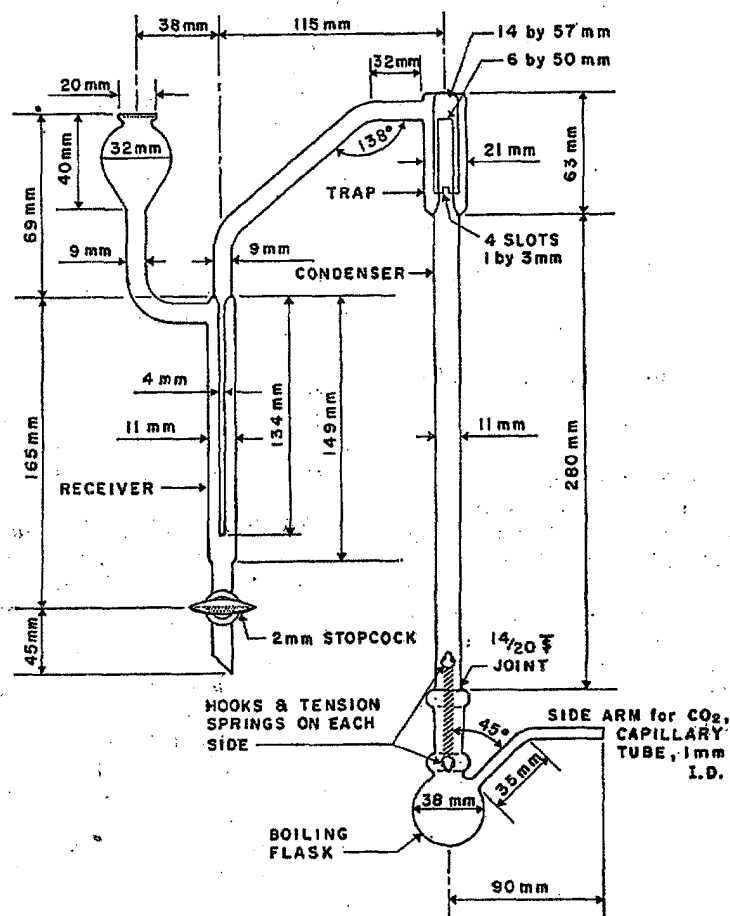


FIG. 1 Distillation Apparatus for Methoxyl Determination

be checked at least once a week. Calculate the normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, N , as follows:

$$N = (A/B) \times 0.1 \quad (6)$$

where:

A = 0.1000 N $\text{K}_2\text{Cr}_2\text{O}_7$ solution added, mL, and
 B = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for the titration, mL.

As an alternative procedure, the $\text{Na}_2\text{S}_2\text{O}_3$ solution may be standardized against arsenic trioxide (As_2O_3) (National Institute of Standards and Technology standard sample No. 83) or potassium iodate (KIO_3).

33.11 *Starch Indicator Solution.*

33.12 *Sulfuric Acid (1+9)*—Carefully mix 1 volume of concentrated H_2SO_4 (sp gr 1.84) with 9 volumes of water, adding the H_2SO_4 gradually while mixing.

34. Procedure

34.1 Dry the sample at 105°C for at least 30 min. Through the condenser, add to the trap in the distillation apparatus (Fig. 1) enough of the phosphorus slurry to make the trap about half full (Note 2). Add 19 to 20 mL of the bromine solution to the receiver. Weigh 50 to 60 mg of the dry sample, to the nearest 0.1 mg, into a gelatin capsule and drop it into the boiling flask. (The weighing should be done as

rapidly as possible without sacrificing accuracy, since dry hydroxypropyl methylcellulose picks up moisture rapidly.)

NOTE 2—Water may be used in the trap to scrub out entrained vapors of iodine quite successfully. If the test method is to be used as a routine control test, this may be advisable for safety purposes. If so, check the accuracy of the apparatus with the water trap against samples that have been run using the phosphorus slurry trap.

34.2 Add a few small glass beads or chips of clay plate and then 6 mL of the HI. Moisten the ground-glass joint with 2 drops of the HI, then fasten to the distillation assembly. Connect the source of CO_2 to the side arm of the flask. Pass a current of CO_2 into the apparatus at the rate of about 2 bubbles/s. Immerse the flask in the oil bath, maintained at 150°C , and heat for 40 min.

34.3 Add 10 mL of sodium acetate solution to a 500-mL Erlenmeyer flask and wash into it the contents of the receiver; dilute to 125 mL with water. Add formic acid dropwise, with swirling, until the brown color of bromine is discharged, and then add about 6 drops more. A total of 12 to 15 drops is usually required. After about 3 min add 3 g of KI and 15 mL of H_2SO_4 (1+9) and titrate immediately with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ solution to a light straw color. Add a little starch indicator solution and continue the titration to the disappearance of the blue color.

34.4 *Blank*—Make a blank determination, using the same amounts of reagents and the same procedure as for the sample. (Usually, about 0.1 mL of 0.1 *N* Na₂S₂O₃ solution is required.)

35. Calculation

35.1 Calculate the percent of methoxyl as follows:

$$M = [(A - B)C \times 0.00517] / D \times 100 \quad (7)$$

where:

M = Methoxyl, total (methyl + hydroxypropyl groups calculated as methoxyl),

A = Na₂S₂O₃ solution required for titration of the sample, mL,

B = Na₂S₂O₃ solution required for titration of the blank, mL,

C = normality of the Na₂S₂O₃ solution, and

D = sample used, g.

35.2 In 35.1 the percent OCH₃ represents the total of methyl and hydroxypropyl groups calculated as methoxyl. To obtain the corrected methoxyl content, the total alkoxyl must be corrected for the percent OC₃H₆OH obtained in Section 41. The percent OC₃H₆OH shall be first corrected by a factor of 0.93 (an average obtained by running Morgan determinations on a large number of samples) for the propylene produced from the reaction of HI with the hydroxypropyl group as follows:

$$A = B - (C \times 0.93 \times 31/75) \quad (8)$$

where:

A = corrected OCH₃, %,

B = total OCH₃, %, and

C = OC₃H₆OH, %.

HYDROXYPROPOXYL CONTENT⁷

36. Scope

36.1 This test method covers the determination of the hydroxypropoxyl content of hydroxypropyl methylcellulose. The figure obtained from this analysis is used in determining the corrected percent methoxyl content.

37. Summary of Test Method

37.1 The hydroxypropoxyl group of hydroxypropyl methylcellulose is oxidized by hot chromic acid to acetic acid and this in turn is titrated with 0.02 *N* sodium hydroxide solution. Procedures are also given for (1) eliminating the error resulting from oxidation of the cellulose backbone, which yields an apparent hydroxypropyl value, and (2) preparing a synthetic hydroxypropyl methylcellulose standard using methylcellulose and propylene glycol.

⁷ References for the hydroxypropoxyl determination are as follows: Dow Method No. MC-15, "The Determination of the Hydroxypropyl Group in the Presence of an Alkyl Group in Hydroxypropyl Methylcellulose."

Lemieux, R. U., and Purves, C. B., "Quantitative Estimation as Acetic Acid of Acetyl, Ethylidene, Ethoxy, and Hydroxyethyl Groups," *Canadian Journal of Research*, Vol B-25, 1947, p. 485.

Morgan, P. W., "Determination of Ethers and Esters of Ethylene Glycol," *Industrial and Engineering Chemistry*, Analytical Edition, IECHA, Vol 18, 1946, p. 500.

38. Apparatus

38.1 *Chromic Acid Oxidation Apparatus*, as illustrated in Fig. 2.⁸

38.2 *Oil Bath*, equipped with an electrical heating device so the bath can be maintained at 155°C.

38.3 *pH Meter*, expanded scale, capable of giving reproducible results within ± 0.1 pH units and equipped with glass and calomel electrodes.

39. Reagents

39.1 *Chromium Trioxide Solution (30 %)*—Dissolve 60 g of chromium trioxide (CrO₃) in 140 mL of organic-free water.

39.2 *Nitrogen*.

39.3 *Potassium Iodide (KI)*.

39.4 *Sodium Bicarbonate (NaHCO₃)*.

39.5 *Sodium Hydroxide, Standard Solution (0.02 N), carbon dioxide-free*—Standardize against primary standard potassium hydrogen phthalate (KHC₈H₄O₄) using a pH meter to an end point of pH 7.0 ± 0.1.

39.6 *Sodium Thiosulfate Standard Solution (0.1 N)*—Dissolve 24.8 g of sodium thiosulfate (Na₂S₂O₃) and 0.2 g sodium bicarbonate (NaHCO₃) in freshly boiled water. Dilute to 1 L with water. Standardize against potassium iodate (KIO₃) using starch indicator to determine the end point.

39.7 *Sodium Thiosulfate Standard Solution (0.02 N)*—Dilute 200 mL of 0.1 *N* sodium thiosulfate standard solution to 1 L with water. Prepare fresh solutions daily as needed.

39.8 *Sulfuric Acid (1+165)*—Carefully add, while stirring 10 mL of concentrated H₂SO₄ (sp gr 1.84) to 165 mL of distilled water.

39.9 *Methylcellulose*, free of foreign material such as other substituted celluloses or glycols that will break down to acetic acid.

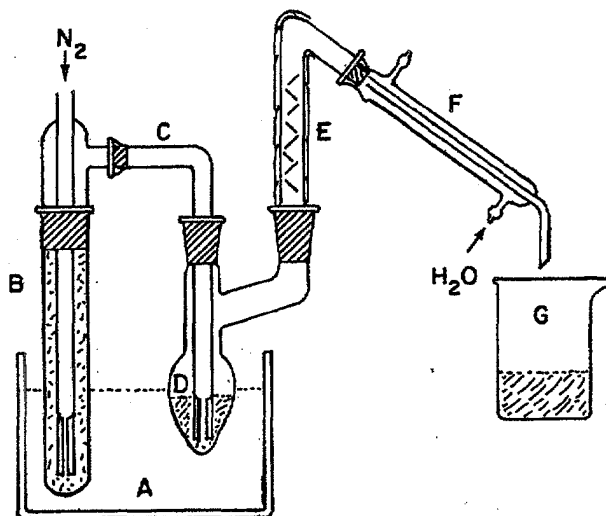
39.10 *Propylene Glycol*.

39.11 Water, organic-free, obtained by distillation or by ion-exchange treatment and to pass the following test: To 100 mL of water add 10 mL of dilute H₂SO₄ (1+16.5), heat to boiling, and add 0.1 mL of potassium permanganate (KMnO₄) solution (approximately 0.1 *N*). The water must retain a pink coloration after boiling for 10 min.

40. Procedure

40.1 Weigh to the nearest 0.0001 g about 100 mg of the sample (previously dried at 105°C for ½ h) and transfer to flask, *D*, and add 10 mL of 30 % CrO₃ solution. Fill the steam generator, *B*, to the bottom of the standard-taper joint and then assemble the apparatus as shown in Fig. 2. Immerse the steam generator and sample flask in the oil bath to the level of the CrO₃ solution. Start the condenser cooling water and pass nitrogen gas through the flask at a rate of 1 bubble per second. Raise the temperature of the bath to 155°C within ½ h and maintain it at this temperature until the end of the determination. Too rapid an initial rise in temperature results in high blanks. Distill until 50 mL of distillate has

⁸ The following parts, manufactured by Ace Glass, Inc., Vineland, NJ 08360 (properly modified), have been found satisfactory for this test method: Steam generator, trap vacuum, No. 8753; Reaction flask, 25-mL, No. 9293 P; Vigneaux column, 100-mL, No. 9224; Adapter bleeder tube, No. 9222; Condenser, micro, 100-mL, No. 9195.



A—Oil bath equipped with an electric heater capable of heating the bath at the desired rate and maintaining the temperature at 155°C.
 B—Steam generator consisting of a 25 by 150-mm test tube and a gas inlet tube with a 1/4 to 1 1/4-mm capillary tip.
 C—Adapter bleeder tube with a 1/4 to 1 1/4-mm capillary tip.
 D—Reaction flask consisting of a 25-mL conical bottom micro boiling flask modified to provide a sidearm outlet.
 E—Vigreux column, 95 mm long, wrapped with aluminum foil.
 F—Micro condenser with a 100-mm jacket.
 G—Beaker, 150-mL, Berzelius, graduated.

FIG. 2 Oxidation and Distillation Apparatus.

been collected. Detach condenser, *F*, from the Vigreux column, *E*, and wash with water, collecting the washings in the beaker containing the distillate. Titrate the solution with standard 0.02 *N* NaOH solution to a pH of 7.0 ± 0.1 using the expanded-scale pH meter. Record the volume, *V*, of standard NaOH used. Add approximately 0.5 g of NaHCO₃ followed by 10 mL dilute H₂SO₄ (1+165). After evolution of carbon dioxide (CO₂) has ceased, add 1 g of KI, mix well, and allow the solution to stand in the dark for 5 min. Titrate the liberated iodine with 0.02 *N* Na₂S₂O₃ to the disappearance of the yellow color. Record the volume, *Y*, of standard Na₂S₂O₃ used. This titration, *Y* mL, when multiplied by the empirical factor, *K*, appropriate to the particular apparatus and reagents in use, gives the acid equivalent not caused by acetic acid. The acetic acid equivalent is (*V* - *KY*) mL of 0.02 *N* NaOH solution.

40.2 Empirical Factor, *K*—The empirical factor, *K*, for each apparatus is obtained by running a blank determination in which the cellulose ether is omitted. The acidity of a blank run for a given apparatus and given reagents is in a fixed ratio to the oxidizing equivalent of the distillate in terms of Na₂S₂O₃ solution as follows:

$$K = (V_b \times N_1) / (Y_b \times N_2) \quad (9)$$

where:

V_b = 0.02 *N* NaOH solution required in blank run, mL,
N₁ = normality of the 0.02 *N* NaOH solution,
Y_b = 0.02 *N* Na₂S₂O₃ required in blank run, mL, and
N₂ = normality of the 0.02 *N* Na₂S₂O₃ solution.

40.3 Methylcellulose Blank—Conduct several determinations using methylcellulose according to the given procedure.

40.4 Hydroxypropoxyl Standard—Since primary standards of hydroxypropyl methylcellulose are not available, a

synthetic standard may be prepared by weighing 100 mg of methylcellulose into the reaction flask and adding 1.0 mL of an aqueous solution containing 1.0 g of propylene glycol in 100 mL. Thus, a secondary standard hydroxypropyl methylcellulose can be established by repeated analysis by this method using properly standardized conditions.

41. Calculation

41.1 Calculate the percent of uncorrected hydroxypropoxyl as follows:

$$A_U = [(V_a N_1 - K Y_a N_2) \times 0.075] / W \times 100 \quad (10)$$

where:

A_U = OC₃H₆OH (uncorrected), %

V_a = 0.02 *N* NaOH solution required for titration of the sample, mL,

N₁ = normality of the 0.02 *N* NaOH solution,

K = empirical factor,

Y_a = 0.02 *N* Na₂S₂O₃ solution required for titration of the sample, mL,

N₂ = normality of the 0.02 *N* Na₂S₂O₃ solution, and

W = sample used, g.

41.2 Calculate the percent of corrected hydroxypropoxyl as follows:

$$A_C = A_U - B \quad (11)$$

where:

A_C = OC₃H₆OH (corrected), %, and

B = OC₃H₆OH obtained from the methylcellulose blank determination, % (40.3).

VISCOSITY

42. Scope

42.1 This test method covers the determination of the

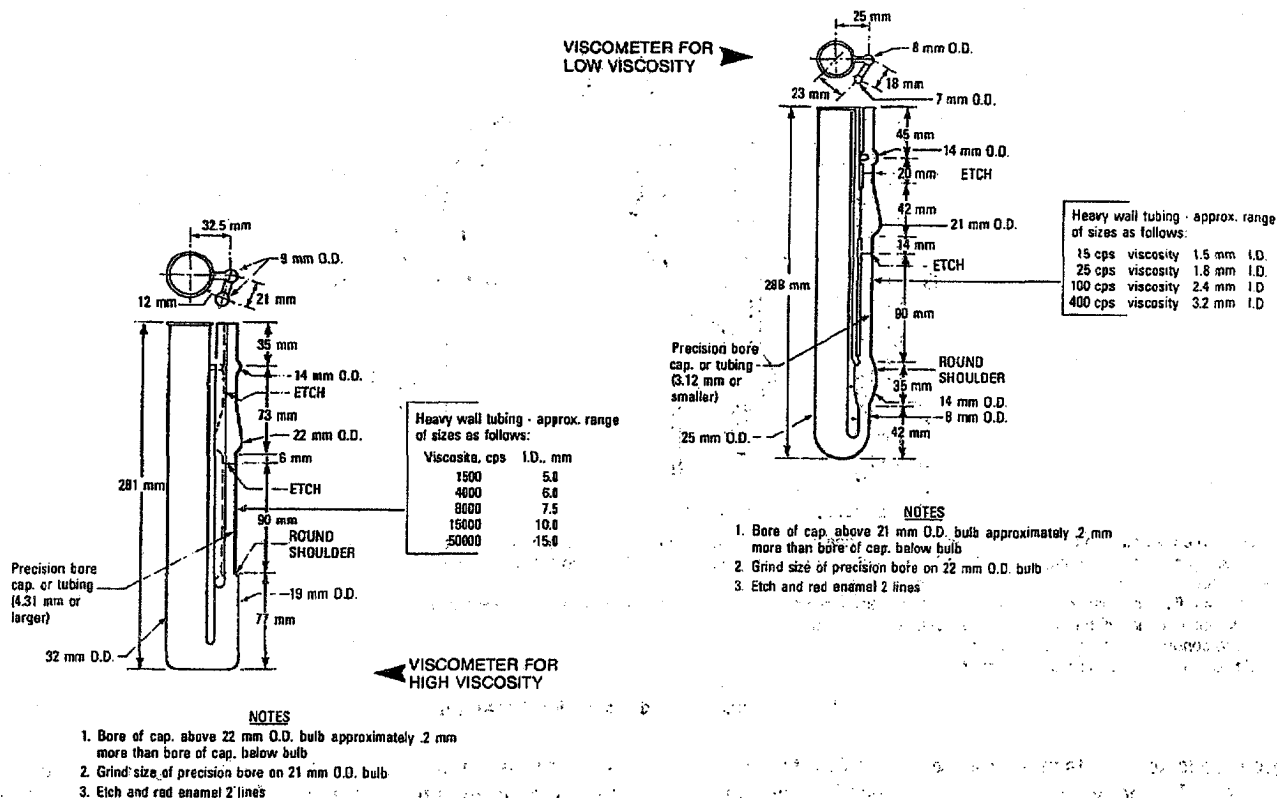


FIG. 3 Hydroxypropyl Methylcellulose Viscometers

apparent viscosity of 2 % water solutions of hydroxypropyl methylcellulose. The viscosities found by this test method do not necessarily correspond to values obtained from other possible test methods.

43. Summary of Test Method

43.1 A 2 % water solution of hydroxypropyl methylcellulose is measured by use of an Ubbelohde tube viscometer. This 2 % solution is based on a dry mass of the product, for example, corrected mass for moisture found in the moisture analysis.

44. Apparatus

44.1 *Viscometer*, as shown in Fig. 3:

NOTE 3—If a viscometer has been repaired, it should be recalibrated before it is used again. Even minor repairs can cause significant changes in the K value.

44.2 *Mechanical Stirrer*.

45. Procedure

45.1 Determine the moisture content of a portion of the sample. Since cellulose and its water-soluble derivatives are hygroscopic, exposure of the sample to the atmosphere should be kept to a minimum. Changes in moisture content can introduce large errors into the accuracy of the determination, and this step should never be omitted if precise results are desired. The suggested method is to weigh out a 2-g portion in a suitable dish, dry at 105°C for 1 h, or until

constant mass is obtained after cooling in a desiccator.

45.2 Correcting for the moisture content, weigh out enough of the undried sample to give 2.000 g of solids, calculated as follows:

$$\text{Mass of sample, g} = [100 / (100 - \text{moisture content, \%})] \times 2 \quad (12)$$

Place the sample in an 8-oz (250-mL) wide-mouth bottle. This weighing step is critical in obtaining good checks and should be done on a good balance sensitive to 1 mg. Masses to the nearest 0.01 g will be sufficiently accurate.

45.3 Add 98.0 g of hot water (85 to 90°C) to the 8-oz (250-mL) bottle containing the 2-g sample.

45.4 Agitate with a mechanical stirrer for 10 min, then place the bottle in an ice bath (0 to 5°C) until solution is complete (at least 20 min). The stirrer assembly should be equipped with a one-hole stopper or bottle cap so that no water vapor is lost during agitation. De-air the solution by some means such as centrifuging.

45.5 When solution is complete as evidenced by the absence of partially swollen or undispersed particles, determine the viscosity in a methylcellulose viscometer at $20 \pm 0.1^\circ\text{C}$. Two precautions should be observed at this point: (1) the solution should be essentially free of air bubbles, and (2) the temperature of the material in the tube should be checked to make certain that it is actually at the bath temperature. The methylcellulose viscosity tube (Fig. 3) consists of three parts: a large filling tube with a reservoir at its lower extremity, *A*; the orifice tube, *B*; and an air vent to the reservoir, *C*; when *B* is filled, *C* should be closed to

prevent the sucking of air bubbles into the orifice tube. Before the sample is allowed to flow through the orifice for the viscosity determination, the vent *C* should be opened so that the column of solution in *B* will flow into the reservoir against atmospheric pressure. Failure to open *C* before running the viscosity will result in false values.

46. Calculation

46.1 Calculate the viscosity as follows:

$$V = Kdt \quad (13)$$

where:

V = viscosity, cP,

K = viscometer constant (Note 4),

d = density of the sample solution at 20/20°C (Note 5), and

t = time for the solution to pass from the upper to the lower mark of the viscometer, s.

NOTE 4—The viscometer constant is determined by passing a standard oil of known viscosity through the tube and determining the time of flow. The above equation can then be solved for *K*.

NOTE 5—For routine work, the density of solutions of hydroxypropyl methylcellulose may be assumed to be 1.00.

pH

47. Procedure

47.1 Determine the pH of the viscosity solution from 45.4, using any suitable pH meter which is standardized according to Test Method E 70.

SOLIDS

48. Scope

48.1 This test method covers the determination of the level of water-insoluble matter in hydroxypropyl methylcellulose.

49. Summary of Test Method

49.1 Hydroxypropyl methylcellulose is dispersed in hot water and then cooled to complete solution. Water-insoluble matter is settled by centrifugal force and measured volumetrically.

50. Apparatus

50.1 *Oil Tubes*, graduated, long-form, 100-mL tapered ASTM, conforming to the requirements prescribed in Sec-

tion 3 and Fig. 1 of Test Methods D 96.

50.2 *Centrifuge*, capable of whirling filled centrifuge tubes at a speed that will produce a centrifugal force of 725 times gravity.

51. Procedure

51.1 Add 1.50 g of bone-dry hydroxypropyl methylcellulose to 148.5 g of 90°C water in a 2¼ by 6-in. (57 by 152-mm) bottle and agitate vigorously for about 15 min or until the material has become finely divided. Place an ice bath around the bottle and agitate the mixture until the solution is effected. This usually requires about 15 min.

51.2 Place 100 mL of this 1 % solution in an oil tube, cool to 10°C, and centrifuge at 725 times gravity for 5 min. The solution temperature should be below 20°C when finished. Read the percent by volume of solids from the graduations on the tube.

DENSITY

52. Scope

52.1 This test method covers the determination of the bulk density of hydroxypropyl methylcellulose.

53. Summary of Test Method

53.1 A weighed amount of hydroxypropyl methylcellulose is transferred to a 250-mL volumetric graduated cylinder and the graduate vibrated to settle the powder.

54. Apparatus

54.1 *Vibrator*—A magnetic-type electric vibrator attached to the vertical support rod of a ring stand approximately 1 ft (0.6 m) above the base. A condenser clamp of sufficient size to hold a 250-mL graduated cylinder also shall be attached to the above rod. The base of the stand should be weighted.

55. Procedure

55.1 Place 50.0 g of powdered hydroxypropyl methylcellulose in a 250-mL graduated cylinder and clamp to the ring stand support. Allow the cylinder to vibrate for 3 min; then observe the level to which the powder has contracted.

56. Calculation

56.1 Calculate the density, *D*, as follows:

$$D = 50/r \quad (14)$$

where *r* = observed reading.

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Standard Test Methods for Hydroxyethylcellulose¹

This standard is issued under the fixed designation D 2364; the number immediately following the designation indicates the year of original adoption 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 testing of hydroxyethylcellulose.

1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 9
Ash	10 to 17
Viscosity	18 to 24
Density	25 to 30
Molar Substitution	31 to 40

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 1193 Specification for Reagent Water²

D 1347 Test Methods for Methylcellulose³

D 1695 Terminology of Cellulose and Cellulose Derivatives³

D 2363 Test Methods for Hydroxypropyl Methylcellulose³

E 1 Specification for ASTM Thermometers⁴

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.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.

3.2 Unless otherwise indicated, reference to water shall be understood to mean reagent water, conforming to Specification D 1193.

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 D01.36 on Cellulose.

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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 14.03.

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."

MOISTURE

4. Scope

4.1 This test method covers the determination of the volatile content of hydroxyethylcellulose.

5. Significance and Use

5.1 The results of this test are used for calculating the total solids in the sample; and, by common usage, all materials volatile at this test temperature are designated as moisture.

5.2 Moisture analysis (along with sulfated ash) is a measure of the amount of active polymer in the material and must be considered when determining the amount of hydroxyethyl cellulose to use in various formulations.

6. Apparatus

6.1 *Oven*, gravity-convection, capable of maintaining a temperature of $105 \pm 3^\circ\text{C}$.

6.2 *Weighing Bottles*, low-form, 50 mm in inside diameter by 30 mm in height, or equivalent.

6.3 *Analytical Balance*.

7. Procedure

7.1 Weigh 5 g of sample to the nearest 0.001 g in a tared and covered weighing bottle.

7.2 Place it in an oven at 105°C for 3 h with the cover removed. Replace the cover; cool in a desiccator, and weigh.

8. Calculation

8.1 Calculate the percent moisture, M , as follows:

$$M = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, g, and

B = sample used, g.

9. Precision and Bias

9.1 Statistical analysis of intralaboratory test results on samples containing from 1 to 6 % moisture indicate a precision of ± 0.2 % absolute at the 95 % confidence level.

9.2 No statement on bias can be made as no suitable reference material is available as a standard.

ASH—AS SULFATE

10. Scope

10.1 This test method covers the determination of the residue on ignition of hydroxyethylcellulose after a specimen has been treated with sulfuric acid.

11. Summary of Test Method

11.1 A specimen is moistened with sulfuric acid, the

excess acid evaporated, the carbonaceous matter burned off, and the residue ignited in a muffle furnace, cooled, and weighed.

12. Significance and Use

12.1 Excessive ash can affect solution clarity and film properties. The ash (along with moisture) is a measure of the amount of active polymer in the material and must be considered when determining the amount of hydroxyethyl cellulose to use in various formulations.

13. Apparatus

13.1 *Dishes*, platinum, 50 to 75-ml capacity.

13.2 *Muffle Furnace*, maintained at $575 \pm 25^\circ\text{C}$.

14. Reagents

14.1 *Sulfuric Acid* (*sp gr* 1.84)—Concentrated sulfuric acid (H_2SO_4).

15. Procedure

15.1 Weigh, to the nearest 0.001 g, about 5 g of the dried sample into a tared platinum dish. Moisten the entire specimen with about 2 mL of H_2SO_4 . Then cautiously heat over a small flame until sulfur trioxide (SO_3) fumes cease to be evolved.

15.2 Increase the heat, ignite the specimen, and heat as necessary to burn off the volatile matter.

15.3 Place the dish in a 575°C muffle furnace for 1 h, or longer if required, to burn all of the carbon.

15.4 Remove the dish, allow to cool somewhat, place in a desiccator, and cool to room temperature. Weigh the dish and residue to the nearest 0.001 g.

16. Calculation

16.1 Calculate the percent of ash (as sulfate), C , as follows:

$$C = (A/B) \times 100 \quad (2)$$

where:

A = ash, g, and

B = sample used, g.

17. Precision and Bias

17.1 Statistical analysis of interlaboratory test results on samples containing 2 to 5 % ash (as sulfate) indicates a precision of ± 0.3 % absolute at the 95 % level.

17.2 No statement on bias can be made as no suitable reference material is available as a standard.

VISCOSITY

18. Scope

18.1 This test method is an arbitrary method of determining the viscosity of aqueous solutions of hydroxyethyl-

cellulose in the viscosity range from 10 to 10 000 cP at 25°C .

18.2 The concentration to be used for the test shall be agreed upon between the purchaser and the seller. It shall be such that the viscosity of the solution will fall within the range of this test.

18.3 The results for the viscosity of hydroxyethylcellulose by this test method will not necessarily check with results from other types of instruments used for viscosity measurements.

18.4 The determinations are run on a calculated dry basis; that is, the amount of hydroxyethylcellulose required for the desired concentration on a dry basis is calculated from the known moisture content.

19. Significance and Use

19.1 This test method is intended for referee purposes. The Brookfield spindles and speeds given in Table 1 are recommended for this purpose, but slight deviations from Table 1 may occasionally be found convenient for individual application.

19.2 This test method determines the relative ability of the polymer to thicken aqueous solutions and is therefore related to the concentration required in various formulations to achieve the desired finished product viscosity.

20. Apparatus

20.1 *Viscometer*, Brookfield-type.⁶

20.2 *Container*, glass jar, 12-oz (350-cm³) approximately 2½ in. (64 mm) in outside diameter and 6 in. (152 mm) high.

20.3 *Mechanical Stirrer*—Agitator as shown in Fig. 17, attached to a variable-speed motor capable of 1500 r/min.

20.4 *Water Bath*, constant-temperature, set at 25°C and capable of maintaining that temperature to within $\pm 0.2^\circ\text{C}$.

20.5 *Thermometer*—An ASTM Saybolt Viscosity Thermometer having a range from 19 to 27°C and conforming to the requirements for Thermometer 17C, as prescribed in Specification E 1.

21. Procedure

21.1 Determine the moisture in accordance with Sections 4 to 9.

21.2 Calculate the dry-basis specimen mass, M , in grams necessary to make 250 g of test solution as follows:

$$M_s = 100 A / (100 - B) \quad (3)$$

where:

A = desired dry mass of specimen, g, and

B = percent moisture in the weighed specimen.

21.3 Add the specimen to the jar. Then add sufficient distilled water to make a total of 250 g of solution. Calculate the mass of water, M_w , in grams as follows:

$$M_w = 250 - S \quad (4)$$

where S = sample mass, g.

21.4 Place the agitator in the solution allowing a min-

TABLE 1 Viscometer Spindles Required for Given Speeds

Viscosity Range, cP	Spindle No.	Speed, rpm	Scale	Factor
10 to 100	1	60	100	1
100 to 200	1	30	100	2
200 to 1000	2	30	100	10
1000 to 4000	3	30	100	40
4000 to 10 000	4	30	100	200

⁶ Model LVF, available from Brookfield Engineering Laboratories, Inc., Stoughton, MA has been found satisfactory for this purpose.

⁷ An agitator made with 1½ in. (38 mm), three-bladed propellers, No. 9240K, available from A. H. Thomas Co., P.O. Box 779, Philadelphia, PA 19105 has been found satisfactory for this purpose.

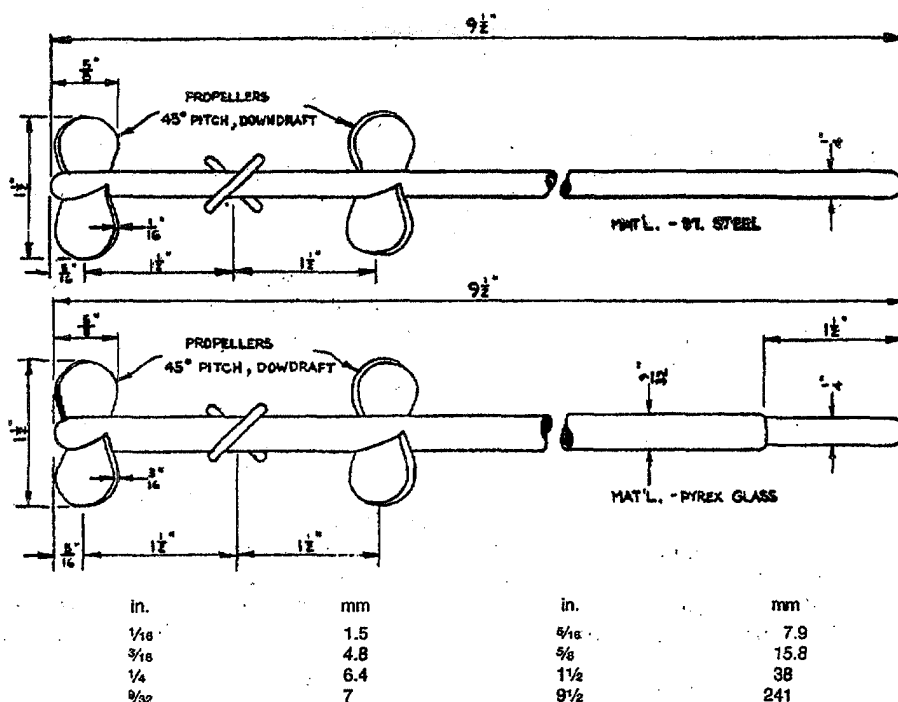


FIG. 1 Agitator

imum clearance between the agitator and the bottom of the container. Stir at approximately 1500 r/min until the specimen is completely dissolved.*

21.5 Remove the agitator from the motor and transfer the specimen container, with the agitator in it, to the constant temperature bath. Allow it to stand for 30 min. At frequent intervals, check the specimen temperature with a thermometer and mix by hand with the agitator to ensure that the test temperature is reached within the 30-min time interval.

21.6 Remove the specimen container from the bath and measure the viscosity with the Brookfield viscometer, following the Brookfield operating instructions, selecting the proper spindle and speed from Table 1. Allow the spindle to rotate until a constant reading is obtained.

NOTE 1—If the room temperature is considerably greater or less than 25°C, the entire operation of stirring, standing, and measurement should be conducted with the specimen suspended in the water bath.

22. Calculation

22.1 Calculate the viscosity, V , in centipoises as follows:

$$V = r \times f \quad (5)$$

where:

r = reading and

f = factor.

23. Report

23.1 Report the following information:

23.1.1 Results, as Brookfield viscosity at 25°C,

23.1.2 Solution concentration,

23.1.3 Spindle used, and

23.1.4 Speed used.

24. Precision

24.1 The difference between the average of the results obtained by a given operator using a given viscometer and the average of the results obtained by a different operator using a different viscometer should not exceed 10 % of the mean of the averages.

DENSITY

25. Scope

25.1 This test method covers the determination of the bulk density of hydroxyethylcellulose.

26. Summary of Test Method

26.1 A weighed amount of hydroxyethylcellulose is transferred to a 250-ml graduated cylinder and the graduate vibrated to settle the powder.

27. Significance and Use

27.1 Density can relate to dry flow properties, rate of dissolution, lumping, packaging, and storage space requirements.

28. Apparatus

28.1 *Vibrator*—A magnetic-type electric vibrator attached to the vertical support rod of a ring stand approximately 1 ft (0.3 m) above the base. A condenser clamp of sufficient size to hold a 250-ml graduated cylinder also shall be attached to the above rod. The base of the stand should be weighted.

29. Procedure

29.1 Place 50.0 g of hydroxyethylcellulose in a 250-mL graduated cylinder and place in the condenser clamp. Turn on the vibrator and allow the cylinder to vibrate for 3 min. Record the level (in millilitres) to which the specimen has compacted.

29.2 Alternatively, the specimen may be compacted manually. Tap it on a hard surface by dropping the cylinder repeatedly from a height of about 1 in. (25 mm) until the volume of the sample remains constant. In order to prevent cylinder breakage, cover the tapping surface with a 1/8 to 1/4-in. (3 to 6-mm) thick rubber sheet, or use a plastic graduated cylinder.

30. Calculation

30.1 Calculate the density, D , in grams per millilitre as follows:

$$D = 50/r_o \quad (6)$$

where R_o = observed reading, mL.

31. Scope

31.1 This test method covers the determination of the molar substitution (MS) of hydroxyethoxyethylene pendant groups in purified hydroxyethylcellulose (HEC).⁸

32. Terminology

32.1 Definition:

32.1.1 *molar substitution, MS*—the average number of oxyethylene groups substituted onto each anhydroglucose unit (see also Terminology D 1695).

33. Summary of Test Method

33.1 In this test method, the pendant groups are cleaved from the cellulose by hydriodic acid and converted to volatile

⁸ This test method was first published by Morgan, P. W. in *Industrial and Engineering Chemistry, Analytical Edition*, Vol 18, 1946, p. 500.

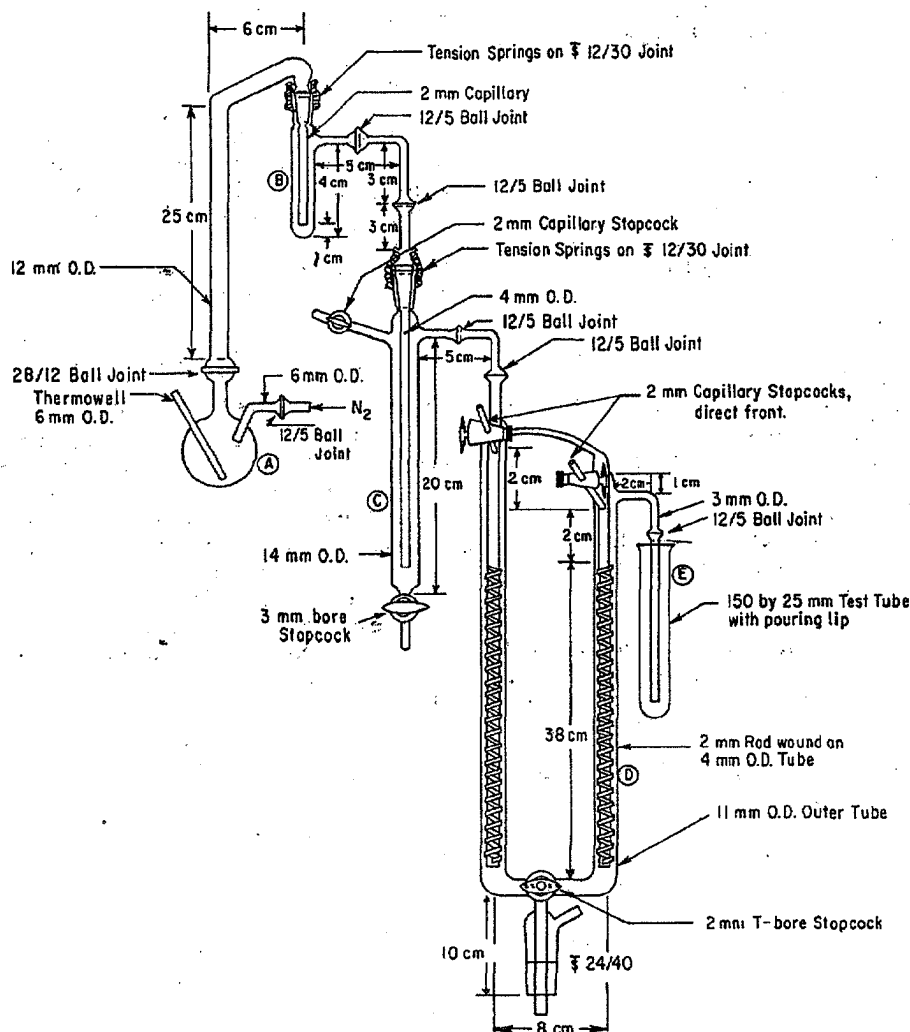


FIG. 2 Molar Substitution (MS) Assembly

ethyl iodide and ethylene. These volatile products are collected in alcoholic silver nitrate and bromine-bromide scrubbers, respectively. The total hydroxyethyl content is calculated on the basis of the sum of both products.

34. Significance and Use

34.1 This test method determines the amount of substituent groups added to the cellulose backbone. The level can affect solution properties, rheology, solubility parameters, and film properties.

35. Interferences

35.1 Interfering materials include glycols, low molecular weight alcohols, and salts of oxidizing acids. Presence of glycols or alcohols, or both, leads to high MS values since reaction of these compounds with hydriodic acid is similar to that of HEC. If present, they must be either removed or determined separately with the results being included in the MS calculations. Large amounts of salts of oxidizing acids also interfere giving low MS values. If any such salt is present in the HEC, its individual permissible level must be determined. Salts of other acids do not interfere chemically. In either case, the amount of the salt in the sample must be determined separately, in accordance with Method D 2364 - 69, and its percent included in the MS calculations in Section 39.

36. Apparatus

36.1 Assemble the apparatus as shown in Fig. 2. The main parts in the drawing are designated as follows:

36.1.1 *Reaction Flask (A)*.

36.1.2 *Condenser Trap (B)*.

36.1.3 *Side Arm Absorber (C)*, containing silver nitrate solution to scrub out ethyl iodide.

36.1.4 *Twin Spiral Tube Scrubber (D)*, containing bromine-bromide solution to scrub out ethylene.

36.1.5 *Test Tube (E)*, containing potassium iodide solution.

36.1.6 *Heat Gun*⁹ (not shown).

36.2 All equipment must be clean, dry and free of leaks. Use clamps or springs at all glass joint connections.

37. Reagents

37.1 *Ammonium Thiocyanate, Standard Solution (0.05 N)*—Prepare 0.05 N solution using distilled water, and standardize in accordance with 8.2 of Test Methods D 1347.

37.2 *Bromine-Bromide Reagent (0.12 N)*—Thoroughly mix 12 mL of liquid bromine and 60 g of sodium bromide (NaBr) in about 3600 mL of acetic acid. The resultant solution should be about 0.12 N. Store in an amber bottle in a dark cabinet.

37.3 *Ferric Nitrate Indicator Solution*—Prepare by dissolving 5.0 g of ferric nitrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] in 100 mL of distilled water.

37.4 *Hydriodic Acid (sp gr 1.7)*—Methoxy grade 57 % hydriodic acid (HI), without preservative.

37.5 *Inert Gas Purge*—Commercial grade nitrogen or carbon dioxide (CO_2).

37.6 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

37.7 *Potassium Iodide Solution (100 g/L)*—Dissolve 100 g of potassium iodide (KI) in distilled water and dilute to 1 L.

37.8 *Red Phosphorus*.

37.9 *Silver Nitrate, Alcoholic Solution (33 g/L)*—Dissolve 33 g of silver nitrate (AgNO_3) in 110 mL of distilled water, and acidify with a few drops of concentrated nitric acid (HNO_3 , sp gr 1.42). Dilute the resultant aqueous solution to a total volume of 1 L using absolute ethanol.

NOTE 2—Denatured alcohol formula S.D. 2B may be used.

37.10 *Sodium Thiosulfate Solution (0.1 N)*—Prepare a 0.1 N solution using distilled water and standardize in accordance with Test Methods D 2363.

37.11 *Starch Indicator Solution (10 g/L)*—Prepare a solution of starch in water with a preservative.

37.12 *Sulfuric Acid (1+16)*—Add 60 mL of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) to 940 mL of distilled water.

38. Procedure

38.1 Dry the sample at 105 to 110°C for 1 h; store in desiccator.

38.2 Add approximately 0.1 g of red phosphorus to condenser trap B and add sufficient water to cover the tip of the capillary (Note 3). Pipet exactly 10 mL of the alcoholic AgNO_3 solution to side arm absorber C. Pipet exactly 20 mL of 0.12 N bromine-bromide solution into the spiral scrubber D with the T-bore stopcock turned so that the solution will flow to both spirals. Fill the 6-in. (152-mm) tube at position E with 30 mL of the KI solution.

NOTE 3—The presence of a sulfur chemical in the HEC will cause a blackening of the precipitate in the AgNO_3 trap. This can be overcome by using a cadmium sulfate solution (50 g/L) in place of water for suspending the red phosphorus in trap B.

38.3 Weigh, to the nearest 0.1 mg, the appropriate amount of dried HEC specimen (Note 4) into reaction flask A. Pipet approximately 10 mL of HI into the reaction flask (Notes 5 and 6). Attach the flask to the assembly. Adjust the flow rate of the purge gas to one bubble per second or preferably to 5 cm^3/min , as measured by a rotameter (not shown in drawing). Heat the reaction flask A with any suitable heat source to an internal temperature of $127 \pm 2^\circ\text{C}$. Allow the reaction to continue to completion at this temperature for 90 min or until the liquid in trap C is essentially clear, whichever is longer.

NOTE 4—The specimen of HEC should contain about 0.0010 to 0.0015 mol of oxyethylene. Thus, approximate specimen weights for given MS levels are as follows: 0.45 g for 0.5 MS HEC; 0.26 g for 1.0 MS HEC; 0.15 g for 2 to 2.5 MS HEC; and 0.1 g for 3 or 4 MS HEC.

NOTE 5—Purge the HI bottle with nitrogen during the entire time the bottle is open in order to exclude oxygen.

NOTE 6—Occasionally, erratic, low MS values are obtained when a sample apparently fails to dissolve in the hot HI. This can be overcome by adding 3 g of molten phenol (USP grade, without preservative) after the HEC sample has been added.

38.4 When the specimen decomposition in flask A is completed, heat the AgNO_3 trap to 50 to 60°C with a heat gun. Do not boil. Continue reaction conditions for 15 min maintaining a $127 \pm 2^\circ\text{C}$ temperature in flask A.

⁹ Model HG501La, 14A, available from the Maswter Appliance Co. or its equivalent has been found satisfactory for this purpose.



Standard Test Method for Silica in Cellulose¹

This standard is issued under the fixed designation D 2438; the number immediately following the designation indicates the year of original adoption 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 silica in cellulose and is applicable to all pulps. It is designed to measure up to 200 μg of silica (SiO_2) in 50 mL of 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 Document

- 2.1 *ASTM Standard:*
D 1348 Test Methods for Moisture in Cellulose²

3. Summary of Test Method

3.1 The cellulose is ashed and hydrochloric acid (HCl)-soluble compounds are extracted. The ash is then fused with sodium carbonate (Na_2CO_3) to form soluble silicates. The salt is dissolved, acidified, and converted to a yellow-colored complex of silicomolybdic acid. The complex is reduced to silicomolybdenum blue by treatment with aminonaphthol sulfonic acid plus sodium sulfite (Na_2SO_3). The concentration of silica is determined spectrophotometrically. When compared with gravimetric procedures for silica, the spectrophotometric method has better specificity, sensitivity, and precision.

4. Significance and Use

4.1 Low-silica specifications are required by viscose rayon processors. The general claims are that (1) silica may affect the filterability of viscose by forming insoluble silicates, (2) silica and the formation of insoluble silicates can contribute to spinneret crater formation during spinning (deposits which build up around spinneret holes to deform yarn cross section and ultimately block the holes; these deposits occur on the acid-wet face), and (3) particulate silica can blind spinneret holes or pass through and cause partial or complete discontinuity in yarn.

5. Apparatus

- 5.1 *Dish*, platinum.
5.2 *Stirring Rod*, plastic.

5.3 *Spectrophotometer*, for measuring absorbance at 700 nm.

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 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 distilled water.

6.3 In some cases, it may be necessary to select specific lots of chemicals to avoid high blank corrections. Minimize the use of glassware; particularly avoid holding alkaline solutions in glassware. Store all reagents in plastic bottles.

6.4 *1-Amino-2-Naphthol-4-Sulfonic Acid Solution*—Dissolve 7 g of anhydrous sodium sulfite (Na_2SO_3) in 100 mL of water. Add 1.5 g of 1-amino-2-naphthol-4-sulfonic acid. Dissolve 90 g of sodium bisulfite (NaHSO_3) in 800 mL of water. Blend the two solutions and filter through a double thickness of an acid-washed general purpose filter paper into a 1000-mL volumetric flask. Dilute to volume.

NOTE 1—Some batches of commercial aminonaphtholsulfonic acid are incompletely soluble or produce dark-colored solutions. These are not satisfactory for silica analysis. The reagent solution should always be discarded if a dark color appears with age. Stability of the solution is improved by refrigeration and protection from light.

6.5 *Ammonium Molybdate Solution*—Dissolve 100 g of ammonium heptamolybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{23} \cdot 4\text{H}_2\text{O}$) in approximately 800 mL of water. Adjust to a pH of 8 by adding sodium hydroxide (NaOH). Dilute to 1000 mL.

NOTE 2—If necessary, filter the ammonium molybdate solution. With the pH adjusted to 8, the reagent is stable indefinitely. If the pH adjustment is not made, a precipitate will form on standing and the solution must be discarded. The added NaOH can be a significant source of SiO_2 contamination. Use a 50 % NaOH solution that has not been in glassware. About 35 mL of this solution are required for 1000 mL of molybdate solution.

6.6 *Hydrochloric Acid Solution A*—Dilute 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1 volume of water. Determine the volume of this acid that is required to adjust the pH of a solution of 1 g of sodium

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 D01.36 on Cellulosics.

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

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."

carbonate (Na_2CO_3) in 15 mL of water of 4.

6.7 *Hydrochloric Acid, Solution B*—Dilute 150 mL of concentrated HCl to 1000 mL with water. Determine the volume of this acid that is required to adjust the pH of a solution of 1 g of Na_2CO_3 in 15 mL of water to 4.

6.8 *Oxalic Acid Solution* (100 g/L)—Dissolve 10 g of oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) in water and dilute to 100 mL.

6.9 *Silica, Standard Solution*—The standard solution may be prepared from SiO_2 or silicic acid (H_2SiO_3). In either case, add about 0.5 g of material to a platinum crucible and dehydrate by heating in a muffle furnace at 1050 to 1100°C, for 1 h. Cool in a desiccator and, without delay, weigh 0.1000 g of the dry sample into a platinum crucible. Add 1.00 g of Na_2CO_3 to the dish, and fuse in a muffle furnace at 850 to 900°C. Hold in the melted state for at least 5 min and swirl occasionally to ensure complete mixing of the silica and the fused carbonate. Allow to cool, add water to dissolve the melt, and transfer quantitatively to a 1000-mL volumetric flask. Dilute to volume, and transfer to a plastic bottle for storage. Discard after 7 days. The standard solution contains the equivalent of 100 μg of SiO_2 /mL.

6.10 *Sodium Carbonate*—Granular solid anhydrous sodium carbonate (Na_2CO_3). Select a lot that does not contain more than 20 ppm of SiO_2 by this test method.

7. Test Specimen

7.1 Select a specimen weight, on the basis of the anticipated silica level in the pulp, to provide 500 μg , or more, of SiO_2 . Weigh out a second specimen for determination of moisture in accordance with Test Methods D 1348.

8. Procedure

8.1 Weigh the specimen into a platinum dish. Ash in a muffle furnace at $575 \pm 25^\circ\text{C}$. Cool the ash, add 10 mL of HCl Solution A, and simmer on a hot plate (under a hood). Continue heating until all of the acid has evaporated and the dry residue has been baked for at least 10 min. Add a small volume of water to dissolve the soluble chlorides. Heat, and stir with a rubber-tipped or plastic stirring rod. Filter through an ashless, medium-speed filter paper and discard the filtrate. Place the filter paper in the original platinum dish, and ignite in the muffle furnace at 575°C .

8.2 Cool the dish, add 1.00 g of solid Na_2CO_3 , and melt in a muffle furnace at 850 to 900°C. Swirl gently to wet all the ash with the melt. Hold in the melted state for 5 to 6 min. Remove the melt from the furnace, allow to cool, and add approximately 15 mL of water. Stir with a plastic rod until solution is complete, transfer quantitatively, without filtration, to a 50-mL flask, and dilute to volume. There may be a small amount of insoluble carbonate which will settle rapidly. Without delay, transfer an aliquot that is expected to contain not more than 200 μg of SiO_2 to a 50-mL volumetric flask. The volume of this aliquot must not exceed 25 mL. Immediately add 5 mL of HCl Solution B plus the calculated volume of HCl Solution A or B that would be required to adjust the amount of carbonate contained in the test aliquot to pH 4. The total volume at this stage must not exceed 35 mL. When small specimen aliquots are used, add water to bring the volume to 25 to 35 mL.

8.3 A reagent blank is required. Prepare for the blank by adding 5 mL of HCl Solution B to approximately 25 mL of

water in a 50-mL volumetric flask.

8.4 For each series of analyses, prepare a color standard equivalent to 200 μg of SiO_2 . Prepare for the standard by adding 2 mL of the standard silica solution to a 50-mL volumetric flask. Dilute to approximately 25 mL and add 5 mL of HCl Solution B.

8.5 A correction for silica in Na_2CO_3 is also required. Fuse 1 g of Na_2CO_3 as though it were a specimen. After cooling, dissolve in a small volume of water and transfer to a 50-mL volumetric flask. Immediately add HCl Solution A to adjust to pH 4, then add 5 mL of HCl Solution B. Do not dilute beyond 35 mL. Develop color in this flask as described in 8.6.

8.6 Develop color in the blank, Na_2CO_3 , standard, and test solutions as follows:

8.6.1 Add 5 mL of ammonium molybdate solution and allow to stand at room temperature for 20 min.

NOTE 3—A yellow-colored complex of silicomolybdic acid forms at this stage. If this color does not occur in the presence of silica, the pH control is not correct. Solutions must be at a pH of 0.9 to 1 before addition of the ammonium molybdate reagent and a pH of 1.1 to 1.3 following addition of this reagent.

8.6.2 Add 2 mL of oxalic acid solution, followed by 5 mL of amino naphthol reagent, dilute to volume with water, and mix well.

8.6.3 Allow to stand at room temperature for 20 min.

8.7 Within 1 h after color development, read the absorbance of all solutions against the reagent blank with a spectrophotometer at 700 nm.

8.8 Prepare a standard curve of absorbance versus concentration by drawing a straight line through the origin and the observed value of the 200- μg standard. Convert all absorbance values for test solutions to SiO_2 weight units by reference to this curve.

9. Calculation

9.1 Calculate the parts per million of SiO_2 in the pulp as follows:

$$\text{SiO}_2, \text{ ppm} = [(50A/B) - C]/W$$

where:

A = SiO_2 in the aliquot, μg .

B = aliquot, mL,

C = SiO_2 in the Na_2CO_3 , ppm, and

W = dry specimen, g.

10. Report

10.1 Report the result to the nearest part per million. Random replicate determinations should not differ by more than 10 % of the observed value.

11. Precision and Bias

11.1 A precision of less than 10 % relative is found by replicate runs by a single analyst.

11.2 No statement of bias can be made as no suitable reference material exists for determining bias.

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 Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment¹

This standard is issued under the fixed designation D 2448; the number immediately following the designation indicates the year of original adoption 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 specific resistance of the aqueous leachate from a pigment as an index of water-soluble salt content. This test method is applicable to white pigments and colored pigments (organic and inorganic). The water-soluble salts content is a function of the specific resistance of the solution formed by extracting the pigment with water.

1.2 This test method is based on a water to pigment ratio of 9+1. The leachate yield (minimum 160 mL) sufficient for rinsing the cylinder dip cell and thermometer plus the minimum 80 mL required for the measurement to determine the quantity of pigment to 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 Document

- 2.1 *ASTM Standard:*
D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 A weighed quantity of pigment is added to water and boiled. For hydrophobic pigments methyl alcohol is used to facilitate wetting. After filtration, the specific resistance of the filtrate is determined using a conductivity bridge.

4. Significance and Use

4.1 This test method provides a reliable means for the determination of the relative amounts of these salts when comparing different lots of the same pigment grade. Water-soluble salts also affect the water resistance and blister resistance of coatings, especially primers for steel. Conductivity measurements, however, cannot be used as the *only* method to determine and compare the amount of water soluble salts of pigments with the same chemical composition but produced by different manufacturing processes.

5. Apparatus

5.1 *Centrifuge* (optional), having a 500-mL capacity per cup.

5.2 *Filter Paper* (Note 1)—For high reliability avoid use of filter paper containing soluble material. Each filter should be washed with reagent water in the funnel until resistance of the filtrate is greater than 200 000 $\Omega \cdot \text{cm}$.

NOTE 1—The size of the filter paper will be determined by how voluminous the pigment is. Some organic pigments require at least a 185-mm paper for proper filtering.

5.3 *Filter Aid* (optional)—The use of a filter aid may be desirable with some pigments to improve filtration. However, the filter aid must be treated to meet the same specifications for filter paper as given in 5.2.

5.4 *Ungraduated Cylinders*, approximately 35 mm wide by 125 mm deep.

5.5 *Thermometer*, graduated in 0.2°C intervals.

5.6 *Conductivity Bridge*.³

5.7 *Conductivity Cell*,³ having a cell constant, K , of 1. The cell constant recommended for various ranges of electrolytes is as follows:

5.7.1 For specific resistances of less than 250 $\Omega \cdot \text{cm}$, use a cell with a constant of 10 or more.

5.7.2 For specific resistances from 250 to 200 000 $\Omega \cdot \text{cm}$, use a cell with a constant of 1. This covers the range for most pigments.

5.7.3 For specific resistances of reagent water or of over 200 000 $\Omega \cdot \text{cm}$, use a cell with a constant of 0.1.

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 as defined by Type II of Specification D 1193.

6.3 *Methanol*.

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 D01.31 on Pigment Specifications.

Current edition approved Nov. 29, 1985. Published January 1986. Originally published as D 2448 - 66 T. Last previous edition D 2448 - 73 (1979)¹.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ Any commercially produced conductivity bridge and conductivity cell is satisfactory.

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 Pharmacopoeia."

6.4 *Potassium Chloride, Standard Solution (0.02 N)*—Prepare a 0.02 N potassium chloride (KCl) solution with water by dissolving 1.491 g of KCl (heated before weighing for 1 h at 105°C) in water, followed by dilution to 1 L.

7. Determination of Cell Constant

7.1 Prepare a 0.002 N solution of KCl by diluting the 0.02 N KCl solution described in 6.4 with water. Cool and measure the specific resistance, R_m , at $25 \pm 0.5^\circ\text{C}$ as described in 8.2.2.

7.2 Calculate the cell constant, K , as follows:

$$K_{25} = (R_m/R_s)$$

where:

R_m = specific resistance at 25°C (see 8.2.2), and

R_s = specific resistance in ohm centimetres of an 0.002 N KCl solution at 25°C (Table 1) = $3427 \Omega \cdot \text{cm}$.

NOTE 2—In general the "cell constant" is not greatly affected by variations in the strength of the KCl solution, but for greatest accuracy, measurements should be made at or near the specific resistance of the solution to be measured and at values that utilize the medium range of the scale of the conductivity bridge, using the same multiplier tap.

7.3 The specific resistances of KCl solutions are shown for concentrations from 0.001 N to 0.073 N at 25°C in Fig. 1. This curve, almost a straight line, was made from published values of specific conductances and equivalent conductances of KCl solutions at 25°C . Table 1 gives values of the specific resistances of KCl solutions for those concentrations useful for pigment testing.

8. Procedure

8.1 *Hydrophobicity Test*—Test a small amount of pigment with boiling water to see if it is water-wettable. Pigments that do not wet well with water are probably hydrophobic and should be treated as described in 8.3. If the pigment wets

TABLE 1 Specific Resistances of Solutions of Potassium Chloride at 25°C

Normality	$\Omega \cdot \text{cm}$
0.001	6802 (1)
0.002	3427 (1)
0.005	1393 (1)
0.01	707.3 (1,2)
0.02	361.5 (3)

easily, proceed as described in 8.2.

8.2 Hydrophilic Pigments:

8.2.1 A 20.0-g specimen weight is usually sufficient for pigments easily wet with water. Add 20.0 g of the pigment to 180 g of boiling water in a tared, 400-mL beaker with stirring rod. (Usually a 250-mL beaker is sufficient for white pigments. Some white pigment, because of tendency to foam and crawl, can be handled better in a larger beaker. Usually 20-g specimens of organic pigments require a 600-mL beaker to allow adequate room for foaming. Boil slowly for 5 min with occasional stirring. Cool to about 60°C and add water to bring the net weight back to 200 g. Stir thoroughly. Filter directly through fine-texture paper (Note 1) or separate the solids by centrifuge using clean, dry cups, or cups washed with some of the slurry, followed by decanting the supernatant liquid through a filter. In either case discard the first 10 mL through the filter.

8.2.2 Cool the filtrate to about 20°C . Rinse the footed cylinder and the conductivity cell, previously rinsed with water, with the leachate. Fill the footed cylinder with the leachate to be measured and place the conductivity cell into the leachate. Move the dip cell up and down to remove all air bubbles. Adjust the temperature slowly to 25°C and, with the cell submerged so that the vent is $\frac{1}{2}$ in. (12.7 mm) below the

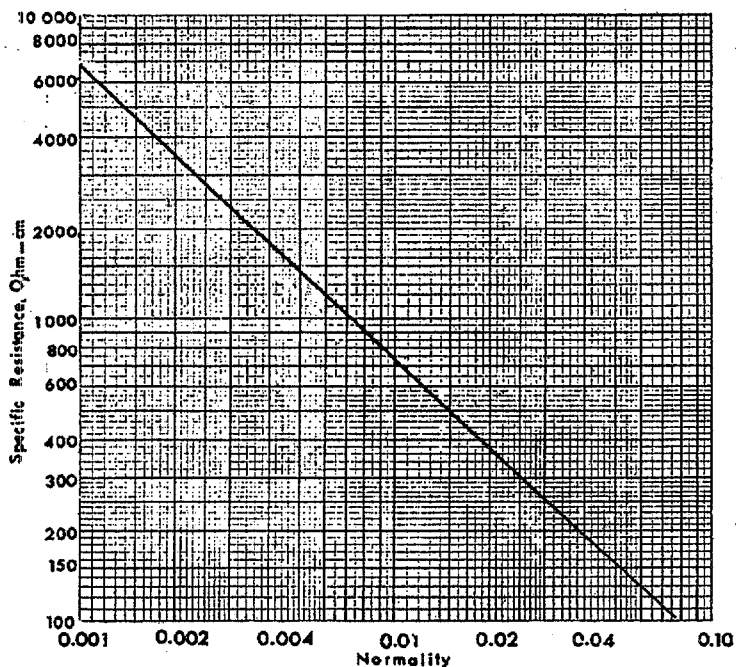


FIG. 1 Specific Resistance of Potassium Chloride at 25°C

surface of the liquid and centered upright in the cylinder, make at least five measurements of the specific resistance at $25 \pm 0.5^\circ\text{C}$, using the conductivity bridge with the multiplier set to give a reading near the center of the scale, following the instructions supplied with the instrument to obtain a balance.

8.3 Hydrophobic Pigments—A modification of the procedure given in 8.2 is necessary for organic pigments that are not easily wet with water. Wet 30.0 g of pigment with 5 to 20 g of alcohol, as required, to produce a smooth wet paste. Complete the addition of water to bring the net weight to 300 g by diluting with boiling water in a tared beaker (1000 mL has been found satisfactory), with a stirring rod. Boil, cool, filter, and determine the reading as outlined in 8.2.2.

9. Calculation

9.1 Calculate the specific resistance, R_{25} , in ohm centi-

metres, of the pigment at 25°C by taking the mean R_o , of the five or more readings made and divide by the cell constant, K , determined in accordance with Section 7.

10. Precision and Bias

10.1 On the basis of a laboratory study of this test method, the following criteria should be used for judging the acceptability of 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 5 % for white pigments and 7 % for colored organic pigments.

10.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 10 % for white pigments and 15 % for colored organic pigments.

10.2 *Bias*—Bias has not been determined for this test method.

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Standard Test Method for Identification of Carboxylic Acids in Alkyd Resins¹

This standard is issued under the fixed designation D 2455; the number immediately following the designation indicates the year of original adoption 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 determination of the carboxylic acids in alkyd resins, including resin-modified alkyds. It may be used for analyzing polyesters but additional peaks may appear from monomers such as styrene.

1.2 The constituents of three resin samples have been correctly identified in collaborative work by three laboratories. It is apparent that quantities as low as 1 % can readily be detected.

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 1193 Specification for Reagent Water²

D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints³

3. Summary of Test Method

3.1 The resin specimen is subjected to rapid transesterification with lithium methoxide to form methyl esters that are separated by programmed temperature gas-liquid chromatography on polar and nonpolar columns, then identified by their retention relative to a standard. If the presence of maleic or fumaric acids is indicated, an alternative transesterification with boron trifluoride is necessary.

4. Significance and Use

4.1 The presence of carboxylic acids in alkyd resins has a direct effect on the crosslinking capability of the resin. This test method helps the user determine the reactivity of the resin.

5. Apparatus

5.1 *Linear Programmed Temperature Gas Chromatograph*—Any instrument with programming features should be suitable if equipped with a hot-wire type of detector and

adaptable to the following operating conditions:

Detector cell temperature, °C	300
Detector cell current, mA	150
Injection port temperature, °C	330
Helium flow at exit, cm ³ /min	85
Programmed temperature details:	
Approximate column heating rate, °/min	4.0
Starting column temperature, °C:	
Polyester-Carbowax	125
Silicone grease, DC-11	75
Finishing column temperature, °C:	
Polyester-Carbowax	225
Silicone grease, DC-11	250

5.2 Columns:

5.2.1 *Polar*—Bend a 6-ft (1.8-m) length of 1/4-in. (6.4-mm) copper tubing into a U-shape and fill one side with 20 % Carbowax 20M⁴ on acid-washed 60 to 80-mesh Chromosorb W⁵ and fill the other side with 20 % diethylene glycol succinate on the same solid support. Maintain constant vibration during the filling and add small amounts of the packing material alternately to each side. Condition at 225°C until “bleeding” reaches a minimum. Mount this column so that the sample passes first through the polyester section. Two 3-ft (0.9-m) sections, packed separately and joined together may be used if preferred.

5.2.2 *Nonpolar*—Pack a 6-ft (1.8-m) length of 1/4-in. (6.4-mm) copper tubing with 20 % silicone grease on acid-washed 60 to 80-mesh Chromosorb W.⁵ Condition at 250°C until “bleeding” reaches a minimum.

5.3 *Syringe*, having a fixed needle, 10- μ L capacity.

5.4 *Separatory Funnel*, 250-mL.

5.5 *Steam Bath*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests (See Section 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, 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, refer-

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.

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² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ Carbowax 20M is a trademark of Union Carbide Corp., 39 Old Ridgebury Rd., Danbury, CT 06817.

⁵ Chromosorb W is Manville Sales Corp., Filtration and Minerals, P.O. Box 5108, Denver, CO 80217-5108.

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.”

TABLE 1 Relative Retention Data for Methyl Esters of Some Carboxylic Acids (Formed by Lithium Methoxide Transesterification)

Methyl Ester of Acid	Triacetin = 1	
	6-ft (1.8-m) Polyester-Carbowax	6-ft (1.8-m) Silicone Grease
Pelargonic	0.29	0.83
Succinic	0.44	0.49
Benzoic	0.47	0.62
Orthotoluic	0.49	0.76
Fumaric	0.63 (0.38) ^A	0.89 (0.49) ^A
Maleic	0.64 (0.52) ^A	0.66 (0.47) ^A
Lauroic	0.66	1.29
Adipic	0.71	0.83
Itaconic	0.74	0.83
Diglycolic	0.79	0.70
Tertiary butyl benzoic	0.83	1.16
Myristic	0.90	1.55
Tetrahydrophthalic	0.97	1.07
Azelic	1.08	1.31
Palmitic	1.14	1.79
Sebacic	1.19	1.43
Terephthalic	1.22	1.23
Orthophthalic	1.26	1.17
Isophthalic	1.26	1.26
Stearic	1.36	2.02
Oleic	1.41	1.98
Linoleic	1.49	1.98
Linolenic	1.60	1.98

^A When transesterified with boron trifluoride in methanol.

ences to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.3 Boron Trifluoride Gas.

6.4 *Lithium Methoxide in Methanol*—Add small pieces of metallic lithium, about the size of a small pea, one at a time to a flask containing absolute methanol which is chilled in an ice bath. Periodically titrate a few millilitres with standardized acid until the normality reaches 0.5 or more. If the normality exceeds 0.5, add a calculated amount of absolute methanol to adjust the reagent to 0.5 N. Filter before each use. Keep tightly stoppered; it may be necessary to discard after one month.

6.5 *Methanol*, absolute.

6.6 *Methylene Chloride*.

6.7 *Sulfuric Acid (1+5)*—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) with 5 volumes of water.

6.8 *Triacetin*.

7. Hazards

7.1 The reagents and samples used in this test method may, under some conditions, be hazardous. Refer to the manufacturer's Material Safety Data Sheets for specific handling and safety precautions. Safe laboratory procedures and all applicable OSHA regulations are to be followed.

8. Procedure

8.1 Pour into a 125-mL flask, a specimen of resin containing approximately 0.3 g of nonvolatile material (Note 1) and add 15 mL of the 0.5 N lithium methoxide reagent. Add an antibumping stone, attach a short air condenser, and place on a steam bath. Swirl constantly until solution is effected; then boil for 2 min. Remove the flask from the bath promptly at the end of the timed 2-min period, remove the condenser, and add 5 mL of H_2SO_4 (1+5) at once. Transfer

the contents of the flask to a separatory funnel and dilute to 50 mL with water. Add 35 mL of methylene chloride and shake vigorously. Separate and wash the solvent layer with 15-mL portions of water until all sulfuric acid is removed; then withdraw the solvent into a small beaker. Place the beaker in a warm water bath and remove as soon as all of the solvent has been expelled. If solid methyl esters are present, add tetrahydrofuran dropwise with warming until all or most of the specimen has dissolved.

NOTE 1—Drying of the resin solutions is not usually necessary, but interference may come from a few high-boiling mineral solvents. They can be removed by dissolving the specimen in 3 mL of chloroform or acetone, drying the specimen with a current of air in a warm water bath, redissolving, and redrying about three times.

8.2 Mount the 6-ft (1.8-m) polyester-Carbowax column in position so that the specimen will pass first through the polyester side and heat the column to starting temperature. Pick up about 5 μ L of specimen in the syringe followed by approximately 0.2 μ L of triacetin and introduce onto the column. Engage the mechanism for increasing column temperature immediately. When maximum column temperature is reached, maintain the temperature until all components emerge. Obtain a chromatogram in like manner with only the triacetin.

8.3 Repeat the chromatographic separation with the 6-ft (1.8-m) silicone grease column following the operating conditions described for this column in 5.1 and obtain a separate chromatogram with only the triacetin.

9. Identification

9.1 Calibration is always recommended and can be made directly with known methyl esters or by treating known esters as described in 8.1 (Note 2). In most cases, the peaks can be identified by their relative position on the chromatograms from the data given in Table 1, in which relative retention is calculated from the position of triacetin which should emerge between 20 and 25 min from the air peak under the conditions described.

NOTE 2—The acids maleic, fumaric, and itaconic do not form true methyl esters when treated as described in 8.1 so that their esters cannot be used directly for calibration; treatment with boron trifluoride will be necessary. Adducts of maleic and fumaric acid will not be detectable under any conditions.

9.2 Since maleic and fumaric acids have the same relative retention, distinction can be made only by repeating the transesterification and substituting concentrated boron trifluoride in methanol as the catalyst, using 5 mL of reagent and boiling for 5.0 min. Prepare the catalyst by bubbling the gas into a flask containing chilled absolute methanol until the titration of 1 mL of the reagent, when diluted with 25 mL of methanol, uses 11 to 12 mL of 0.5 N KOH in methanol, titrating to the yellow end point with thymol blue indicator solution. At the end of the 5-min reflux, transfer the specimen to a separatory funnel with 50 mL of water and 35 mL of methylene chloride and shake vigorously. Filter the methylene chloride layer (no washing is necessary) and evaporate. Obtain a chromatogram of the resultant mixed esters as before and calculate their retention relative to triacetin. Identify from Table 1.

9.3 Since fatty acid methyl esters are eluted it may sometimes be possible to identify the drying oil present in the sample, but a more precise method, Method D 2245, based on quantitative calculation of the fatty acid composition is recommended.

10. Precision and Bias

10.1 No statement is made about either the precision or bias since the results of this test method are qualitative rather than quantitative.

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Standard Test Method for Identification of Polyhydric Alcohols in Alkyd Resins¹

This standard is issued under the fixed designation D 2456; the number immediately following the designation indicates the year of original adoption 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 qualitative determination of the polyols in alkyd resins, including resin-modified alkyds. It may be used for analyzing polyesters, but additional peaks may appear from monomers such as styrene.

1.2 The constituents of three resin samples have been correctly identified in collaborative work by five laboratories. Quantities as low as 1 %, obtained by blending samples of known composition, were readily detected.

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.* For a specific hazard statement, see Section 7.

2. Referenced Document

2.1 ASTM Standard:

D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The resin specimen is subjected to aminolysis to form amides of the carboxylic acids and to release the polyols present, followed by direct acetylation with acetic anhydride. The acetates are extracted and then separated by application of programmed temperature gas chromatography.

4. Significance and Use

4.1 The types of polyhydric alcohols used in the synthesis of alkyd resins has a direct effect on crosslinking capabilities of the resin. This test method helps determine the reactivity of the resin.

5. Apparatus

5.1 *Linear Programmed Temperature Gas Chromatograph*—Any instrument with programming features should be suitable if equipped with a thermal conductivity detector (TCD)³ and adaptable to the following operating conditions:

Detector cell temperature, °C	300
Detector cell current, mA	150
Injection port temperature, °C	330
Helium flow at exit, cm ³ /min	60

Programmed temperature details:

Approximate column heating rate, deg/min	7.9
Starting column temperature, °C	50
Finishing column temperature, °C	225

5.2 *Columns (Note 1)*—A 4-ft (1.2-m) length of 1/4-in. (6.4-mm) outside diameter copper tubing packed with 10 weight % of Carbowax 20M⁴ on 60 to 80 mesh Chromosorb W-AW,⁵ and conditioned at 240°C until “bleeding” reaches a minimum.

NOTE 1—Commercially available columns may be selected and used provided they are capable of performing operations equivalent to the columns specified in this test method.

5.3 *Syringe*, having a fixed needle, 10- μ L 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 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 as defined by Type IV of Specification D 1193.

6.3 *Acetic Anhydride*.

6.4 *n-Butylamine*, boiling point 76 to 78°C.

6.5 *Chloroform*.

7. Hazards

7.1 The reagents and samples used in this test method may, under some conditions, be hazardous. Refer to the manufacturer's Material Safety Data Sheets for specific handling and safety precautions. Safe laboratory handling procedures and all applicable OSHA regulations are to be followed.

8. Procedure

8.1 Weigh a sample of resin containing not less than 1.0 g of nonvolatile material into a 125-mL flask (Note 1) and add 6 mL of *n*-butylamine. Reflux under a water-cooled con-

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.

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² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ Thermal conductivity detector is adequate within the scope of this test method. However, a flame ionization detector (FID) may be used if desired without compromising results of the analysis.

⁴ Carbowax 20M is a trademark of Union Carbide Corp., 39 Old Ridgebury Rd., Danbury, CT 06817.

⁵ Chromosorb W-AW is manufactured by Manville Products Corporation, Lompoc, CA and is available from most sources of chromatography supplies.

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.”

denser for 1 h. Cool and add 25 mL of acetic anhydride slowly and cautiously through the top of the condenser and reflux the mixture for 90 min. Cool, add 35 mL of water, boil for 5 to 10 min, and cool. Transfer to a separatory funnel, filtering if necessary, using water sparingly for the transfer. Extract twice with 25-mL portions of chloroform, shaking vigorously for each extraction. Combine the chloroform extracts and wash with 50-mL portions of water until acid-free. Filter through rapid paper freshly dampened with chloroform, collecting the filtrate in a 250-mL beaker. Reduce the volume to 5 or 10 mL by evaporating the solvent with a current of air in a warm water bath (Note 3).

NOTE 2—Drying of the resin solution is not usually necessary but interference may come from a few high-boiling mineral solvents. They can be removed by dissolving the specimen in 3 mL of chloroform or acetone, drying the specimen with a current of air in a warm water bath, redissolving, and redrying about three times.

NOTE 3—If all the chloroform is expelled, there is a danger of losing some of the lower-boiling polyol acetates.

8.2 Inject approximately 5 μ L of the remaining specimen into the chromatograph, using the operating conditions tabulated in 5.1 and holding the temperature at 225°C at the completion of the program until all the volatile components have emerged.

9. Identification

9.1 Calibration is always recommended and can be made with polyol acetates or by treating known polyols in accordance with 8.1. In most cases, the peaks can be identified by their relative position on the chromatogram from the data given in Table 1, in which the relative retention is calculated

TABLE 1 Relative Retention Data for Polyol Acetates

Acetate of Polyol	Relative Retention Time (Second Reagent Peak = 1)
2,3-Butylene glycol	0.70
Propylene glycol	0.72
Ethylene glycol	0.76
1,3-butylene glycol	0.82
Neopentyl glycol	0.84
1,4-Butanediol	0.98
Dipropylene glycol	1.05
Diethylene glycol	1.16
Glycerol	1.24
Trimethylol ethane	1.33
Trimethylol propane	1.37
Triethylene glycol	1.43
(o-Phthalic acid derivative)	(1.54)
Pentaerythritol	1.81
Mannitol	3.08
Sorbitol	3.24

from the second of the two peaks that are produced by the reagents which appear between 14 and 16 min under the programming conditions described in 5.1.

NOTE 4—With many alkyd resin specimens, a peak will appear with relative retention of 1.54 due to the presence of *ortho*-phthalic anhydride in the resin.

10. Precision and Bias

10.1 The precision and bias for this test method has not been determined.

11. Keywords

11.1 alkyd resin; aminolysis; gas chromatography; polyester resin; polyhydric alcohol; polyol; polyol acetates

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Standard Test Method for Isocyanate Groups in Urethane Materials or Prepolymers¹

This standard is issued under the fixed designation D 2572; the number immediately following the designation indicates the year of original adoption 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 7421 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 This test method covers the determination of the isocyanate group (NCO) content of a urethane intermediate or prepolymer.

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 8.*

2. Referenced Document

2.1 ASTM Standard:

D 841 Specification for Nitration Grade Toluene²

3. Summary of Test Method

3.1 The urethane prepolymer is allowed to react with an excess of di-*n*-butylamine in toluene (Note 1). After the reaction is complete, the excess of di-*n*-butylamine is determined by back titration with standard hydrochloric acid.

NOTE 1—Solvents other than toluene may be needed in some cases, for example, anhydrous dimethylformamide (DMF), but the procedure has not been evaluated using them.

4. Significance and Use

4.1 The percent NCO is used to establish unit ratios for stoichiometric reactions with co-reactants.

5. Interferences

5.1 Phosgene, the carbamyl chloride of isocyanate, hydrogen chloride, or any other acidic or basic impurities of sufficient strength will interfere. The concentration of these compounds is usually sufficiently low that their effect on the determination is negligible.

6. Apparatus

6.1 *Polyethylene Stoppers (or corks)* covered with aluminum foil to fit 250-mL Erlenmeyer flasks.

6.2 *Magnetic Stirrer* with TFE-fluorocarbon-covered stirring bar.

6.3 *Pipet*,³ 25-mL.

6.4 *Buret*,³ 50 or 100-mL.

6.5 *Analytical Balance*.

7. Reagents

7.1 *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 that 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 *Bromphenol Blue Indicator*, prepare solution 0.1% by mixing 0.10 g of acid, nonwater soluble bromphenol blue with 1.5 mL of 0.1 *N* sodium hydroxide solution and diluting to 100 mL with distilled water.

7.3 *Di-*n*-butylamine Solution*, 0.1 *N* in dry toluene. Not standardized.

7.4 *Hydrochloric Acid Solution*, 0.1 *N* standardized using bromphenol blue indicator solution.

7.5 *Isopropyl Alcohol*.

7.6 *Toluene*, anhydrous, conforming to Specification D 841 or equivalent dried with molecular sieve pellets.

8. Hazards

8.1 Diisocyanates are strong skin irritants and sensitizers. They are also hazardous to the lungs. (TLV for toluene 2,4-diisocyanate is 0.02 ppm.) Avoid contact with skin, eyes, and clothing, and avoid breathing these substances. Use with adequate ventilation. In case of contact, wash with plenty of water and flush eyes for 15 min. Call a physician before re-use. See Supplier's Material Safety Data Sheet for additional information.

9. Procedure

9.1 Weigh to 0.1 g a specimen containing approximately 1.1 milliequivalents of NCO (for example, 1.0 g of prepolymer containing approximately 5 % NCO) in a 250-mL Erlenmeyer flask.

³ Burets and pipets shall conform to National Institute of Standards and Technology tolerances.

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."

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.

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² *Annual Book of ASTM Standards*, Vol 06.03.

9.2 Add 25 mL of dry toluene (Note 2), place a stopper in the flask, and swirl by hand or on a mechanical agitator to dissolve the prepolymer. Solution may be aided by warming on a hot plate.

NOTE 2—If the polymer is insoluble, add 10 mL of dry, analytical grade acetone to the toluene.

9.3 Using a pipet, add 25.00 mL of 0.1 *N* di-*n*-butylamine solution and continue swirling for 15 min with stopper in place.

9.4 Add 100 mL of isopropyl alcohol and 4 to 6 drops of bromphenol blue indicator solution. Titrate with 0.1 *N* hydrochloric acid to a yellow end point.

9.5 Run a blank titration including all reagents above but omitting the specimen.

10. Calculations

10.1 Calculate the NCO content as follows:

$$\text{NCO, \%} = \frac{(B - V) \times N \times 0.0420}{W} \times 100$$

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where:

B = volume of HCl for titration of the blank, mL,
V = volume of HCl for titration of the specimen, mL,
N = normality of HCl,
 0.0420 = milliequivalent weight of the NCO group, and
W = grams of specimen weight, g.

11. Precision and Bias

11.1 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 0.11 % absolute NCO.

11.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 0.4 % absolute NCO.

12. Keywords

12.1 Toluene, 2,4-diisocyanate based intermediate; toluene, 2,4-diisocyanate based prepolymer; urethane intermediate; urethane prepolymer



Standard Test Method for Chlorine in Cellulose¹

This standard is issued under the fixed designation D 2641; the number immediately following the designation indicates the year of original adoption 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 method² covers the determination of chlorine in cellulose. Procedures for total chlorine, water insoluble chlorine, and alcohol-benzene insoluble chlorine are described. The range is 5 to 1000 ppm on cellulose. Total chlorine determinations (including ashing) can be performed at a rate of one determination every 30 min.

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 1794 Test Method for Alcohol-Benzene Soluble Matter in Cellulose³

3. Summary of Test Method

3.1 The total chlorine determination is a measure of the water soluble chlorides as well as the chlorine bound in the pulp.

3.2 This test method makes use of the oxygen flask technique for the combustion of the pulp sample. After combustion, the chloride ions are absorbed in dilute sodium hydroxide, the solution is evaporated to dryness, and the residue is redissolved in a small amount of dilute nitric acid. The solution is diluted with acetone and titrated with 0.0025 M silver nitrate solution using a potentiometric titrator.

4. Significance and Use

4.1 Measurement of total chlorine levels gives the sum of water soluble chlorides and the organically bound chlorine compounds present in a pulp sample. Inorganic chlorine is known to be detrimental to the performance of many types of cellulose derivatives such as in viscose manufacturing.

4.2 Organic chlorine compounds are of interest from the environmental standpoint. The levels of organic chlorine compounds in pulp mills and in pulp products is under investigation in light of the suspected toxicity of these compounds to aquatic life.

5. Apparatus

5.1 *Potentiometric Titrator*, preferably equipped with a recorder. The use of a recorder is considered essential at low levels of chlorine.⁴

5.2 *Electrodes*, glass or mercurous sulfate reference electrode and silver-silver chloride indicator electrode.

5.3 *Combustion Flasks*, 1000-mL conical, borosilicate glass, with 35/25 ball and joint stopper and 5000-mL round bottom, borosilicate glass, with 65/40 ball and joint stopper. The flasks are equipped with perforated platinum sample carriers sealed into extended stem of flask stopper.

5.4 *Igniter*, suitable for igniting the sample in the flask. An "infrared igniter" is preferred.⁵

5.5 *Specimen Wrappers*, black paper.

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 *Water*—Deionized and chloride-free. (It is essential that only this type of water be used in this test method.)

6.3 *Silver Nitrate, Standard Solution (0.0025 M)*—Dissolve 0.4247 g of silver nitrate (AgNO_3) in water, add 3 mL of concentrated nitric acid (HNO_3 , sp gr 1.42); and dilute to 1000 mL. Standardize daily against sodium chloride solution. 1 mL of 0.0025 M AgNO_3 solution is equivalent to 0.0886 mg of chlorine.

6.4 *Sodium Chloride, Standard Solution (0.0025 M)*—Dissolve 0.1462 g of sodium chloride (NaCl) in water and dilute to 1000 mL.

6.5 *Nitric Acid (1+100)*—Dilute 10 mL of concentrated nitric acid (HNO_3 , sp gr 1.42) to 1000 mL with water. Store in a glass-stoppered bottle.

6.6 *Sodium Hydroxide, Standard Solution (0.1 N)*—Transfer the contents of a standard concentrated volumetric ampoule into a polyethylene bottle and dilute to 1000 mL.

6.7 *Acetone*.

¹ This test method is under the jurisdiction of the ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D01.36 on Cellulosics.

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² See Rioux, J. P., and Hurtubise, F. G., "Determination of Chloride Ions in Pulp and Paper," *TAPPI*, Vol 48, January 1965, pp. 11-14.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ One suitable titrator-recorder is the Radiometer TTT-1 titrator equipped with the Radiometer SBU-1 titration assembly, manufactured by Radiometer America Inc., 811 Sharon Drive, Westlake, OH 44145.

⁵ The Thomas-Ogg apparatus manufactured by the Arthur H. Thomas Co., Philadelphia, PA, has 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."

7. Report

7.1 Report the results to the nearest parts per million of cellulose dried at 100 to 105°C.

8. Precision and Bias

8.1 A precision of less than 10 % relative is found by replicate runs by a single analyst.

8.2 No statement of bias can be made as no suitable reference material exists for determining bias.

TOTAL CHLORINE

9. Standardization

9.1 Pipet an aliquot of the 0.0025 *M* NaCl solution into a titration vessel. Add 5 mL of 0.1 *N* NaOH solution, evaporate, and treat as in 10.7.

9.2 Carry out a blank titration by igniting a black paper wrapper, omitting the specimen. Subtract this titration from those obtained for specimens.

10. Procedure

10.1 Cut a suitable air dry test portion of the sample into 0.5-in. (13-mm) squares and weigh to the nearest 0.001 g on a black paper specimen wrapper.

NOTE—For samples containing 5 to 100 ppm chlorine, 1-g air dry test portions are burnt in a 5-L flask; for samples containing 100 to 1000 ppm chlorine, 0.3-g air dry test portions are burnt in a 1-L flask.

10.2 Place the test portion wrapped in the black paper in the platinum basket.

10.3 Add 5.0 mL of 0.1 *N* NaOH solution to the combustion flask. Introduce the specimen in the flask and allow a stream of oxygen to pass through the flask until the air is completely displaced. Then, stopper the flask with a pinch clamp.

10.4 Turn on the igniter and focus the infrared light beam on the black paper fuse on the wrapper until ignition occurs. This operation should be carried out behind a protective shield.

10.5 After the specimen has burned completely, allow the flask to cool until all vapors are absorbed by the NaOH solution. Transfer the solution to a titration vessel and rinse the flask with deionized water (approximately 50 mL), adding the rinsings to the solution.

10.6 Evaporate the solution to approximately 5 mL on an asbestos-covered hot plate. Then evaporate to dryness under an infrared lamp, and allow to cool.

10.7 Add 4 mL of HNO₃ (1+100) and swirl to dissolve any residue on the walls of the vessel. Add 25 mL of acetone, titrate with 0.0025 *M* AgNO₃ solution, and record the titration curve.

11. Calculation

11.1 Calculate the chlorine content, *C*, in parts per million as follows:

$$C = (AB/W) \times 1\,000\,000 \quad (1)$$

where:

A = 0.0025 *M* AgNO₃ solution required for titration of the specimen, corrected for the blank, mL,

B = chlorine equivalent of the 0.0025 *M* AgNO₃ solution, g/mL, and

W = specimens used, g.

WATER-INSOLUBLE CHLORINE

12. Procedure

12.1 Using clean scissors, cut and place 4.0 ± 0.01 g of air dry sample in a paper thimble, place in a Soxhlet extractor and extract in accordance with Test Method D 1794, using water instead of alcohol-benzene.

12.2 Remove a 2-g portion of the sample from the thimble. Dry this portion at 105°C for 1 h. Determine the chlorine in accordance with Section 9.

12.3 Calculate the chlorine content in accordance with Section 11.

ALCOHOL-BENZENE INSOLUBLE CHLORINE

13. Procedure

13.1 Replace the thimble with the remaining pulp in the extractor, and extract in accordance with Test Method D 1794.

13.2 Dry the extracted pulp at 60°C for 1 h. Determine the chlorine in accordance with Section 9.

13.3 Calculate the chlorine content in accordance with Section 11.

LIGNIN-BOUND CHLORINE

14. Calculation

14.1 The Lignin-Bound Chlorine discussed by Howard and Histed⁷ is numerically identical to the Alcohol-Benzene Insoluble Chlorine as determined in Section 13.

RESIN-BOUND CHLORINE

15. Calculation

15.1 Calculate the Resin-Bound Chlorine, *C_{RB}*, as follows:

$$C_{RB} = A - B \quad (2)$$

where:

A = water-insoluble chlorine and

B = alcohol benzene insoluble chlorine.

WATER-SOLUBLE CHLORINE

16. Calculation

16.1 Calculate the Water-Soluble Chlorine, *C_{WS}*, as follows:

$$C_{WS} = A - B \quad (3)$$

where:

A = total chlorine and

B = water-insoluble chlorine.

⁷ See Howard, E. J., and Histed, J. A., *TAPPI*, Vol 47, November 1964, pp. 655-662, and Histed, J. A., *TAPPI*, Vol 47, November 1964, pp. 669-673.

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Standard Practices for Testing Alkyd Resins¹

This standard is issued under the fixed designation D 2689; the number immediately following the designation indicates the year of original adoption 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 cover test methods for testing alkyd resins as listed in Table 1. They are the test methods most often used to characterize alkyds. All of the analytical test methods were subjected to interlaboratory testing using oil-modified alkyds. Their adaptability to other types of alkyds has not been studied. Although each test method specifies a recommended amount of specimen for starting a separate analysis, several of the procedures can be conducted on the same starting specimen, if desired. For example, the tests for unsaponifiable matter, fatty acid content, oil identification, and phthalic acid content could all be run on a consecutive basis, if all were required.

2. Referenced Documents

2.1 ASTM Standards:

- D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester²
- D 563 Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions³
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)⁴
- D 1259 Test Methods for Nonvolatile Content of Resin Solutions³
- D 1306 Test Method for Phthalic Anhydride Content of Alkyd Resins and Esters Containing Other Dibasic Acids (Gravimetric)³
- D 1397 Test Method for Unsaponifiable Matter in Alkyd Resins and Resin Solutions³
- D 1398 Test Method for Fatty Acid Content of Alkyd Resins and Alkyd Resin Solutions³
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁵
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)⁶
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁶
- D 1639 Test Method for Acid Value of Organic Coating Materials⁵

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 D01.33 on Varnish and Resins, Including Shellac.

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² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vols 06.01 and 06.03.

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

D 2090 Test Method for Clarity and Cleanness of Paint and Ink Liquids⁷

D 2245 Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints²

D 2455 Test Method for Identification of Carboxylic Acids in Alkyd Resins³

D 2690 Test Method for Isophthalic Acid in Alkyd and Polyester Resins³

D 2998 Test Method for Polyhydric Alcohols in Alkyd Resins³

D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus²

3. Significance and Use

3.1 These practices should be used as a reference for any alkyd resin analyst who wants general information about classifying alkyd resins. In each case, the significance and use of the specific classification item will be found in the referenced test method.

4. Nonvolatile Content

4.1 A unique test method for determining nonvolatile matter in solutions of alkyd resins in volatile organic solvents provides for the drying of very thin films of resin quickly, thus minimizing chances for volatiles to be trapped or drying oils to oxidize. Test Method A of Test Methods D 1259, was thoroughly tested with alkyd resins with outstanding repeatability of 0.1 % and reproducibility of 0.2 %. This procedure, sometimes referred to as the "foil" test method, should be considered the "referee" test method for all alkyd analyses requiring maximum precision.

5. Acid Value

5.1 Acid value of an alkyd resin is expressed as the number of milligrams of potassium hydroxide required to neutralize the free acidity of 1 g of nonvolatile material under the conditions of the test. Test Method D 1639, was especially prepared for this purpose.

6. Unsaponifiable Matter

6.1 The unsaponifiable matter in alkyd resins is the ethyl ether-soluble, water-insoluble portion that remains after a relatively large specimen has been subjected to an aqueous-alcoholic saponification. The yield is normally low and is an

⁷ Annual Book of ASTM Standards, Vols 06.02 and 06.03.

TABLE 1 Test Methods for Testing Alkyd Resins

Test Method	Section	ASTM Designation
Nonvolatile content	4	D 1259
Acid value	5	D 1639
Unsaponifiable matter	6	D 1397
Fatty acid content	7	D 1398
Identification of oils and acids	8	D 2245
Phthalic anhydride content	9	D 563
		D 1306
		D 2690
Isophthalic acid content	10	
Polyhydric alcohol content	11	D 2998
Identification of carboxylic acids	12	D 2455
Flash point	13	D 93
		D 3278
Color	14	D 1209
		D 1544
Density	15	D 1475
Viscosity	16	D 1545
Clarity	17	D 2090

indication of purity insofar as the presence of modifying resins will affect the result. Certain other resins can interfere due to variations in degree of saponification and solubility in ether. The test method is therefore not applicable to alkyds containing such other resins as styrene, rosin, phenols, and formaldehyde condensates. The unsaponifiable matter is determined gravimetrically in accordance with Test Method D 1397 and may be calculated on the nonvolatile or solution basis according to prior agreement.

7. Fatty Acid Content

7.1 The total fatty acids in alkyd resins can be measured gravimetrically on a separate specimen in accordance with Test Method D 1398. This test method provides for the isolation of fatty acids after saponification under anhydrous conditions, removal of the salts of the dicarboxylic acid by filtration, removal of the unsaponifiable matter and original solvents by benzene extraction of the filtrate, and finally ether extraction of the acidified aqueous layer that remains. The test method was prepared for use with orthophthalic alkyds and, if isophthalic acid is present, a considerable amount will appear in the extracted oil acids where it is readily detected as white crystals. Interference from isophthalic acid can be avoided by redissolving the oil acids in benzene, filtering into a weighed beaker and drying as before. Some modifying agents such as urea, melamine, phenols, rosin, and styrene will contaminate the isolated fatty acids in varying degrees and Test Method D 1398 is considered inapplicable in the presence of such other resins. If the fatty acids isolated by Test Method D 1398 are to be examined further for identification (Section 8), a small, weighed crystal of hydroquinone to serve as an antioxidant, should be added prior to the evaporation of the ether.

8. Identification of Oils and Oil Acids

8.1 The oils and oil acids contained in oil-modified alkyd resins can be identified by gas chromatographic separation of their methyl esters that are formed in the presence of margaric acid added as an internal standard. The test method is not applicable to fatty acids that have polymerized or oxidized to such an extent that no characteristic monomeric fatty acids remain. The test is conducted in accordance with

Method D 2245, and the sample may be obtained as described therein or from material remaining from the fatty acid determination of Section 7.

9. Phthalic Anhydride Content

9.1 Alkyd resins that are known to contain phthalic anhydride as the only dicarboxylic acid and are not modified with resins such as phenolics, urea- or melamine-formaldehyde may be analyzed in accordance with Test Method D 563. Styrene does not interfere so that styrenated alkyds may be tested by the same test method.

9.2 Determination of phthalic anhydride in alkyd resins that contain isomers of phthalic acid or other dibasic acids such as maleic, fumaric, etc., must be made in accordance with Test Method D 1306, in which the phthalic anhydride is determined gravimetrically as the lead salt.

10. Isophthalic Acid Content

10.1 A rapid and simple procedure for the gravimetric determination of isophthalic acid in alkyd resins is provided in Test Method D 2690. There is no interference in this test method from styrene monomer or polymer or from other dicarboxylic acids except terephthalic acid, which is recovered quantitatively.

11. Polyhydric Alcohol Content

11.1 The polyhydric alcohols in alkyds can be determined qualitatively and quantitatively in accordance with the procedure in Test Method D 2998. The resin to be analyzed is dried, subjected to aminolysis, and treated to form trimethylsilyl ether derivatives of the alcohols which are separated by gas-liquid chromatography. An internal standard is used for quantitative purposes. Flame-ionization detectors cannot be used with Test Method D 2998.

12. Identification of Carboxylic Acids

12.1 Method D 2455 describes a procedure for the qualitative determination of all the carboxylic acids in alkyd resins, including resin-modified alkyds. This procedure makes use of gas chromatographic separation of the methyl esters of the respective acids that are formed by direct transesterification of the resin with lithium methoxide. The acids, including the fatty acids, are identified by their relative retention time which is given for 23 acids and others may be added if desired. Since maleic and fumaric acids react differently than the other acids encountered, an alternative procedure is supplied for their differentiation. It is sometimes possible to identify the drying oils in the alkyd from the fatty acid esters that appear in this procedure, but it is not as reliable for oil identification as Method D 2245 (Section 8).

13. Flash Point

13.1 In the Setaflash closed tester, a small specimen is injected by means of a syringe and its flash temperature is determined visually, as described in Test Method B of Test Method D 3278. This procedure is faster, more convenient, and somewhat more precise than the Pensky-Martens procedure described in Test Method B of Test Method D 93;

however, results obtained by either test method are acceptable.

14. Color

14.1 The Gardner color method, Test Method D 1544, compares a specimen to glass filters by means of a comparator, while Test Method D 1209 compares the specimen to calibrated liquids in Nessler tubes. It is recommended that Test Method D 1544 (Gardner Color Scales) be used for the color specification where practical. Where particularly light color is required, Test Method D 1209 (platinum-cobalt scale), may be used when agreed upon between the producer and the user. There is no reliable correlation between the two test methods. In general, Test Method D 1209 is to be preferred for light-colored liquids while Test Method D 1544 is generally used for darker liquids, such as drying oils, varnishes, and coating resins.

15. Density

15.1 Test Method D 1475 describes the determination of

density by either the pycnometer or weight cup test methods. Either test method is acceptable for alkyd resins.

16. Viscosity

16.1 The viscosity of resin solutions may be determined by the bubble-time test method, in accordance with Test Method D 1545. Viscosity is measured at 77°F (25°C) by comparing the time for a bubble to traverse a standard tube containing the resin, to the time for standard oils under identical conditions. The bubble seconds are approximately equal to stokes and may be converted to poises by dividing by density.

17. Clarity

17.1 Clarity may be determined in accordance with Test Method D 2090, wherein a specimen is placed in a bubble viscosity tube and rated subjectively for seven factors relating to clarity.

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Standard Test Method for Isophthalic Acid in Alkyd and Polyester Resins¹

This standard is issued under the fixed designation D 2690; the number immediately following the designation indicates the year of original adoption 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 gravimetric determination of the isophthalic acid content of alkyd resins and polyesters. There is no interference from styrene monomer or polymer or from other dicarboxylic acids except terephthalic acid.

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 Document

2.1 ASTM Standard:

D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The resin is saponified with alcoholic potassium hydroxide and benzene to precipitate entirely the potassium salt of the phthalic acid isomer. Since the salt contains entrained impurities, it is dissolved in water and diluted to volume. An aliquot portion is acidified under conditions that release the insoluble acid in filterable form. It is then isolated, weighed, and corrected for its slight solubility in water.

4. Significance and Use

4.1 This test method is used to determine the amount of isophthalic acid contained in alkyd and polyester resins. Use of this test method provides a means whereby the relative applicability of the alkyd or polyester resin to the particular end use may be estimated by the buyer and the seller.

5. Apparatus

5.1 *Flask and Condenser*—A 250-mL Erlenmeyer flask fitted with an air-cooled glass reflux condenser 30 in. (760 mm) in length. The connection between the flask and condenser should be a standard-taper 24/40 ground joint.

5.2 Heat Source:

5.2.1 *Combination Hot Plate and Magnetic Stirrer*, or

5.2.2 *Heating Mantle and Magnetic Stirrer Base and Bar*.

5.3 *Büchner Funnel*, fritted glass, medium porosity, 150-mL capacity.

5.4 *Volumetric Flask*, 100-mL volume.

5.5 *Fritted-Glass Filter Crucible*, medium porosity, 30-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 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

6.3 *Alcohol-Benzene Wash Solution (2+3)*—Mix absolute ethyl alcohol (Note 1) with benzene in the proportion of 2 volumes of alcohol to 3 volumes of benzene.

NOTE 1—The alcohol may be denatured Formula 2-B, but must be anhydrous.

6.4 *Alcoholic Potassium Hydroxide Solution (0.5 N)*—Dissolve 33 g of KOH in 1 L of absolute ethyl alcohol (Note 1) by reflux or by standing overnight. Protect against carbon dioxide absorption. Filter just before use.

6.5 *Benzene* (anhydrous).

6.6 *Ether* (anhydrous).

6.7 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

6.8 *Methyl Purple Indicator Solution*.

7. Hazards

7.1 The reagents and samples used in this test method may, under some conditions, be hazardous. Refer to the manufacturer's material safety data sheets for specific handling and safety precautions. Safe laboratory handling procedures and all applicable OSHA regulations are to be followed.

8. Procedure

8.1 Weigh a portion of resin containing about 400 mg of isophthalic acid (usually 1 to 3 g of vehicle) into a 250-mL Erlenmeyer flask and dissolve in 10 mL of benzene. Add 100 mL of freshly filtered 0.5 N alcoholic KOH solution and insert a magnet stirrer bar. Attach the air condenser and

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.

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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."

reflux for 1½ h with constant stirring by means of a heating mantle or combination stirrer-hot plate.

8.2 Remove from the source of heat, remove the condenser, and fill the flask to the neck with benzene. Cool the solution to room temperature with stirring, allow to settle a few minutes, then filter with suction through a 150-mL fritted glass Büchner funnel of medium porosity. Transfer and wash to bulk of the precipitate with alcohol (2+3). Rinse the flask with about 25 mL of ethyl ether and add to the funnel, draining thoroughly with suction.

8.3 Dry the funnel and flask in a 105°C oven for about 30 min. Dissolve the dipotassium salts in small portions of water by first adding them to the dried flask to remove all traces of the precipitated salts, and then to the funnel collecting them in a clean suction flask. Transfer the filtrate with water to a 100 mL volumetric flask and dilute to the mark.

8.4 Mix the solution thoroughly and withdraw a 50-mL aliquot by means of a pipet and transfer to a 125-mL Erlenmeyer flask. If it is necessary to use smaller aliquots, make the volume up to 50 mL with water. Add a magnetic stirrer bar and about 2 drops of methyl purple indicator solution and while stirring acidify the solution by adding HCl (sp gr 1.19) dropwise until the color changes, then add 7 drops in excess.

8.5 Continue the stirring for about 5 min and collect the precipitated isophthalic acid in a weighed fritted-glass filter crucible of medium porosity. Do not wash or add any water at this time; instead, collect the filtrate in a clean flask and use it in five 10-mL portions to transfer the precipitate quantitatively to the crucible. The filtrate may be reused if necessary. Now wash the collected solution in the crucible twice with 3-mL portions of distilled water added from a pipet and directed against the sides of the crucible while rotating so as to effect maximum washing with the two small measured volumes of water. Dry the crucible in an oven at 105°C for 1 h, cool in a desiccator, and weigh. If the collected isophthalic sample exceeds 200 mg, withdraw a smaller aliquot from the remaining solution, dilute to 50 mL with water and repeat the precipitation procedure. If less than 50

mg are collected, repeat from the beginning with a larger resin specimen.

9. Calculation

9.1 Calculate the percent of isophthalic acid *I* as follows:

$$I = \frac{(P + 0.005) \times 100 \times 100}{SWA}$$

where:

P = weight of precipitate, g,

S = weight of resin specimen, g,

W = nonvolatile content expressed as a decimal, and

A = size of aliquot, mL (usually 50).

10. Precision

10.1 On the basis of an interlaboratory study in which one operator in each of six laboratories made duplicate determinations at each of six laboratories on two samples of different manufacture, the within and between laboratory standard deviations were found to be:

Isophthalic Acid Content, %	Standard Deviation, %	
	Within Laboratory	Between Laboratory
30	0.025	0.18
50	0.083	0.44

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*—Two results obtained by the same operator should be considered suspect if they differ by more than the following:

Isophthalic Acid Content, %	Differences, %
30	0.15
50	0.50

10.1.2 *Reproducibility*—Two results, each the mean of duplicate measurements, obtained by operators in different laboratories should be suspect if they differ by more than the following:

Isophthalic Acid Content, %	Differences, %
30	0.6
50	1.5

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Last ASTM Designation: D 2742 - 79

Standard Methods for Chemical Analysis of Tribasic Lead Phosphosilicate

These methods cover the chemical analysis of the pigment commercially known as tribasic lead phosphosilicate. Formerly under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials, these methods were discontinued in 1990.

Last ASTM Designation: D 2744 - 68 (Reapproved 1979)^{e1}

**Standard Specification for
Tribasic Lead Phosphosilicate**

This specification covers the material known commercially as tribasic lead phosphosilicate.
Formerly under the jurisdiction of Committee D-1 on Paint and Related Coatings and Materials, this specification was discontinued in 1988.



Standard Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements¹

This standard is issued under the fixed designation D 2745; the number immediately following the designation indicates the year of original adoption 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 relative tinting strength of white pigments by reflectance measurements of black tints.

1.2 This test method is applicable only for comparing the test pigment with a reference standard of the same type and grade.

NOTE 1—Test Method D 332 describes a procedure for visual assessment of blue tinted samples.

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 332 Test Method for Relative Tinting Strength of White Pigments by Visual Observation²

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 Pigment is dispersed in a vehicle and let down with additional vehicle that has been tinted. Dispersion and let-down are accomplished with a mechanical muller. Both the test and standard pigments are treated identically. Opaque drawdowns are made of the paint and the Y tristimulus values (green-filter reflectance) of the wet films are measured. The relative tinting strength of the test pigment is calculated directly from the reflectance values.

4. Significance and Use

4.1 Tinting strength is one of the most important properties of a white pigment. This test method provides a means of testing this property for quality control.

4.2 This test method is a referee method, and the vehicle for preparing the dispersion and the black for tinting are suggested but others may be used provided both the pur-

chaser and the seller agree to the changes.

4.3 The results obtained with a muller do not necessarily agree with industrial situations where different dispersing conditions exist. However, dispersing with a muller is a fast, relatively inexpensive way of testing tinting strength for routine quality control.

5. Apparatus and Materials

5.1 *Muller*, automatic,⁴ equipped with a weight that exerts a permanent 50-lbf (220-N) and an additional weight exerting a 50-lbf making a total of 100-lbf (445-N). The two glass plates shall be kept sharp by removing from the machine and grinding them face-to-face with No. 303 optical emery or equivalent, and water.

5.2 *Spatula*—A flexible spatula having a chromium-plated or plastic blade 3 to 6 in. (75 to 150 mm) long and another with a 3-in. tapered blade.

5.3 *Balances*—(1) A balance sensitive to 20 mg and (2) an analytical balance sensitive to 0.4 mg.

5.4 *Vehicle*—Because the choice of vehicle may affect results, a solvent-free vehicle (excluding refined or low-bodied linseed oil)⁵ should be agreed upon by the purchaser and the seller.

5.5 *Tinting Black*—A lamp black predispersed in a vehicle similar in nature to the test vehicle.⁶

5.6 *Chart*—Either gray or white lacquered charts⁷ cut to a convenient size.

5.7 *Colorimeter*—A filter colorimeter meeting the requirements of Test Method E 97.

5.8 *Film Applicator*—2 or 3 in. (50 or 75 mm) wide with a clearance of 6 mils (150 μ m) to produce wet films about 3 mils (75 μ m) thick. This applicator when used as suggested in the test method will produce an R_{∞} film.⁸

6. Standard

6.1 A standard white pigment of the same type as the

⁴ A satisfactory muller is supplied by the Hoover Color Corp., 13 Cordier St., Irvington, NJ 07111.

⁵ Suggested vehicles include a 50-50 mixture of Aroplaz 1271 and 1278 (Spencer Kellogg Division of Textron, Inc., P.O. Box 807, Buffalo, NY 14240), No. 3 Litho Varnish (Inmont Corp., Printing Ink Div., Germantown Ave. and New Market St., Philadelphia, PA 19123), and Castor Oil AA, USP (NL Industries, NL Chemicals Div., P.O. Box 700, Hightstown, NJ 08520).

⁶ Tinting black should be agreed upon between the purchaser and the seller. Suggested materials include Lampblack PF 4340 (Daniel Products Co., 400 Claremont Ave., Jersey City, NJ 07304) and Lampblack 5-24-A-710 (The Hilton-Davis Chemical Co., 2235 Langdon Farm Road, Cincinnati, OH 45237).

⁷ Suitable charts are available from The Leneta Co., P.O. Box 86, Ho-Ho-Kus, NJ 07423.

⁸ A Bird Film Applicator, available from Bird and Sons, Walpole, MA 02081, 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 D01.26 on Optical Properties.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 2745 - 68 T. Last previous edition D 2745 - 80.

² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

TABLE 1 Examples of Preparation of Black Letdown Vehicles

Pigment Type	Approximate Pigment Density	Pigment, g	Clear Vehicle, g	Black Letdown Vehicle, g	Approximate PVC of Final Paint, %	Predispersed Black, ^a g	Clear Vehicle, ^a g
Titanium dioxide:							
rutile	4.1	3.000	2.000	1.000	20	6.80	93.20
anatase	3.8	3.000	2.250	1.000	20		
Lithopone	4.3	5.000	1.220	1.000	35	2.25	250.00
Zinc oxide, leaded	5.9	5.000	2.460	1.000	20		
Zinc oxide	5.6	5.000	2.660	1.000	20		
White lead	6.8	3.000	0.810	1.000	20		

^a Daniel Products Co. Lampblack F is used for this example.

^b A 50-50 (by weight) composite vehicle, Aroplaz 1271 and 1278, is the "vehicle" used for this example.

sample to be tested, as agreed upon by the purchaser and the seller.

7. Procedure

7.1 Place two concentric circles under the base plate of the muller so that they can be clearly seen through the plate. The circles can be drawn on a paper and inserted under the plate, or they can be drawn directly on the underside of the bottom plate. The inner circle should be 2½ in. (63 mm) in diameter and the outer circle 4½ in. (114 mm) in diameter.

7.2 Make a preliminary test by dispersing standard white pigment in a clear vehicle at the pigment volume concentration (PVC) given in Table 1 and adding tinting black to determine the amount of black required to provide a reflectivity of 0.42 ± 0.03 . Prepare a "black letdown vehicle" by adding tinting black to clear vehicle so that 1.000 g of the black letdown vehicle will contain the same amount of black that provided this reflectivity. Three examples are given in Table 1.

7.3 Weigh the pigment to the nearest g with the analytical balance in accordance with Table 1 and transfer to the lower plate of the muller. Add clear vehicle to the pigment in accordance with Table 1 (Note 2). Mix with a spatula until a paste is obtained. "Pick up" the paste with the spatula and spread it within the area between the two concentric circles on the plate. Close the muller, mull 25 times, and collect the paste. Thoroughly mix the black letdown vehicle in its container. Add 1.000 g of black letdown vehicle as specified in Table 1 to the white paste and mix in thoroughly with a spatula. Again spread the paste within the two concentric circles, mull 25 times, pickup the paste, spread within the area, and mull a final 25 times more. Prepare a reference standard with each series of test pigments by this same procedure.

NOTE 2—An analytical balance is specified for referee-type of testing; for routine work, weighing to 20 mg has been found satisfactory as an alternative procedure. Among the methods of accurately weighing vehicles (either the clear or black letdown vehicles) are these two alternatives: (1) Place a small beaker of vehicle on the weighing pan of the balance. Remove and transfer vehicle employing the spatula ultimately intended for preparing a paste on the muller. Use the weigh-by-difference method. (2) Add the vehicle to a tared metal or plastic plate till the correct weight is reached; transfer to the muller by scraping off the plate with the spatula. If the vehicle tends to run, weigh both pigment and vehicle on the plate making a crater in the pile of pigment to contain the vehicle.

7.4 Standardize the colorimeter for measurement of the green filter reflectance (that is, Y-value) with, if necessary, a wet-film adapter in place. Adjust the colorimeter to the

approximate expected reading. Draw down the sample paint on the chart (lacquered side) with the applicator blade (5.8). Place the film immediately on the reading port, cover the back of the chart with a flat, black panel (for example, black structural glass) to keep the chart flat and prevent exterior light from entering the colorimeter, and read the reflectance immediately (Note 3), recording the value. Clean the port of the colorimeter thoroughly with a rag. Draw down the standard paint and read immediately as before (Note 4).

NOTE 3—Flooding will cause drifting in the reflectance reading of some films. When the colorimeter is already adjusted to the expected reading, an experienced operator can read the reflectance with high precision in a very short interval. Consequently the time lapse between draw-down and reflectance reading should be as short as possible.

NOTE 4—Although the method is primarily intended as a rapid method, it obviously can be adapted to measuring the tinting strength in a dry film of paint. For this an appropriate amount of cobalt drier is added to the black letdown vehicle during its preparation (see 7.2), and the films are allowed to dry as needed before making the reflectance measurements. It should be cautioned that tinting-strength values measured on dry films may differ from those measured on wet films.

8. Calculation of Tinting Strength

8.1 Calculate the tinting strength as follows:

$$TS = [(1 - R_{\infty})^2 / 2R_{\infty}]_u / [(1 - R_{\infty})^2 / 2R_{\infty}]_s (T)$$

where:

TS = tinting strength of test pigment;

R_{∞} = measured reflectant factor (as a decimal),

T = assigned tinting strength of standard, usually 100 %, and subscripts "u" and "s" refer to the test and standard pigments, respectively.

9. Report

9.1 Report the following information:

9.1.1 Tinting strength rounded to three significant figures,

9.1.2 Identification of the pigment, the reference standard, and the arbitrary assigned value, T_r , of the tinting strength for the reference standard. (Also identify the vehicle and colorant employed.), and

9.1.3 Name and model number of the colorimeter employed.

10. Precision

10.1 On the basis of an interlaboratory test of this test method in which seven laboratories tested four materials covering a broad range in tinting strength using five types of colorimeters, the standard deviation was found to be 1.16 within laboratories and 1.72 among laboratories. Based on this, the following criteria should be used for judging the acceptability of results at the 95% confidence level when the

standard is assigned a value of 100:

10.1.1 Repeatability—Two results obtained by the same operator on the same pigment should be considered suspect if they differ by more than 3.3 units.

10.1.2 Reproducibility—Two reported results, obtained by operators in different laboratories, should be considered suspect if they differ by more than 4.9 units.

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Standard Test Method for Sulfur Content of Cellulosic Materials by X-ray Fluorescence¹

This standard is issued under the fixed designation D 2929; the number immediately following the designation indicates the year of original adoption 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 sulfur content of cellulosic materials by X-ray fluorescence.

1.2 Using appropriate standards, the range of the procedure is from approximately 10 ppm to 20 % sulfur.

1.3 This test method is proposed specifically as an alternative to Methods D 871, Sections 29 to 33, and Methods D 817, Sections 39 to 43. As applied to cellulose esters it measures the combined sulfur and sulfur in the accompanying inorganic salts.

1.4 To determine combined sulfur, the sample, when soluble, must first be reprecipitated into dilute acid to remove the noncombined sulfur compounds.

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 Note 3.*

2. Referenced Documents

2.1 ASTM Standards:

D 817 Methods of Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate²

D 871 Methods of Testing Cellulose Acetate²

3. Summary of Test Method

3.1 The sulfur content of cellulose, cellulose modification, or cellulose derivative is determined by measuring the intensity of the secondary sulfur $K\alpha$ X rays emitted on irradiation of the sample with primary X rays of higher energy from an X-ray tube with a target of tungsten or chromium. The sulfur $K\alpha$ radiation is diffracted with a suitable analyzing crystal and detected with a flow proportional counter. The entire path of the secondary radiation is purged with hydrogen or helium, or evacuated to a pressure of 0.5 mm Hg or less. The intensity of the sulfur $K\alpha$ rays, as established by a standard counting period and corrected for background radiation, is then converted to percent sulfur from calibration data.³

4. Significance and Use

4.1 This procedure provides a method for determining sulfur content in cellulosic materials by nondestructive means. Sulfur may be in the form of sulfate esters that may contribute to thermal instability. Sulfur can also be present as salts that can cause haze in solutions.

5. Apparatus

5.1 *Wiley Mill*, equipped with 60-mesh screen.

5.2 *Sample Mold*—Chrome steel die of a size depending on the sample holder to be used.

5.3 *Laboratory Press*, capable of exerting at least 5000 psi.

5.4 *X-Ray Spectrograph*, with following equipment: tungsten or chromium target X-ray tube; hydrogen or helium purging system or vacuum system to reach 0.5 mm Hg (or less, if desired); 20-mil Soller slits; flow proportional counter with 90 % argon-10 % methane gas mixture; and NaCl, ethylenediamine dextro tartrate (EDT), or pentaerythritol (PET) analyzing crystal.

NOTE 1—Radiation from a chromium target tube appears to be more effective than radiation from a tungsten target tube for exciting sulfur $K\alpha$. This may be because a larger part of the "white" radiation of the chromium tube is at the longer wavelength region of the spectrum, or because of the use of thinner windows in the chromium tube. However, either the chromium or the tungsten target tube is suitable for sulfur analysis. If a pulse height analyzer is available as a part of the X-ray instrumentation, its use is very helpful in reducing background radiation. Use of a pulse height analyzer is mandatory for the determination of sulfur below 100 to 200 ppm level.

6. Reagents

6.1 *Ethyl Cellulose Phthalate*.⁴

6.2 *Microcrystalline Cellulose*.⁵

6.3 *Cystine*, NIST primary standard No. 143B.⁶

7. Procedure

7.1 *Instrument Standardization Pellet*—Intimately mix ethyl cellulose phthalate, microcrystalline cellulose, or other selected matrix with finely divided cystine in a weight ratio of approximately 4:1. Press a portion into a pellet in the sample

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 D01.36 on Cellulosics.

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

³ This test method is compiled from four techniques, by incorporating certain features of each, as follows: "Sulfur in Cellulose Esters by X-ray Emission Spectroscopy," Eastman Kodak Co.; "Sulfur in Cellulose Acetate by X-ray Fluorescence," Tennessee Eastman Co.; "X-ray Fluorescence Analysis of Modified

Cottons" by Tripp, Piccolo, Mitcham and O'Connor. *Textile Research Journal*, Vol 34, 1964, p. 773, and FMC Corp., American Viscose Div. information furnished by L. H. Phifer and W. B. Swann.

⁴ Ethyl cellulose phthalate is available from Distillation Products Industries, Rochester, NY 14650, as Eastman Organic Chemical No. 7484.

⁵ Microcrystalline cellulose is available from FMC Corp., American Viscose Div., Avicel Sales, Marcus Hook, PA 19061, as Avicel PH-101.

⁶ Available from National Institute of Standards and Technology, Washington, DC 20899. Eastman White Label or similar grade, available from Eastman Kodak Co., 343 State St., Rochester, NY 14650, is generally sufficiently pure and may be substituted for the NIST material.

hold for 30 s using a pressure of 5000 lb on the mold. The thickness of the sample pellet should be $\frac{1}{16}$ in. or greater.

7.2 Standardization of Instrument:

NOTE 2—Basic standardization techniques and operating parameters vary from instrument to instrument. The manufacturer's literature and the analyst's knowledge of the performance characteristics of the equipment should be used as a guide in performing this function.

7.2.1 Place a standardization pellet in the sample holder and center it so that it covers the entire window of the holder. Evacuate the X-ray path to a pressure of 0.5 mm Hg (or less, if desired) or flush the path with hydrogen or helium. Set the gas flow of the proportional counter as suggested by the manufacturer.

NOTE 3: Precaution—X rays are very hazardous. The equipment should not be turned on until the instruction manual has been read and understood.

7.2.2 Set the goniometer of the X-ray spectrograph at the appropriate sulfur $K\alpha$ angle. Turn the X-ray tube on, using the manufacturer's recommended voltage and current. Selection of the operating voltage and current of the X-ray tube is governed by the type of tube used. Take care not to exceed the rated power output (in watts) of the X-ray tube. Make a plot of the sulfur X-ray intensity versus applied voltage to the detector. The voltage chosen should be approximately midway on the plateau.

7.2.3 If a pulse height analyzer is being used, a common way to arrive at the proper settings is to set the gain of the linear amplifier at approximately half of its maximum. Select the detector voltage about midway on the plateau. Set the minimum pulse rejection voltage at about 5 V and the ΔE at about 10 V. Observe the intensity without the pulse height analyzer on. With the pulse height analyzer on, change either the amplifier gain control or the detector voltage until as near as possible the same intensity as without the analyzer is obtained. Then increase or decrease the ΔE control just until a maximum intensity is reached.

7.3 Calibration for Measurement of Sulfur:

7.3.1 Select five or more representative cellulosic samples whose sulfur contents, as determined by chemical analysis according to Methods D 817 or D 871, completely cover the required range. Since X-ray fluorescence intensities are functions of concentrations per unit volume, prepare standards used to establish the calibration curve in a matrix as nearly identical to that of the sample as possible. The sulfur-containing component should be distributed very uniformly in the matrix. The physical form of the matrix (powder, flake, pellet, or sheet) is a significant variable. For accurate work it may be necessary to have separate calibrations for each form of sample.

7.3.1.1 As a calibration standard for cellulose, microcrystalline cellulose containing intimately mixed known amounts of cystine may be used. A chemical analysis of the standards is generally not necessary.

7.3.2 Prepare the samples for analysis according to 7.4 and 7.5.

7.3.3 Measure the intensities of the sulfur $K\alpha$ X rays of the standard samples according to 7.6.

7.3.4 Prepare a calibration curve relating sulfur $K\alpha$ X-ray intensity expressed as net counts per second to percent sulfur.

7.4 Treatment of Sample Prior to Analysis:

7.4.1 To analyze for total sulfur content, no treatment is required prior to analysis. If the sample has a high absorbing coating (for example, saran, PVC, etc.) these should be removed or a standard containing a matrix of those materials in equivalent concentrations must be prepared.

7.4.2 When analyzing for combined sulfur content, as in a cellulose ester, remove the uncombined sulfur as follows:

7.4.2.1 Dissolve 25 g of sample in approximately 300 mL of acetone, depending on the viscosity. If the sample is of too high acetyl content to be directly soluble in acetone, cool in a dry ice cabinet overnight, then allow to come to room temperature while tumbling or stirring.

7.4.2.2 Filter the solution, if necessary, through felt or a coarse sintered-glass crucible.

7.4.2.3 Precipitate with rapid stirring into a beaker or pail containing 2 to 3 L of acetic acid-water (1:49).

7.4.2.4 Filter through a cloth bag or a Büchner funnel and give two 15-min washes with water using mechanical agitation. A little Na_2CO_3 may be added to the last wash to stabilize samples of high sulfur content.

7.4.2.5 Filter and dry overnight at 60°C.

7.5 Preparation of Specimen for Analysis:

7.5.1 Grind the sample in a Wiley Mill to pass a 60-mesh screen. Mold a quantity of the sample for 30 s with a gage pressure to 5000 lb on the mold. Use sufficient sample to obtain a disk at least $\frac{1}{16}$ in. thick.

7.5.2 Film specimens can be run directly (without grinding) by using a multiplicity of sheet thicknesses provided that the specimen contains no air trapped between the successive layers. If film specimens are coated with a high absorbing coating (for example, saran, PVC, or aluminum), these must be removed if the specimens are to be analyzed as multiple thicknesses. Similarly, a closely wrapped specimen of continuous filament yarn can also be tested directly.

7.6 Measurement of Intensity of Sulfur $K\alpha$ X rays:

7.6.1 Place the specimen in the sample holder of the X-ray spectrograph and center so that it covers the entire sample-holder window.

7.6.2 Evacuate the X-ray path to a pressure of 0.5 mm Hg or less or flush the path with hydrogen or helium.

7.6.3 Record the X-ray count for a standard time period at the sulfur $K\alpha$ angle.

7.6.4 Repeat the count on the opposite side of the specimen pellet if homogeneity is questioned.

7.6.5 If a sulfur-free specimen is available, record the background count. This is unnecessary if the standard's matrices are identical to the samples.

NOTE 4—A background count is desirable at all concentration levels. It is mandatory for measurement of sulfur concentrations under 100 ppm.

NOTE 5—An indication of the precision of this test method is given by the standard counting error, which is:

$$s_c = \sqrt{N_T} + \sqrt{N_B}$$

where:

s_c = standard counting error of $N_T - N_B$,

N_T = counts accumulated at sulfur $K\alpha$ angle during counting interval Δt , and

N_B = counts accumulated at background during same counting interval Δt .

This error level calculation should be used as a guide to counting time.

8. Calculation

8.1 Convert the standard time count values of the spec-

imen and the background to counts per second.

8.2 If the background is measured subtract the background count rate from the count rate of the specimen pellet to obtain the net count rate of the specimen.

8.3 Convert the net count rate of the specimen to percent sulfur by using the calibration curve.

9. Precision and Bias

9.1 The precision of measurement, or standard deviation, σ , is calculated by normal statistical methods as follows.

$$\sigma = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

where:

x = measured value,

$\bar{x}$ = mean value of n measurements, and

n = number of measurements.

9.1.1 The practical significance of σ is that 68 % of all results should be within $\pm 1\sigma$ of the mean, 95 % with $\pm 2\sigma$ and 99.7 % within $\pm 3\sigma$.

9.1.2 Since random processes are involved in x-ray analysis, precision can also be determined from the fact that standard deviation is equal to the square root of the number of counts, N , collected.

$$\sigma N = \sqrt{N}$$

9.1.3 It may be more relevant to express precision in terms of the coefficient of variation, E which is the standard deviation as a percent of the number of counts as follows:

$$E = \frac{\sqrt{N}}{N} \times 100 = \frac{100}{\sqrt{N}}$$

9.2 Bias has not been evaluated for this test method.

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Standard Test Method for Polyhydric Alcohols in Alkyd Resins¹

This standard is issued under the fixed designation D 2998; the number immediately following the designation indicates the year of original adoption 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 and quantitative determination of the polyols in alkyd resins, including resin- and polymer-modified alkyds. Quantities as low as 0.5 % may be detected and measured.

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:

D 1193 Specification for Reagent Water²

E 260 Practice for Packed Column Gas Chromatography³

3. Summary of Test Method

3.1 The resin sample is dried, subjected to aminolysis to release the polyols, treated directly to form the trimethylsilyl (TMS) ether derivatives of the polyhydric alcohols which are separated by gas-liquid chromatography. An internal standard is used for quantitative purposes. The description of apparatus and the recommended operating conditions are presented as a guide to a satisfactory analysis as all suitable variations in temperatures, column lengths, substrates, liquid phases, programming rates, etc., have not been determined.

4. Significance and Use

4.1 This test method is designed to identify polyhydric alcohols and to quantify amounts of polyols contained in alkyd resins. Polyols are significant ingredients in alkyd resins and impact on such important physical parameters of alkyd paints as weatherability, flexibility, moisture resistance, and adhesion. Use of this test method will provide a means whereby the relative applicability of the alkyd resin to the particular end use may be estimated by the buyer and the seller.

5. Apparatus

5.1 *Linear Programmed Temperature Gas Chromatograph*—Any instrument with a thermal conductivity de-

detector and with programming features should be suitable if adaptable to the following recommended operating conditions:

Detector cell temperature, °C	300
Detector cell current, mA	160
Injection port temperature, °C	300
Helium flow at exit, cm ³ /min	80
Programmed temperature details:	
Approximate column heating rate, °C/min	6
Starting column temperature, °C	100
Finishing column temperature, °C	300

NOTE 1—Flame ionization detectors are not recommended for this test as the reaction medium can contaminate the flame jet.

5.2 *Column*—A 16-ft (4.9-m) length of 1/4-in. (6.4-mm) outside diameter copper tubing packed with 20 weight percent of silicone grease⁴ on 60 to 80-mesh flux-calcined diatomaceous earth,⁵ and conditioned at 315°C until "bleeding" reaches a minimum.

5.3 *Column and Instrument Performance*—Note the separation of closely adjacent polyol peaks and express as peak resolution R as follows:

$$R = 2Y/(A + B)$$

where:

Y = distance between maxima of closely adjacent peaks,

A = base width of first peak A , and

B = base width of peak B .

NOTE 2—If the peak resolution is equal to or greater than 1.0, the column and instrument are in satisfactory condition.

5.4 *Syringe*, having a fixed needle, 50- μ L capacity.

5.5 *Erlenmeyer Flask*, 125-mL, with standard joint and water-cooled condenser to fit.

5.6 *Micro Test Tube* or screw cap vial.

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

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.

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² Annual Book of ASTM Standards, Vols 06.03 and 11.01.

³ Annual Book of ASTM Standards, Vol 14.01.

⁴ DC-11 manufactured by Dow-Corning, Midland, MI 48686, or GE SE-30 manufactured by General Electric Silicon Products Div., Waterford, NY 12188, has been found satisfactory for this purpose.

⁵ Chromosorb W available from Manville Sales Corp., Filtration and Minerals, P.O. Box 5108, Denver, CO 80217-5108, has 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."

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 II of Specification D 1193.

6.3 *Bis(trimethylsilyl)trifluoroacetamide*.⁷

6.4 *1,4-Butanediol*.

6.5 *n-Butylamine*.

6.6 *Hexamethyldisilazane (HMDS)*.

6.7 *Methylene Chloride*.

6.8 *Mixed TMS Reagent*—Mix 20 parts by volume of the bis(trimethylsilyl)trifluoroacetamide with 80 parts of the hexamethyldisilazane and protect from moisture. If stored at 60°C in an oven, the reagent remains active and stable.

7. Hazards

7.1 The reagents and samples used in this test method may, under some conditions, be hazardous. Refer to the manufacturer's Material Safety Data Sheets for specific handling and safety precautions. Safe laboratory handling procedures and all applicable OSHA regulations are to be followed.

8. Procedure

8.1 Weigh a specimen of resin containing approximately 2 g of nonvolatile material into the 125-mL flask. Add 3 mL of methylene chloride, mix, and evaporate the solvents in a 60°C bath with a current of air. Repeat the drying twice, dissolving the resin each time in 3 mL of methylene chloride. Check for odor of residual solvent and, if necessary, dissolve and dry a fourth time.

8.2 Weigh accurately about 200 mg of 1,4-butanediol (Note 3) as standard into the flask followed by 10 mL of butylamine added from a pipet. Reflux under the water condenser for 2 h, add 0.5 mL of water to the flask through the condenser, and continue refluxing for 2 additional hours.

NOTE 3—Other polyhydric alcohols may be substituted for the 1,4-butanediol standard if sufficiently pure.

8.3 Place 5 drops of the aminolysis mixture in a micro test tube, add 0.5 mL of the mixed TMS reagent, and mix by shaking. Heat for 30 min at 60°C in an oven. Inject 40 µL onto the silicone grease column of the chromatograph and follow the operating conditions recommended in 5.1. Hold the temperature at 300°C at the completion of the program until all the volatile components have emerged as shown by the chromatogram.

9. Identification

9.1 The peaks on the chromatogram for the polyols can be identified by their relative retention times given in Table 1, if the recommended operating conditions are followed. Calibration is always recommended and can be made by

TABLE 1 Relative Retention Time of Trimethylsilyl Ethers

Polyol	Retention Time Relative to 1,4-Butanediol
Ethylene glycol	0.58
Propylene glycol	0.63
2,3-Butanediol	0.73
1,3-Butanediol	0.83
Neopentyl glycol	0.86
1,4-Butanediol	1.00
Diethylene glycol	1.19
Glycerol	1.29
Trimethylol ethane	1.46
Trimethylol propane	1.63
Triethylene glycol	1.83
Pentaerythritol	1.88

applying the procedure to a synthetic mixture of polyols.

10. Calculation

10.1 Relate the height of each polyol peak to the height of the known amount of internal standard. Establish correction factors by dissolving known amounts (0.1 to 0.5 g) of the various polyols and internal standard in butylamine by warming, and proceeding as in 8.3 for derivative formation and chromatographing. For additional information on calibration and standardization, see Section 9 of Practice E 260.

10.2 Calculate the percent polyol *C* as follows:

$$C = (A \times F \times P) / (I \times S \times N)$$

where:

A = height of polyol peak, mm,

F = correction factor,

P = weight of internal standard, g × 100,

I = height of standard peak, mm,

S = weight of resin specimen, g, and

N = nonvolatile fraction.

NOTE 4—The nonvolatile fraction *N* may be omitted from the calculation if the determination is preferred on a solution basis.

11. Precision

11.1 On the basis of an interlaboratory test of this test method in which one operator in six laboratories analyzed two alkyd resins containing 14.0 and 11.1 % glycerol, 6.8 and 7.2 % pentaerythritol, and 4.2 % ethylene glycol, the within- and between-laboratory standard deviations were found to be as given in Table 2. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at a 95 % confidence level:

11.1.1 *Repeatability*—Results obtained by the same analyst in two successive determinations should be considered suspect if they differ by more than the values given in Table 2.

TABLE 2 Precision of Test Method

Polyol	Degrees of Freedom	Repeatability		Reproducibility	
		Standard Deviation	Least Significant Difference	Standard Deviation	Least Significant Difference
Ethylene glycol	6	0.125	0.44	0.472	1.63
Glycerol	12	0.212	0.65	0.731	2.25
Pentaerythritol	12	0.137	0.42	0.298	0.92

⁷ Bis(trimethylsilyl)trifluoroacetamide is available from the Regis Chemical Co., 1101 N. Franklin St., Chicago, IL 60610, catalog number RC-1 and Pierce Chemical Co., Box 117, Rockford, IL 61105.

1.1.2 *Reproducibility*—Two results, each the mean of duplicate measurements, obtained by analysts in different laboratories should be considered suspect if they differ by more than the values given in the Table 2.

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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 Phthalocyanine Green Pigments¹

This standard is issued under the fixed designation D 3021; the number immediately following the designation indicates the year of original adoption 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 4.1 was editorially changed in May 1987.

1. Scope

1.1 This specification covers chlorinated and chlorinated-brominated copper phthalocyanine green pigments in dry powder form, for use in paints, printing inks, and related products. A variety of commercial types are available to meet the requirements of different end uses.

2. Referenced Documents

2.1 ASTM Standards:

- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1135 Test Methods for Chemical Analysis of Blue Pigments²

3. Composition and Properties

3.1 The pigments shall consist of the product known commercially as phthalocyanine green, with or without phthalocyanine blue or other ingredients incorporated during manufacture to improve or alter the properties of the pigment, but free of any other coloring matter either organic or inorganic. The pigment shall conform to the following requirements:

Moisture and other volatile matter, max, %	3.0
Coloring matter other than phthalocyanines	none

3.2 *Mass Color and Character of Tint*—The mass color, and the character of the tint formed by mixture with a white pigment, and the strength, shall be within mutually agreed-upon limits of a standard acceptable to both the purchaser and the seller.

3.3 *Oil Absorption*—The oil absorption shall be between 90 and 110 % of that of a reference sample mutually agreed upon between the purchaser and the seller.

3.4 *Reaction in Identification Tests*—The pigment shall show the same reaction in identification tests (5.5 to 5.8) as a reference sample mutually agreed upon between the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 1000 lb, two samples shall be taken. At the option of the purchaser, each sample may be tested or samples from the same production may be blended in equal quantities to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Moisture and Other Volatile Matter*—Method A of Test Methods D 280.

5.3 *Mass Color and Tinting Strength*—Test Method D 387.

5.4 *Oil Absorption*—Test Method D 281.

5.5 Identification:

5.5.1 To about 50 mg of the sample in a 50-mL beaker, add 30 mL of H₂SO₄ (sp gr 1.84). Stir occasionally for 15 min, heating if necessary. A dark greenish yellow to a dark reddish color forms, depending on the degree of bromination. Pour the solution into 250 mL of water and stir. The phthalocyanine green will immediately precipitate as a flocculent mass.

5.5.2 Filter off the precipitate, washing once or twice with water. Scrape a small amount of the precipitate off the filter, place on a clean platinum wire moistened with HCl, and subject it to the low flame of a bunsen burner. As the precipitate burns, a light blue-green flame should be clearly evident, indicating organically combined copper.

NOTE—Characteristic spectrophotometric absorption spectra in the near infrared range (700 to 900 nm) are exhibited by dilute solutions of copper phthalocyanine pigments (2 to 50 mg/L) in H₂SO₄ (sp gr 1.84). The absorption maxima are so sharp and well defined that they may be used for positive qualitative identification of the various phthalocyanine pigments. Phthalocyanine green has maxima at approximately 820 and 860 nm for bromine-free types; for the chlorinated-brominated types, the maxima appear at approximately 830 to 840 nm, and 875 to 885 nm.

5.6 *Basic Dye Derivatives*—Test Method D 1135.

5.7 *Other Organic Coloring Matter*—Test Method D 1135.

5.8 *Chrome Green and Iron Blue*—Test Method D 1135.

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 D01.31 on Pigment Specifications.

Current edition approved June 25, 1982. Published August 1982. Originally published as D 3021 - 72. Last previous edition D 3021 - 72 (1976).

² Annual Book of ASTM Standards, Vol 06.02.

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Standard Test Method for Color and Strength of Color Pigments by Use of a Miniature Sandmill¹

This standard is issued under the fixed designation D 3022; the number immediately following the designation indicates the year of original adoption 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 March 1989.

1. Scope

1.1 This test method covers the determination, through the use of a miniature sandmill, of the color and strength of dry color pigments.

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.*

2. Referenced Documents

2.1 ASTM Standards:

- C 778 Specification for Standard Sand²
- D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)³
- D 523 Test Method for Specular Gloss⁴
- D 2244 Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates⁴
- E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁵
- E 308 Test Method for Computing the Colors of Objects by Using the CIE System⁶

2.2 Federal Specifications:⁷

TT-R-266 Resin, Alkyd; Solutions

3. Significance and Use

3.1 This test method is a way of testing the color and strength of pigments by use of a miniature sandmill. It

correlates well with industrial practice and is used for routine quality control.

4. Apparatus

4.1 *Balance*, sensitive to 10 mg with a capacity in excess of 300 g.

4.2 *Miniature Sandmill*—A laboratory disperser equipped with a 1½-in. (41-mm) diameter fiber rotary disk impeller rotating at a constant 8000 r/min under varying load conditions. The shaft upon which the impeller is mounted shall be sufficiently balanced so no whip of the shaft is observed between 0 and 10 000 r/min.⁸

4.3 *Cylinder*, 100-mL graduated.

4.4 *Beakers*, 200-mL tall-form, stainless steel, or polyethylene (approximately 60-mm inside diameter).

4.5 *Strainers*, paper cone, disposable, about 40 mesh.

4.6 *Fiber Disks*, 1½-in. (41-mm) diameter phenolic laminated, ¼ in. (6 mm) thick.⁹

4.7 *Paper Charts*, smooth, surface-coated, the surface of which should be impervious to paint liquids.¹⁰

4.8 *Film Applicator*, with an 8-mil (200-μm) clearance at least 3 in. (75 mm) wide.

4.9 *Color-Measuring Instruments*, as defined in Method E 97 or Practice E 308.

5. Materials

5.1 Samples of standard reference pigments as agreed between the purchaser and the seller.

5.2 *Standard Sand*—Regular 20 to 30-mesh (850 to 600-μm) cement testing sand conforming to Specification C 778. The sand shall be screened to remove all sub 30-mesh particles. Twenty to thirty-mesh glass beads may be used instead of sand.¹¹

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 April 27, 1984. Published July 1984. Originally published as D 3022 - 72. Last previous edition D 3022 - 72 (1978).

² Annual Book of ASTM Standards, Vol 04.01.

³ Annual Book of ASTM Standards, Vol 06.03.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vols 06.01 and 14.02.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094.

⁸ The Sherwin-Williams miniature sandmill has been found satisfactory for this purpose. Drawings are available from ASTM Headquarters. Order PCN 12-430-220-14.

⁹ Suitable disks are available from BYK-Gardner, Inc., Gardner Laboratory, 2435 Linden Lane, Silver Spring, MD 20910.

¹⁰ White and black charts available from The Leneta Co., P. O. Box 576, Ho-Ho-Kus, NJ 07423 have been found satisfactory for this purpose. Equivalent charts may be used.

¹¹ Sand is available from Agsco Division, American Graded Sand Co., 189 E. 7th St., Patterson, NJ 07524. Glass beads are available from Quackenbusch Co., P. O. Box 607, Palatine, IL 60067.

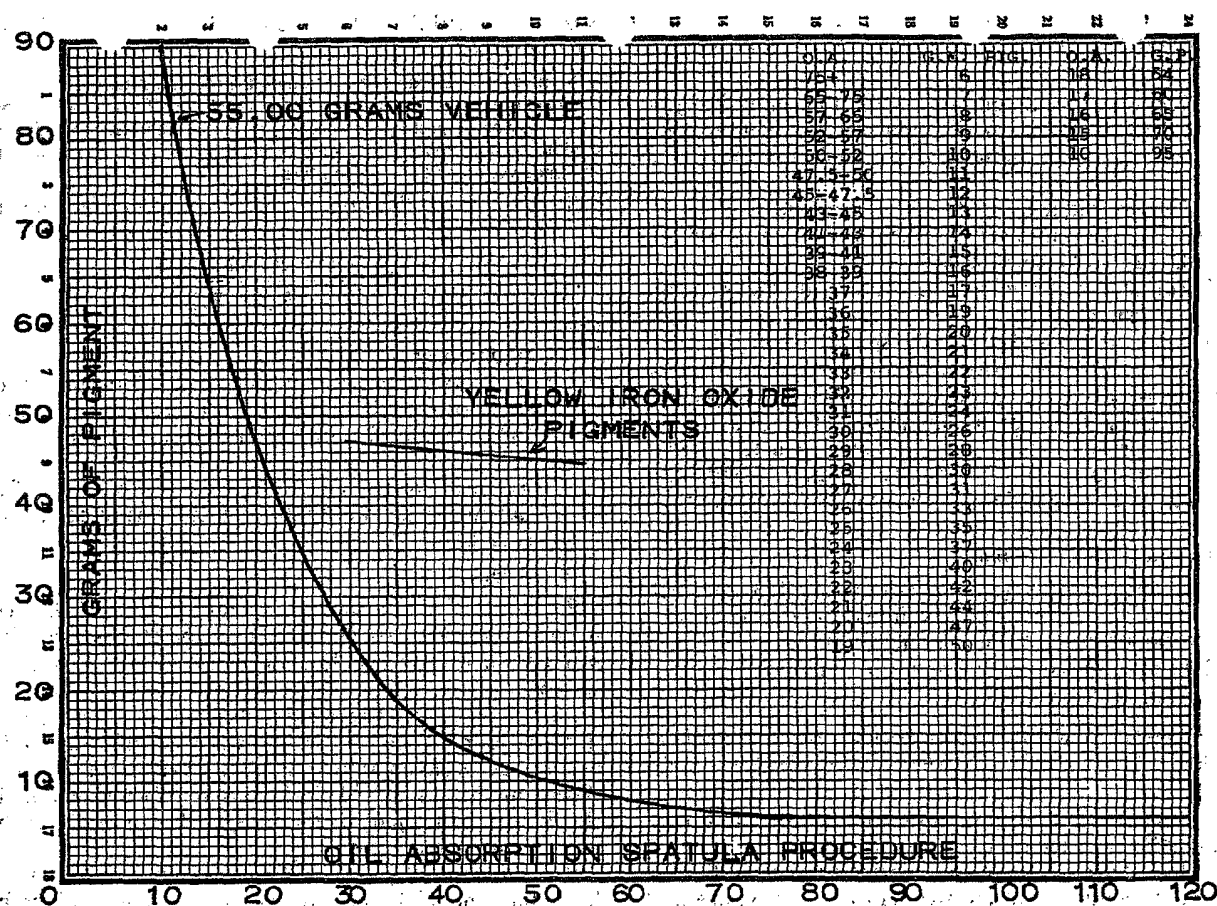


FIG. 1 Oil Absorption Spatula Procedure

5.3 *Grinding Vehicle*—A long oil alkyd¹² meeting U.S. Fed. Spec. TT-R-266 Type 1-A reduced to 47 % solids with mineral spirits conforming to Specification D 235 (2 parts alkyd and 1 part mineral spirits by weight).

5.4 *White Tinting Paint*—A flat white tinting paint compatible with the dispersion vehicle meeting the following requirements.

5.4.1 *Gloss* (60°) less than 4, as determined by Test Method D 523.

5.4.2 *Contrast Ratio* (2-mil (51-μm) dry film): 99.2 min.

5.5 *Drier Blend*—One part 6 % manganese naphthenate, 2 parts 6 % cobalt naphthenate, and 4 parts 24 % lead naphthenate by weight.

6. Hazards

6.1 While operating the mill, keep hands well away from the shaft and disk and be sure that no article of clothing (for example, necktie or long hair) will catch on moving parts. This precaution applies to all cases where the mill is in operation.

¹² A long oil soya or safflower alkyd at 70 % solids with the following characteristics: Nonvolatile 70 ± 1 %, phthalic anhydride 23 % min, fatty acids 60 to 65 %, dihydric alcohol 4 % max, acid number 5 to 10, specific gravity 0.950–0.970, Gardner color 10.

7. Procedure for Mass Color

7.1 Weigh to the nearest 10 mg an appropriate amount of pigment to be tested into a 200-mL tall-form beaker. (The amount of pigment needed can be calculated from the graph shown in Fig. 1 or obtained from the table on the graph.) To the pigment in the beaker carefully weigh in 55.00 ± 0.01 g of the long oil alkyd grinding vehicle (Note 1). Then add 60 mL of 20 to 30-mesh sand from a graduated cylinder and 1.00 ± 0.01 g of the drier blend. Stir the sand, vehicle, and pigment with a spatula until the pigment is completely wetted.

NOTE 1—For routine control, grinding vehicle, drier blend, and white tint paint can be placed in the type of dispensers used in stores to tint custom colors. The following amounts are recommended: 2 fluid oz each of vehicle and of white paint, 1/3 oz-drier. The dispenser shall be reproducible to 2 parts per thousand when dispensing 1-oz charges.

7.2 Carefully clamp the beaker on the miniature sandmill and adjust the height of the disk so that it is between 3/4 and 1 in. (20 to 25 mm) above the bottom of the beaker. Turn on the mill and disperse for exactly 10 min, unless otherwise agreed upon, at a shaft speed of 8000 r/min, accurately timing the length of dispersion with a stopwatch or other suitable timing device.

NOTE 2—For normal control procedure, a 10-min dispersion interval will be sufficient. For ultimate color development use the procedure in Annex A1.

7.3 Shut off the mill and raise the impeller until it is just clear of the liquid in the beaker and turn the mill on again for a few seconds to remove excess mill base from the impeller.

7.4 Remove the material from the mill and filter into a suitable container through a cone strainer or equivalent 40-mesh (420- μ m) disposable screen.

7.5 Repeat the foregoing procedure with the standard reference pigment.

7.6 Draw the test and standard dispersions down simultaneously on a paper chart over a vacuum drawdown plate or other suitable plane surface using the applicator bar specified in 4.8. Compare the color visually while still wet and then set aside in a dust-free area to dry. If desired, evaluate the color difference instrumentally by Method D 2244. Report color difference in units agreed upon between purchaser and seller.

8. Procedure for Tint Color

8.1 From the receiving container used in 7.4, weigh 5.000 $\pm$ 0.010 g of the strained mill base into a 200-mL beaker and add 100.00 $\pm$ 0.05 g of white tinting paint.

8.2 Place the beaker on the mill (without sand) and mix at 8000 r/min for 5 min.

NOTE 3—Some mill bases require vigorous agitation when being dispersed into the paint. Normal agitation on a paint shaker is not satisfactory.

8.3 Repeat the above procedure with the standard reference material and then proceed as directed in 7.6.

8.4 To determine the relative tinting strength of the test material as compared to the standard at the wavelength of maximum absorbance, or at a wavelength agreed upon between purchaser and seller, employ the following procedure.

8.4.1 If the spectrophotometric curves cross or otherwise deviate from a relationship characteristic of effectively identical materials, the method is inapplicable (that is, the method is not applicable to metamer specimens).

8.4.2 When spectrophotometers or abridged spectrophotometers are used as color-measuring instruments, determine the lowest reflectance reading between 420 and 680 nm, or at the agreed band of wavelengths, and use it in the calculation. Read and record the reflectance to at least the nearest 0.001 reflectance unit.

8.4.3 With color-measuring instruments that employ broad wavelength filters, use the lowest reflectance obtained, or the reflectance from the agreed filter, in the calculations.

Read and record the reflectance to at least the nearest 0.001 reflectance unit.

NOTE 4—The calculation is based on theory that, strictly interpreted, requires for the types of material being tested, use of a spectrophotometer or abridged spectrophotometer. However, many users of this test method obtain satisfactory results using color-measuring instruments equipped with broad wavelength filters.

8.4.4 Calculate the tinting strength of color pigments as follows:

$$TS = \frac{T[(1 - R_{\infty})^2/2 R_{\infty}]_u}{[(1 - R_{\infty})^2/2 R_{\infty}]_s}$$

where:

TS = tinting strength,

R = measured reflectance and subscript "u" means reflectance of material under test (that is, unknown) and "s" means reflectance of standard reference material, and

T = assigned TS of the standard reference material, frequently assigned at "100" so that TS is in percent of standard.

9. Precision

9.1 On the basis of an interlaboratory study of this test method in which two operators in five laboratories tested five pigments on each of two days, the precision was found to be as shown.

9.2 *Mass Color*—The within-laboratory standard deviation was found to be 0.97 MacAdam color-difference units and the between-laboratory standard deviation 1.14 units. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.2.1 *Repeatability*—Two results obtained in the same laboratory should be considered suspect if they differ by more than 2.7 MacAdam units.

9.2.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 3.2 MacAdam units.

9.3 *Tint Strength*—The within-laboratory standard deviation was found to be 2.74 % and the between-laboratory standard deviation 3.46 %. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

9.3.1 *Repeatability*—Two results obtained in the same laboratory should be considered suspect if they differ by more than 7.7 %.

9.3.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 9.7 %.

ANNEX

(Mandatory Information)

A1. DETERMINATION OF MAXIMUM COLOR

A1.1 In some cases it may be desirable to determine the maximum color obtainable by this procedure. In this case Section 7 may be modified as follows:

A1.1.1 Carefully determine the total weight of the dispersion mixture and container. Place on the mill and disperse for 10 min. After spinning the impeller free of excess mate-

al, reweigh the container and bring back to the original weight with solvent. Carefully mix the added solvent back into the dispersion and extract 2 g of sandfree base, reduce with 50 g of white paint, and prepare a drawdown. Repeat the above process at 10-min intervals until no further increase in tint strength occurs. Note the time at which maxi-

mum tinting strength occurs and record for future reference.

A1.1.2 Subsequent samples of this pigment can then be run for the optimum length of dispersion time. However, solvent lost during dispersing must be replaced periodically so that viscosity variations will not affect the rate of dispersion.

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Standard Test Method for Solubility Range of Resins and Polymers¹

This standard is issued under the fixed designation D 3132; the number immediately following the designation indicates the year of original adoption 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 10 was added editorially in May 1990.

1. Scope

1.1 This test method covers determination of the solubility of resins and polymers in terms of the region of solubility parameter and hydrogen bonding of solvents in which complete solution occurs. In some cases dipole moment of the solvents may also be required to delineate more exactly the boundaries of solubility.

1.2 This test method is applicable only if the test solutions are of sufficient clarity and freedom from color to allow accurate visual judgement of complete solubility and of low enough viscosity for solution to take place.

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 a specific hazard statement see Note in 6.2.*

2. Terminology

2.1 Definitions:

2.1.1 The solubility parameter δ of a substance is defined as the square root of the "cohesive energy density," or energy of vaporization per unit volume:

$$\delta = (\Delta E/V)^{1/2}$$

where:

ΔE = energy of vaporization, and

V = molar volume.

The value of δ for a volatile liquid can be calculated accurately from the latent heat of vaporization, or approximately from its boiling point. Solubility parameter values for large number of solvents are available in Table 1.

2.1.2 Solvents are also classified according to their hydrogen bonding power, γ . Numerical values for γ may be derived from spectroscopic analysis. In one method,² γ is defined as one-tenth the wavenumber shift observed by Gordy's technique,³ and values range from 0 to about 25.

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.

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² Crowley, J. D., et al, "A Three Dimensional Approach to Solubility," *Journal of Paint Technology*, JPIRA, Vol 38, No. 496, 1966, p. 269; Vol 39, No. 504, 1967, p. 19.

³ Gordy, W., "Spectroscopic Evidence of Hydrogen Bonds," *Journal of Chemical Physics*, JCPA, February 1939, February 1940, March, 1941.

Another method,⁴ which limits values of γ to the range of 2.2 to 10, defines γ by the following equation:

$$\gamma = (0.0359 \times \Delta\nu) + 2.2$$

where ν is the wavenumber shift as determined by Gordy's method. Hydrocarbons, halogenated hydrocarbons and nitro-hydrocarbons have low values of γ ; esters, ethers, ether-alcohols, and ketones are intermediate; and alcohols, amines, and acids have high values.

2.1.3 The solubility parameter δ_m , of a mixture of solvents having parameters, δ_1 , δ_2 , etc., is a function of the molar fraction and molar volume of the components:

$$(\delta_1 x_1 V_1 + \delta_2 x_2 V_2) / (x_1 V_1 + x_2 V_2)$$

in which x_1 and V_1 , x_2 and V_2 , etc., are the corresponding molar fractions and volumes, respectively. If the components have the same molar volumes ($V_1 = V_2$),

$$\delta_m = x_1 \delta_1 + x_2 \delta_2$$

Thus, in a mixture of two components A and B having the same molar volumes and solubility parameter values of δ_A and δ_B

$$\delta_m = \frac{(\text{volume percent A} \times \delta_A) + (\text{volume percent B} \times \delta_B)}{100}$$

2.1.4 Similarly, the hydrogen bonding value, γ_m , of a mixture is determined by:

$$\gamma_m = \frac{(\text{volume percent A} \times \gamma_A) + (\text{volume percent B} \times \gamma_B)}{100}$$

and dipole moment by:

$$\mu_m = \frac{(\text{volume percent A} \times \mu_A) + (\text{volume percent B} \times \mu_B)}{100}$$

3. Summary of Test Method

3.1 Solubility of resinous and polymeric materials is dependent upon the solubility parameter, hydrogen bonding, and dipole moment of the solvents. Solubility parameter is the most important property of the three, followed by hydrogen bonding. Consequently, the solubility of most materials is sufficiently defined by the area of solubility parameter and hydrogen bonding of true solvents. A material is insoluble or incompletely soluble in a solvent if its solubility parameter and hydrogen bonding properties fall

⁴ E. I. du Pont de Nemours & Co., Bulletin PA 12-770, "Solvent Formulating Maps for Elvacite Acrylic Resins, Serial A-70562, July 1970.

outside this region.

3.2 In this test method, the material is tested separately in solvents that cover the entire solubility diagram so that boundaries of complete solubility can be determined.

4. Significance and Use

4.1 This test method is useful for an empirical determination of the solvent(s) in which a resin or polymer may be dissolved. This test method is also applicable to estimate the

TABLE 1 Solvent or Solvent Mixtures

No.	Solvent or Solvent Mixture (Mixture Given in Volume Percent)	Solubility Parameter, δ	Hydrogen Bonding		Dipole Moment, $^{\circ}\mu$
			γ^A	γ^B	
1*	Diisopropyl ether	6.9	11.7	6.6	1.3
2	<i>n</i> -Pentane	7.0	0	2.2	0
3*	<i>n</i> -Heptane	7.3	0	2.2	0
4	50 % Diisobutyl ketone	7.4	10.1	5.9	2.0
	50 % Diisopropyl ether				
5*	Diethyl ether	7.4	13.0	6.9	1.2
6*	50 % <i>n</i> -Heptane	7.6	4.2	3.7	1.4
	50 % Diisobutyl ketone				
7	25 % <i>n</i> -Heptane	7.7	6.3	4.5	2.0
	75 % Diisobutyl ketone				
8	Methyl cyclohexane	7.8	0	2.2	0
9	66.7 % <i>n</i> -Heptane	7.8	2.9	3.3	0.6
	33.3 % <i>n</i> -Butyl Acetate				
10*	Diisobutyl ketone	7.8	8.4	5.2	2.7
11*	57 % Diethyl ether	7.9	11.2	6.3	1.5
	43 % <i>n</i> -Butyl acetate				
12	66.7 % Diethyl ether	8.1	14.9	7.6	1.4
	33.3 % 2-Ethylhexanol				
13*	Cyclohexane	8.2	0	2.2	0
14	40 % <i>n</i> -Heptane	8.2	5.3	4.1	1.1
	60 % <i>n</i> -Butyl acetate				
15	71 % Cyclohexane	8.4	1.3	2.7	0.1
	29 % Toluene				
16*	<i>n</i> -Butyl acetate	8.5	8.8	5.4	1.9
17*	40 % Cyclohexane	8.6	2.7	3.2	0.2
	60 % Toluene				
18	50 % Cyclohexane	8.7	0	2.2	0
	50 % Benzene				
19	50 % <i>n</i> -Butyl acetate	8.7	6.7	4.6	1.2
	50 % Toluene				
20	50 % EGMBE (See No. 23)	8.7	10.9	6.2	1.8
	50 % <i>n</i> -Butyl acetate				
21*	33.3 % Diethyl ether	8.8	16.8	8.2	1.5
	66.7 % 2-Ethylhexanol				
22*	Toluene	8.9	4.5	3.8	0.4
23	2-Butoxyethanol	8.9	13.0	6.9	1.6
24*	Benzene	9.2	0	2.2	0
25	Propylene oxide	9.2	10.0	5.8	1.8
26	56 % EGMBE	9.2	15.5	7.8	1.6
	44 % 2-Ethylhexanol				
27	50 % Methylene chloride	9.3	3.0	3.3	1.0
	50 % Toluene				
28*	50 % Toluene	9.4	7.1	4.8	0.4
	50 % Dioxane				
29*	2-Ethylhexanol	9.5	18.7	8.9	1.7
30	Methyl acetate	9.6	8.4	5.2	1.7
31*	Methylene chloride	9.7	1.5	2.7	1.5
32	66.7 % Methylene chloride	9.8	4.2	3.7	1.1
	33.3 % Dioxane				
33	50 % Dioxane	9.8	5.6	4.2	1.0
	50 % Methylene chloride				
34*	Dioxane	9.9	9.7	5.7	0.4
35	Cyclohexanone	9.9	11.7	6.4	2.7
36*	2-Ethoxyethanol	9.9	13.0	6.9	1.6
37	75 % EGMBE	9.9	14.4	7.4	1.6
	25 % Methyl isobutyl carbinol				
38*	Carbon disulfide	10.0	0	2.2	0
39	30 % EGMBE	10.0	17.0	8.2	1.7
	70 % Methyl isobutyl carbinol				
40	Methyl isobutyl carbinol	10.0	18.7	8.8	1.7
41	66.7 % Methylene chloride	10.2	1.8	2.8	2.2
	33.3 % Nitroethane				
42	66.7 % Dioxane	10.3	7.3	4.8	1.5
	33.3 % Nitroethane				
43*	50 % Toluene	10.4	5.4	4.2	2.2
	50 % Acetonitrile				
44*	50 % EGMBE	10.4	15.9	7.9	1.7

TABLE 1 Continued

No.	Solvent or Solvent Mixture (Mixture Given in Volume Percent)	Solubility Parameter, δ	Hydrogen Bonding		Dipole Moment, $^{\circ}\mu$
			γ^A	γ^B	
45*	50 % <i>n</i> -Amyl alcohol	10.5	18.7	8.9	1.7
46	2-Ethylbutanol	10.6	2.2	3.0	2.9
47	33.3 % Methylene chloride				
	66.7 % Nitroethane	10.8	4.3	3.8	2.8
48	75 % Nitroethane				
	25 % Dioxane	10.8	8.5	5.3	1.6
49	75 % Dioxane				
	25 % Propylene carbonate	10.8	12.3	6.6	3.8
50	Dimethylacetamide	10.8	14.1	7.3	1.6
51	80 % EGME				
	20 % Methanol	10.9	16.8	8.2	1.7
52	33.3 % EGME				
	66.7 % <i>n</i> -Butanol	10.9	18.7	8.9	1.7
53	<i>n</i> -Amyl alcohol (pentanol)	11.0	1.3	2.7	1.8
54	48 % Benzene				
	52 % Nitromethane	11.0	10.7	5.6	2.1
55*	50 % Dioxane				
	50 % Dimethylformamide	11.1	2.5	3.1	3.6
56	Nitroethane	11.1	8.9	4.5	3.4
57*	25 % <i>n</i> -Butyl acetate				
	75 % Acetonitrile	11.4	18.7	8.9	1.7
58*	<i>n</i> -Butanol	11.5	4.4	3.8	3.8
59	50 % Nitroethane				
	50 % Acetonitrile	11.5	15.0	7.6	1.6
60*	65 % EGME				
	35 % Methanol	11.6	16.4	8.2	2.4
61*	66.7 % <i>n</i> -Butanol				
	33.3 % Dimethylformamide	11.7	8.7	5.4	2.2
62	50 % Dioxane				
	50 % Dimethyl sulfoxide	11.9	2.5	3.1	3.5
63*	50 % Nitroethane				
	50 % Nitromethane	11.9	6.3	4.5	3.9
64	Acetonitrile	11.9	13.8	7.2	3.2
65	30 % <i>n</i> -Butanol				
	70 % Dimethylformamide	11.9	18.7	8.9	1.7
66	<i>n</i> -Propanol	12.0	8.0	5.1	3.9
67	70 % Acetonitrile				
	30 % Dimethylformamide	12.0	10.0	5.8	3.8
68*	68 % Dimethylformamide				
	32 % Acetonitrile	12.1	11.7	6.4	3.8
69	Dimethylformamide	12.3	18.7	8.9	1.7
70	50 % <i>n</i> -Propanol				
	50 % Ethanol	12.4	4.0	3.7	3.6
71*	60 % Nitromethane				
	40 % Acetonitrile	12.4	15.2	7.7	2.8
72	50 % Ethanol				
	50 % Dimethylformamide	12.5	6.0	4.4	3.9
73	23 % Dioxane				
	77 % Propylene carbonate	12.6	9.3	5.6	3.9
74	40 % Dimethylformamide				
	60 % Dimethyl sulfoxide	12.6	16.6	8.2	2.3
75	70 % Ethanol				
	30 % Dimethylformamide	12.6	13.1	6.9	3.4
76*	80 % Dimethylformamide				
	20 % Methanol	12.7	2.5	3.1	3.4
77*	Nitromethane	12.8	18.7	8.9	1.7
78*	Ethanol	12.9	10.8	6.1	3.4
79*	72 % Dimethyl sulfoxide				
	28 % Ethanol	13.0	7.7	5.0	4.0
80	Dimethyl sulfoxide	13.2	14.9	7.6	2.8
81*	46 % Methanol				
	54 % Dimethylformamide	13.3	4.9	4.0	5.0
82	Propylene carbonate	13.3	9.9	5.8	3.5
83	80 % Dimethyl sulfoxide				
	20 % Methanol	13.3	11.8	7.0	3.8
	70 % Dimethylformamide				
	30 % Monomethylformamide				

TABLE 1 Continued

No.	Solvent or Solvent Mixture (Mixture Given in Volume Percent)	Solubility Parameter, δ	Hydrogen Bonding		Dipole Moment, $^{\circ}\mu$
			γ^A	γ^B	
84	50 % Ethanol	13.6	18.7	8.9	1.7
85*	50 % Methanol	13.7	16.4	8.1	2.4
	66.7 % Methanol				
86*	33.3 % Dimethylformamide	13.9	14.0	7.8	2.7
	57 % Methanol				
87*	43 % Dimethyl sulfoxide	14.5	18.7	8.9	1.7
	Methanol				
88*	Propylene glycol	15.0	20.6	9.4	2.2
89	63 % Methanol	15.1	16.2	8.8	2.5
90	37 % Monomethylformamide	17.1	20.6	9.6	2.3
	Ethylene glycol				

^A Crowley, J. D., et al, "A Three Dimensional Approach to Solubility," *Journal of Paint Technology*, Vol 38, No. 496, 1966, p 269; Vol 39, No. 504, 1967, p. 19.

^B E. I. du Pont de Nemours & Co., Bulletin PA 12-770, "Solvent Formulating Maps for Elvacite Acrylic Resins," Serial A-70562, July 1970.

^C McLellan, A. L., *Tables of Experimental Dipole Moments*, W. H. Freeman & Co., San Francisco, 1963.

solvents that may be useful for further dilution of a polymer or resin solution without formation of haze or without polymer or resin precipitation.

5. Apparatus

5.1 *Glass Vials*, with screw caps, capacity 5 to 20 mL.

5.2 *Mixing Rolls, Tumblers, or Other Rotary Mixing Machine*.

6. Reagents and Materials

6.1 Solvents and solvent mixtures used in this test method are listed in Table 1, in order of increasing solubility parameter. Those with an asterisk can be used in a preliminary survey to establish the general areas of solubility and nonsolubility. Intermediate solvents are then used to define more closely the solubility limits of a resin.

6.2 *Quality of Solvents*—Each solvent should be a good technical or commercial grade containing not less than 95 %, but preferably 99 %, of the specified compound and should be essentially anhydrous (<0.3 percent water).

NOTE: Warning—Diethyl ether, diisopropyl ether, and dioxane may form explosive peroxides on long storage, particularly if kept in glass bottles exposed to light.

6.3 Solvent mixtures, which are in volume percent, should be made by adding solvent from burets that have been washed with diethyl ether, dried at 65°C and rinsed twice with solvent before filling. Bottles containing mixtures should be tightly capped to prevent evaporation. Condensate above the liquid level should be well mixed in before using.

7. Procedure

7.1 Preparation of Solutions:

7.1.1 The ratio of solute to solvent should correspond as much as possible to the intended use of the material but should be chosen to avoid difficulty in effecting solution because the viscosity must be low enough for mixing to take place. For most film-formers the concentration range is from 40 % for low molecular weight resins to 10 % for polymers that give viscous solutions.

7.1.2 The precision of weighing the solute and solvent should ensure a maximum deviation of ± 5 % in the desired concentration in each series of tests. Report the test concentration with the results since the solubility parameter range is somewhat dependent upon concentration.

7.1.3 Dry the clean vials at 65°C and label or mark them.

Select vials that are sufficiently large to promote flow of viscous solutions. A 15-mL vial containing 5 g of solution has been found satisfactory.

7.1.4 Reduce large lumps of aggregates in the resin or polymer to a convenient size by means that do not introduce contamination but not to a fine powder that may lead to packing or oxidation. With resins that give solutions of low viscosity, the solvent may be added to the solute or vice versa. The former is usually more convenient as the material can first be weighed into all the vials followed by the selected solvents. With high molecular weight resins that tend to gel, the order of addition markedly affects the time required to dissolve the resin and eliminate gel particles. Consequently, the solvent should be weighed into the vial and then the specimen in small portions.

7.1.5 In either procedure, after adding the correct amounts of solute and solvent, cap the vial tightly and mix the contents by shaking or swirling. Tumble or rotate the vials end-over-end for 24 h. One method is to place the vials in a quart or gallon can with their long axes perpendicular to the long axis of the can and rotate the can at a slow speed on mixing rolls. The rate of rotation should not be so fast as to prevent back and forth flow in the vials. One to five revolutions per minute are suitable speeds.

7.2 Interpretation of Results:

7.2.1 At the end of 24 h line up the vials for observation. Allow to stand for a few minutes and then classify the appearance of the contents according to the following ratings:

7.2.1.1 *Complete Solution*—A single, clear liquid phase with no distinct solid or gel particles.

7.2.1.2 *Borderline Solution*—Cloudy or turbid but without distinct phase separation.

7.2.1.3 *Insoluble*—Two phases: either a liquid with separate gel or solid phase or two separate liquids.

7.2.2 Maintain borderline samples at 20. to 27°C for 7 days and observe again to determine if the classification has changed.

7.2.3 Plot the solubility results on a graph using solubility parameter as abscissa, hydrogen bonding as ordinate, and symbols to distinguish the three solubility classes.

7.2.4 Identify areas of complete solubility and insolubility and select additional solvents from Table 1 to define more closely the solubility limits of the resin. Repeat the test with these solvents and also with any that produced anomalous

results, for example, borderline or insoluble between two complete solutions.

7.2.5 Plot the additional test results and draw in the limits of solubility. If anomalies are still present it may be necessary to plot solubility parameter versus dipole moment at eight levels of hydrogen bonding and draw in solubility limits (contour lines) for each level.

7.2.6 The solubility classification in 7.2.1 uses the simplified approach that there are no differences within the soluble and insoluble regions. Actually, it is possible to distinguish degrees of solubility and insolubility. The latter ranges from settling of an apparent or borderline solution, through various levels of gelling and wetting, to complete insolubility when the resin is absolutely unaffected by the solvent. Similarly in the soluble region, not all solutions are identical. With resins that are high in molecular weight or have a wide range in molecular weight distribution, there may be only a few solvents that produce perfectly clear solutions. The other solutions may vary from being slightly cloudy to fairly turbid. Viscosity measurements on the solution, allowing for solvent viscosity, might be used to determine the area of best solubility. Contour lines of degrees of solubility could be drawn if sufficient solvents were tested.

7.2.7 Because there is this gradation from complete solu-

bility to total insolubility, where the borderline is placed may be a matter of personal choice or the end-use of the polymer. High clarity may be required for an unpigmented solution while some turbidity might be acceptable if the material will be pigmented in use.

8. Report

8.1 Results of this test are preferably presented in the form of a graph showing the region of solubility for the material under test. For written reports, state the minimum and maximum solubility parameter, δ , and hydrogen bonding, γ , at which solution took place. If desired, the solubility parameter limits at several levels of hydrogen bonding can be given.

8.2 Report the concentration of resin used in the tests.

9. Precision

9.1 No statement of precision is at present available for this test method.

10. Keywords

10.1 dilution ratio; resin solubility; solubility parameters of resins

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Standard Test Methods for Chemical Analysis of Phthalocyanine Blue and Green Pigments¹

This standard is issued under the fixed designation D 3256; the number immediately following the designation indicates the year of original adoption 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 January 1991.

1. Scope

1.1 These test methods cover procedures for the qualitative chemical analysis of pigments known commercially as copper phthalocyanine blue and green.

1.2 The procedures appear in the following order:

	Section
Identification	5
Moisture and Other Volatile Matter	6
Detection of Basic Dye Derivatives	7
Detection of Other Organic Coloring Matter	8
Detection of Ultramarine Blue	9
Detection of Iron Blue or Chrome Green	10

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 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 1193 Specification for Reagent Water³

3. Significance and Use

3.1 These test methods are suitable for determining if impurities are present and establishing that the required pigments are present. These test methods may be used for manufacturing and purchasing quality control.

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.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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent grade water conforming to Specification D 1193.

5. Identification

5.1 To about 0.05 g of the sample in a 50-mL beaker, add 30 mL of sulfuric acid (H_2SO_4 , sp gr 1.84). Stir occasionally for 15 min, heating if necessary to accelerate the solution of the pigment. Formation of a dark greenish yellow solution indicates phthalocyanine blue and a dark reddish color indicates brominated green pigment. Pour the solution into 250 mL of water and stir. The phthalocyanine pigment should immediately precipitate as a flocculent mass.

5.2 Filter off the precipitate, washing once or twice with water. Scrape a small amount of the precipitate off the filter, place on a clean platinum wire moistened with hydrochloric acid (HCl), and subject it to the low flame of a bunsen burner. As the precipitate burns, a light blue-green flame should be clearly evident. This indicates organically combined copper.

NOTE 1—Characteristic spectrophotometric-absorption spectra in the near infrared range (700 to 900 nm) are exhibited by dilute solutions of copper phthalocyanine pigments (2 to 50 mg/L) in H_2SO_4 (sp gr 1.84). The absorption maxima, being sharp and well-defined, may be used for positive qualitative identification of phthalocyanine pigments. Phthalocyanine green has maxima at approximately 820 nm and 860 nm for bromine-free types; for the chlorinated-brominated types, the maxima appear at approximately 830 to 840 nm and 875 to 885 nm.

6. Moisture and Other Volatile Matter

6.1 Determine moisture in accordance with Method A of Test Methods D 280.

7. Detection of Basic Dye Derivatives

7.1 Add to 1 g of the sample, 50 mL of a mixture of equal parts of ammonium hydroxide NH_4OH (sp gr 0.90) and denatured ethyl alcohol (95 %). Warm gently and filter. Neutralize the filtrate with tartaric acid solution (200 g/L) until slightly acid to litmus. If the solution is colorless, discounting a slight yellow tinge, no basic dye is present.

7.2 If the solution is colored beyond a slight yellow tinge, add about 5 mL of 0.1 N titanium trichloride ($TiCl_3$) solution (Note 2). If a basic dye is present, the color will lighten significantly. If no basic dye is present, no significant color change will occur.

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 D 01.21 on Chemical Analysis of Paints and Paint Materials.

Current edition approved March 27, 1986. Published May 1986. Originally published as D 3256 - 73. Last previous edition D 3256 - 73.

² Annual Book of ASTM Standards, Vol 06.02.

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."

NOTE 2—Titanium trichloride is marketed as a 16% solution in HCl. Mix 7 mL of this solution with 90 mL of HCl (1 + 12) to obtain a reagent approximately 0.1 N. Protect from oxidation.

8. Detection of Other Organic Coloring Matter

8.1 *Procedure*—Weigh about 0.05 g of the sample into each of two 50-mL beakers. Add 25 mL (at room temperature) of denatured ethyl alcohol (95 %) to one beaker and about 25 mL of acetone to the other. Stir each thoroughly for a few minutes and let stand for about 2 h. Filter through two thicknesses of medium-texture, qualitative filter paper. The presence of more than an extremely slight pink, yellow, or blue tint in either filtrate indicates other organic coloring matter is in the pigment. Organic colors resistant to the above reagents are not likely to be in phthalocyanine pigments but, if desired, procedures given in standard references can be followed to establish if they are present.

9. Detection of Ultramarine Blue

9.1 *Procedure*—Warm gently about 1 g of the sample with about 5 mL of HCl (1+1). Decomposition of the ultramarine blue takes place with evolution of H₂S. This may be detected by either its odor or a brown coloration appearing on a strip

of moistened lead acetate paper.

10. Detection of Iron Blue and Chrome Green

10.1 *Procedure*:

10.1.1 To 1 g of the sample in a 100-mL beaker, add 25 mL of NaOH solution (50 g/L). Boil for about 1 min. Dilute to approximately 40 mL and filter.

10.1.2 Add HCl (1+1) to the filtrate until faintly acid to litmus. Add 2 mL of a ferric iron solution [ferric sulfate or ferric alum (20 g/L)]. The development of a blue color reveals the presence of ferrocyanide, and hence iron blue or chrome green in the original pigment. For amounts around 0.05 % iron blue, the color may take 2 to 3 h to develop.

11. Precision and Bias

11.1 No precision or bias information are available for these tests.

12. Keywords

12.1 chrome green, detection of; iron blue, detection of; phthalocyanine blue, analysis of; phthalocyanine green, analysis of; pigment; ultramarine blue, detection of

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Standard Test Methods for Analysis of White Zinc Pigments¹

This standard is issued under the fixed designation D 3280; the number immediately following the designation indicates the year of original adoption 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—Section 24 was added editorially in May 1990.

1. Scope

1.1 These test methods cover procedures for the analysis of white zinc pigments.

1.2 The analytical procedures appear in the following order:

	Section
Preparation of Sample	6
Zinc Oxide	
Total Zinc, Using Diphenylamine as Internal Indicator	7
Total Zinc, Using Uranyl Acetate as External Indicator	8
Total Impurities	9
Total Sulfur	10
Moisture and Other Volatile Matter	11
Leaded Zinc Oxide	
Total Lead	12
Total Zinc	13
Total Sulfur	14
Total Impurities	15
Moisture and Other Volatile Matter	16
Water-Soluble Salts	17
Zinc Sulfide	
Zinc Oxide	18
Zinc Sulfide	19
Water-Soluble Salts	20
Moisture and Other Volatile Matter	21
Barium Sulfate	22
Titanium Dioxide	23

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 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²

D 1193 Specification for Reagent Water³

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.

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² Annual Book of ASTM Standards, Vol 06.02.

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

D 1394 Test Methods for Chemical Analysis of White Titanium Pigments²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes⁴

3. Summary of Test Methods

3.1 Zinc Oxide:

3.1.1 *Total Zinc*—Determined using diphenylamine as an internal indicator and also using uranyl acetate as an external indicator. Total impurities are calculated.

3.1.2 *Total Sulfur*—Determined as BaSO₄ and calculated to sulfur.

3.1.3 *Moisture and Volatile Matter*—Determined in accordance with Method A of Test Methods D 280.

3.2 Leaded Zinc Oxide:

3.2.1 *Total Lead*—Determined as PbSO₄ and calculated to percent PbO.

3.2.2 *Total Zinc*—Determined on the filtrate from procedure in 13.1.1 in accordance with methods in Sections 8 or 9.

3.2.3 *Total Sulfur*—Determined as BaSO₄ and calculated to percent SO₃.

3.2.4 *Total Impurities*—Calculated from compositional data.

3.2.5 *Moisture and Other Volatile Matter*—Determined in accordance with Method A of Test Methods D 280.

3.2.6 *Water Soluble Salts*—Determined gravimetrically.

3.3 Zinc Sulfide:

3.3.1 *Total Zinc*—Determined using uranyl acetate external indicator in accordance with Section 9.

3.3.2 *Zinc Sulfide*—Determined in accordance with Sections 8 or 9 and calculating ZnO to ZnS.

3.3.3 *Water Soluble Salts*—Determined in accordance with Section 18.

3.3.4 *Moisture*—Determined in accordance with Method A of Test Methods D 280.

3.3.5 *Barium Sulfate*—The sample is treated with N₂SO₄ and Na₂CO₃ and the residue of BaCO₃ is dissolved in HCl and (NH₄)₂SO₄ added to precipitate BaSO₄, which is weighed.

3.3.6 *Titanium Dioxide*—Determined in accordance with Test Methods D 1394.

4. Significance and Use

4.1 White zinc pigments find considerable use in white

⁴ Annual Book of ASTM Standards, Vol 14.02.

paints, and as such it is useful to formulators and users to be able to monitor the amounts of these pigments in whole paints. It is also of interest to raw material suppliers and paint producers to check the specifications of each pigment.

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

5.3 Concentration of Reagents:

5.3.1 Concentrated Acids and Ammonium Hydroxide—When acids and ammonium hydroxide are specified by name or chemical formula only it should be understood that concentrated reagents of the following specific gravities or concentrations are intended:

Acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$	99.5 %
Hydrochloric acid, HCl	sp gr 1.19
Hydrofluoric acid, HF	48 %
Nitric acid, HNO_3	sp gr 1.42
Sulfuric acid, H_2SO_4	sp gr 1.84
Ammonium hydroxide, NH_4OH	sp gr 0.90

The desired specific gravities or concentrations of all other concentrated acids are stated whenever they are specified.

5.3.2 Diluted Acids and Ammonium Hydroxide—Concentrations of diluted acids and ammonium hydroxide, except when standardized, are specified as a ratio stating the number of volumes of the concentrated reagents to be diluted with a given number of volumes of water, as in the following example: HCl (1+99) means 1 volume of concentrated HCl (sp gr 1.19) diluted with 99 volumes of water.

6. Preparation of Sample

6.1 Grind dry pigments, if lumpy or not finely ground, to a fine powder for analysis. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. Mix the sample in all cases thoroughly before taking specimens for analysis.

6.2 Separate pigments from paints or pastes, grind to a fine powder, pass through a 180- μm (No. 80) sieve (Note 1) to remove any skins, thoroughly mix, and oven dry at 105°C. Moisten such pigments after weighing with a little alcohol before adding reagents for analysis.

NOTE 1—Detailed requirements for this sieve are given in Specification E 11.

6.3 Preserve all samples in stoppered bottles or containers.

⁵ "Reagent Chemicals, American Chemical Society Specifications," American Chemical Society, 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."

ZINC OXIDE

7. Total Zinc, Using Diphenylamine as Internal Indicator

7.1 Reagents:

7.1.1 Diphenylamine Indicator Solution (10 g/L)—Dissolve 1 g of diphenylamine in 100 mL of H_2SO_4 .

7.1.2 Potassium Ferrocyanide (1 mL = 0.008 g Zn)—Dissolve 35 g of $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$ in water and dilute to 1 L and add 0.3 g of potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$). Standardize the solution by titrating against zinc (320 to 340 mg), following the procedure described in 6.2. Calculate the grams of zinc equivalent to 1.00 mL of the solution.

7.2 Procedure—Weigh to 0.1 mg about 0.4 g of the sample into a tall form 400-mL beaker. Moisten with about 20 mL of water, and dissolve in 15 mL of HCl . Neutralize with NH_4OH , using litmus as the indicator. Add an excess of 15 mL of H_2SO_4 (1+2) and dilute to 200 mL. Heat to approximately 60°C, add 2 drops of diphenylamine indicator solution and while stirring vigorously, titrate with $\text{K}_4\text{Fe}(\text{CN})_6$ solution to the color change from purple to a persistent yellowish green.

NOTE 2—The true end point is a sharp, persistent change from a purple to a yellowish green. At the beginning of the titration, a deep blue color is developed after addition of a few millilitres of $\text{K}_4\text{Fe}(\text{CN})_6$ solution. About 0.5 to 1.0 mL before the true end point is reached, the solution changes from a blue to a purple color. After the purple color is developed, the titration should be continued dropwise to the persistent yellowish green end point.

7.3 Calculation—Calculate the percent total zinc as ZnO , A , as follows:

$$A = \left[\frac{V_2 Z \times 1.245}{S_1} \right] \times 100 \quad (1)$$

where:

V_2 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the specimen, mL,

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL,

S_1 = specimen weight, and

1.245 = molecular weight ZnO (81.38)/molecular weight Zn (65.38).

8. Total Zinc, Using Uranyl Acetate as External Indicator

8.1 Reagents:

8.1.1 Uranyl Acetate Indicator Solution (50 g/L)—Dissolve 5 g of $\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ in 100 mL of water and make slightly acid with acetic acid.

8.1.2 Potassium Ferrocyanide, Standard Solution (1 mL = 0.008 g Zn)—Prepare and standardize as in 7.1.2. Run a blank titration with the same amounts of reagents and water. Calculate the zinc equivalent of the solution as follows:

$$Z = W(V - B) \quad (2)$$

where:

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL,

W = zinc used,

V = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the zinc, mL, and

B = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the blank, mL.

8.2 Procedure:

8.2.1 Weigh to 0.1 mg about 0.4 g of the sample into a tall-form 400-mL beaker. Moisten with about 20 mL of

water and dissolve by adding 10 mL of HCl. Add NH_4OH until slightly alkaline to litmus paper. Add HCl until just acid, and then add 3 mL in excess. Dilute to about 250 mL with hot water and heat nearly to boiling. Titrate with $\text{K}_4\text{Fe}(\text{CN})_6$ solution, stirring constantly, until a drop of phenyl acetate indicator tested in a white porcelain spot plate shows a brown tinge after standing 1 min.

8.2.2 *Blank*—Run a blank titration with the same amounts of reagents and water.

8.3 *Calculation*—Calculate the percent total zinc as ZnO , A , as follows:

$$A = \left[\frac{((V - B)Z \times 1.245)}{S} \right] \times 100 \quad (3)$$

where:

V = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the sample, mL,
 S = sample used, g, and
 1.245 = molecular weight ZnO (81.38)/molecular weight Zn (65.38).

9. Total Impurities

9.1 *Calculation*—Calculate the percent total impurities, A , as follows:

$$A = 100 - (L + Z_1 + S_5) \quad (4)$$

where:

L = total lead as PbO , %,
 Z_1 = total zinc as ZnO , %, and
 S_5 = total sulfur as SO_3 , %.

10. Total Sulfur

10.1 Reagents:

10.1.1 *Bromine Water (saturated)*.

10.1.2 *Aluminum*—Reagent grade granular aluminum.

10.1.3 *Barium Chloride Solution* (100 g $\text{BaCl}_2 \cdot \text{L}$)—Dissolve 117 g $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in water and dilute to 1 L.

10.2 Procedure:

10.2.1 Weigh to 0.1 g about 10 g of the sample into a 400-mL beaker. Add 50 mL of saturated bromine water, 100 mL of water, and 35 mL of HCl. Boil until all the bromine has been expelled, cool, and add 3 to 5 g of granular aluminum. Heat to boiling, filter, and wash well with hot water.

10.2.2 Dilute the filtrate to 300 mL with water, neutralize with NH_4OH , and add 6 drops of HCl. Heat to boiling and add 25 mL of hot BaCl_2 solution dropwise, with constant stirring. Allow to stand in a warm place for at least 2 h.

10.2.3 Filter, using a weighed Gooch crucible, or a fine textured filter paper and wash well with hot water. Dry and ignite in a muffle furnace for 30 min. Cool and weigh as BaSO_4 .

10.3 *Calculation*—Calculate the percent sulfur, A , as follows:

$$A = \left[\frac{(P \times 0.1374)}{S_2} \right] \times 100 \quad (5)$$

where:

P = BaSO_4 precipitate, g,
 S_2 = specimen weight, g, and
 0.1374 = molecular weight of sulfur (32.064)/molecular weight BaSO_4 (233.43).

11. Moisture and Other Volatile Matter

11.1 *Procedure*—Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

LEADED ZINC OXIDE

12. Total Lead

12.1 Procedure:

12.1.1 Weigh to the nearest 0.1 mg about 0.5 g of the sample into a 400-mL beaker. Dissolve in 250 mL of water and 20 mL of HNO_3 (Note 2). Add 5 mL of H_2SO_4 and evaporate to dense white fumes. Cool, add 50 mL of 95 % alcohol and 200 mL of water, and let stand cold 1 to 2 h. Filter, using a weighed Gooch crucible. Wash the precipitate with H_2SO_4 (1+99) and combine the filtrate and washings. If the zinc content of the sample is known to be 40 % or over, reserve the filtrate and washings for the determination of total zinc (Section 13).

NOTE 3—If the sample contains calcium or barium, the lead and zinc should be separated by precipitation with H_2S after solution in HCl, making alkaline with NH_4OH and then acid with acetic acid. Dissolve the PbS and ZnS in dilute HNO_3 and determine the lead and zinc as above.

12.1.2 Ignite the precipitate in the crucible at dull red heat ($550 \pm 50^\circ\text{C}$) for 20 min, cool, and weigh.

12.2 *Calculation*—Calculate the percent total lead as PbO , A , as follows:

$$A = \left[\frac{(P_1 \times 0.736)}{S_3} \right] \times 100 \quad (6)$$

where:

P_1 = PbSO_4 precipitate, g,
 S_3 = sample used, g, and
 0.736 = molecular weight PbO (223.21)/molecular weight PbSO_4 (303.28).

13. Total Zinc

13.1 *Reagents*—See 7.1 or 8.1, whichever is applicable.

13.2 Procedure:

13.2.1 If the zinc content of the sample is known to be 40 % or over, determine zinc on the filtrate obtained in 12.1.1. If the zinc content is known to be under 40 %, weigh to the nearest 0.1 mg about 1 g of the sample, precipitate and filter off the lead as PbSO_4 as described in 12.1.1, and determine zinc on the filtrate.

13.2.2 Evaporate the appropriate filtrate to dryness and determine zinc as directed in Sections 7 or 8.

14. Total Sulfur

14.1 *Reagents*—See 10.1.3.

14.2 Procedure:

14.2.1 Weigh to the nearest 0.1 mg about 1 g of the sample, into a 400-mL beaker. Add 10 mL of water, 3 g of NH_4Cl , and 5 mL of HCl saturated with bromine. Heat on a steam bath for about 15 min. Add 50 mL of water, neutralize with dry Na_2CO_3 in excess. Boil 10 to 15 min. Allow to settle, dilute to 250 mL with hot water, filter, and wash with hot water.

14.2.2 Redissolve the residue in HCl, reprecipitate as before, and wash with hot water. Acidify the combined filtrates with HCl, adding 5 mL in excess. Boil 10 to 15 min. Neu-

tralize with NH_4OH and add 6 drops excess HCl . Heat to boiling and add 25 mL of hot BaCl_2 solution dropwise, with constant stirring. Allow to stand in a warm place for at least 2 h.

14.2.3 Filter, using a weighed Gooch crucible or a fine textured filter paper, and wash well with hot water. Dry and ignite in a muffle furnace for 30 min. Cool and weigh.

14.3 *Calculation*—Calculate the percent sulfur as SO_3 , A , as follows:

$$A = \left[\frac{(P_2 \times 0.343)}{S_4} \right] \times 100 \quad (7)$$

where:

P_2 = BaSO_4 precipitate, g,

S_4 = specimen weight, g, and

0.343 = molecular weight SO_3 (80.07)/molecular weight BaSO_4 (233.43).

15. Total Impurities

15.1 *Calculation*—Calculate the percent of total impurities, A , as follows:

$$A = 100 - (L + Z_1 + S_3) \quad (8)$$

where:

L = total lead as PbO , %,

Z_1 = total zinc as ZnO , %, and

S_3 = total sulfur as SO_3 , %.

16. Moisture and Other Volatile Matter

16.1 *Procedure*—Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

17. Water-Soluble Salts

17.1 *Procedure*:

17.1.1 Weigh to the nearest 1 mg about 5 g of the sample into a 500-mL graduated flask. Add 250 mL of water at room temperature and shake for 10 min. Dilute the solution to the mark with water, mix by shaking, and filter through a dry, fine paper.

17.1.2 Transfer 400 mL of the clear filtrate to a weighed platinum dish and evaporate to dryness at $105 \pm 2^\circ\text{C}$, cool in a desiccator, and weigh rapidly.

17.1.3 *Blank*—Make a blank determination at the same time in a similar manner.

17.2 *Calculation*—Calculate the percent water-soluble salts, W , as follows:

$$W = \left[\frac{(R - B_2)}{S_5} \right] \times 100 \quad (9)$$

where:

R = dry salts obtained from the sample, g,

B_2 = water-soluble salts obtained from the blank, g, and

S_5 = sample represented in the aliquot used, g.

ZINC SULFIDE

18. Zinc Oxide

18.1 *Reagents*—See 8.1.

18.2 *Procedure*:

18.2.1 Weigh to 1 mg about 5 g of the sample into a 500-mL Erlenmeyer flask and add 200 mL of acetic acid (3+97). Shake continuously for 20 min and transfer to a 500-mL

volumetric flask. Dilute to the mark and filter through a dry, fine paper.

18.2.2 Transfer 400 mL of the clear filtrate to a 600-mL beaker. Add 20 mL of HCl and neutralize with NH_4OH , using litmus paper. Add HCl until just acid and then add 3 mL in excess. Heat nearly to boiling and titrate with $\text{K}_4\text{Fe}(\text{CN})_6$ solution as described in 8.2.

18.3 *Calculation*—Calculate the percent zinc oxide, A , as follows:

$$A = \left[\frac{((V_3 - B_3) Z \times 1.245)}{S_6} \right] \times 100 \quad (10)$$

where:

V_3 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the specimen, mL,

B_3 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the blank, mL,

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL,

S_6 = sample represented in the aliquot used, g, and

1.245 = molecular weight ZnO (81.38)/molecular weight Zn (65.38).

19. Zinc Sulfide

19.1 *Reagents*—See Section 7 or 8, whichever is applicable.

19.2 *Procedure*—Weigh to the nearest 0.1 mg a portion of the sample into a tall-form 400-mL beaker. Moisten with water, add 10 mL of H_2SO_4 (1+1), and evaporate to dryness. Cool, and proceed as directed in 7.2 or 8.2.

19.3 *Calculation*—Calculate the percent zinc sulfide, C , as follows:

$$C = \left[\frac{((V_4 - B_4) Z \times 1.491)}{S_7} \right] \times 100 - 1.20A \quad (11)$$

where:

V_4 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the specimen, mL,

B_4 = $\text{K}_4\text{Fe}(\text{CN})_6$ solution required for titration of the blank, mL,

Z = zinc equivalent of the $\text{K}_4\text{Fe}(\text{CN})_6$ solution, g/mL,

S_7 = sample used, g,

A = percent of zinc oxide (Section 17),

1.20 = factor for conversion of percentage of ZnO to the ZnS equivalent, and

1.491 = molecular weight ZnS (97.44)/molecular weight Zn (65.38).

20. Water-Soluble Salts

20.1 *Procedure*—Determine water-soluble salts as directed in Section 17.

21. Moisture

21.1 *Procedure*—Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

22. Barium Sulfate

22.1 *Reagents*:

22.1.1 *Ammonium Sulfate Solution* (30 g/L)—Dissolve 30 g of $(\text{NH}_4)_2\text{SO}_4$ in water and dilute to 1 L.

22.1.2 *Methyl Orange Indicator Solution*—Dissolve 0.1 g of methyl orange in 100 mL of water. Filter if necessary.

22.1.3 *Sodium Carbonate Solution* (2 g/L)—Dissolve 2 g of Na_2CO_3 in water and dilute to 1 L.

22.2 *Procedure:*

22.2.1 Weigh to the nearest 1 mg about 1 g of the sample into a 400-mL beaker. Moisten with water, and add 100 mL of H_2SO_4 (1+1), and evaporate to dryness. Cool, add 200 mL of water, and carefully break up the residue. Boil for 15 min. Filter, and wash with water.

22.2.2 Transfer the residue and paper to a platinum crucible and burn off the paper. Cool, add 6 g of Na_2CO_3 , and mix. Cover with 2 g of Na_2CO_3 , and fuse for 40 min over a Meker burner. Cool and leach the fusion with 200 mL of hot water in a 400-mL beaker. Filter and wash with hot Na_2CO_3 solution.

22.2.3 Dissolve the BaCO_3 with hot HCl (1+4), catching the filtrate in a 600-mL beaker. Wash the paper with water. Neutralize with NH_4OH , using methyl orange as the indicator. Add HCl until just acid and then add 6 drops in excess. Dilute to 400 mL, heat to boiling, and add 25 mL of hot $(\text{NH}_4)_2\text{SO}_4$ solution slowly, while stirring. Allow to stand in a warm place for at least 4 h.

22.2.4 Filter off the precipitate, using a weighed Gooch

crucible or a fine textured filter paper, and wash with hot water until free of chlorides. Dry, ignite in a muffle furnace for 30 min, cool, and weigh.

22.3 *Calculation*—Calculate the percent barium sulfate (BaSO_4), A , as follows:

$$A = \left(\frac{P_3}{S_8} \right) \times 100 \quad (12)$$

where:

P_3 = BaSO_4 precipitate, g, and

S_8 = sample used, g.

23. Titanium Dioxide

23.1 *Procedure*—Determine titanium dioxide in accordance with Sections 9 to 13 of Test Methods D 1394. If the pigment being analyzed is relatively low in TiO_2 , the use of a larger specimen than that specified in Test Methods D 1394 should give more accurate results.

24. Keywords

24.1 analysis; barium sulfate; lead; sulfur; uranyl acetate; white zinc pigment; zinc

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Standard Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments¹

This standard is issued under the fixed designation D 3360; the number immediately following the designation indicates the year of original adoption 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 particle-size distribution in the sub-sieve size range of the common extender pigments such as aluminum silicate (kaolin clay), magnesium silicate (talc), calcium carbonate (calcite or dolomite or precipitated calcium carbonate), and mica pigments, and may also be extended to the denser prime pigments such as the white titanium pigments (rutile or anatase) and similar mineral pigments when and if such information is of concern. Particle-size distribution has significance in the evaluation of rheological and pigmentary properties of pigments in paint and also may sometimes be used to characterize the identity or grade of pigments.

1.2 Sedimentation methods having as their basis Stoke's law have found general acceptance for this purpose. Results are expressed in terms of equivalent spherical diameter (e.s.d.), the diameter of a sphere having the same specific gravity as the particle in question and which settles at the same rate. Most mineral pigment particles are more or less asymmetrical, but despite differences in the relationship between equivalent spherical diameter and actual dimensions, the results of a sedimentation particle-size analysis can be correlated readily with many pigment properties.

1.3 Procedures limited to gravitational sedimentation^{2,3,4,5} are relatively inaccurate for pigment particles smaller than about 1 μm e.s.d., and centrifugal procedures may be required for the much finer ranges. Nevertheless, the data obtained above the 1 μm limitation provide useful information. This test method is particularly applicable to pigments if a major fraction of the particles fall in the range from about 15 to 1.5 μm , but have a total particle-size range of at least two decades.

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 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments⁶
- D 422 Method for Particle-Size Analysis of Soils⁷
- D 1193 Specification for Reagent Water⁸
- E 100 Specification for ASTM Hydrometers⁹
- E 300 Practice for Sampling Industrial Chemicals¹⁰

3. Summary of Test Method

3.1 For the determination of particle-size distribution by the application of Stokes' law to the sedimentation of particulate material out of an initially homogeneous suspension, any systematic set of measurements which permit the determination of the suspension density (that is, the "percent solids") at some defined distance beneath the surface of the suspension at some appropriately selected series of sedimentation time durations can be converted to a particle-size distribution. In this procedure, the suspension density is estimated at the effective distance beneath the suspension surface of the center of gravity of a floating hydrometer observed at a series of convenient time intervals selected to increase roughly exponentially.

NOTE 1—Any alternative system that provides an equivalent set of measurements (for example, the Andreasen Pipet Method,⁴ or any of the optical sampling methods based on change in turbidity, light scattering, and light or X-ray absorption, etc.) will also yield a particle-size distribution. Methods based on optical measurements, however, are much less generally applicable because of certain technological limitations too complex to be dealt with here.

4. Apparatus

4.1 *Stirring Apparatus*, commonly known as a "malted milk mixer," with a vertical high-speed shaft (approximately 10 000 r/min) tipped with a 25-mm diameter sine-wave impeller. The preferred mixing cup is a stainless steel cup about 180 mm deep and slightly tapered from an outside diameter at the top of about 100 mm to about 70 mm at the bottom. Some very fine particle pigments may require additional shear for complete dispersion. In some circum-

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 D01.31 on Pigment Specifications.

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² Norton, F. H., and Speil, S., *Journal of the American Ceramic Society*, Vol 21, 1938, p. 89.

³ Casagrande, A., "Hydrometer Method for Determination of Fineness Distribution of Soils," Julius Springer, Berlin, 1934.

⁴ Andreasen, A. H. M., "The Evaluation of Ground Materials," *Kolloid Beihfte*, Vol 27, 1928, p. 349.

⁵ Lane, Marvin K., "Improved Method for Measuring Particle Size Distributions by Gravity Sedimentation with a Hydrometer," *A Work Manual* by Lane, M. K., 4147 West Byron St., Chicago, IL 60641.

⁶ *Annual Book of ASTM Standards*, Vol 06.02.

⁷ *Annual Book of ASTM Standards*, Vol 04.08.

⁸ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁹ *Annual Book of ASTM Standards*, Vols 05.03 and 14.03.

¹⁰ *Annual Book of ASTM Standards*, Vols 06.03 and 15.05.

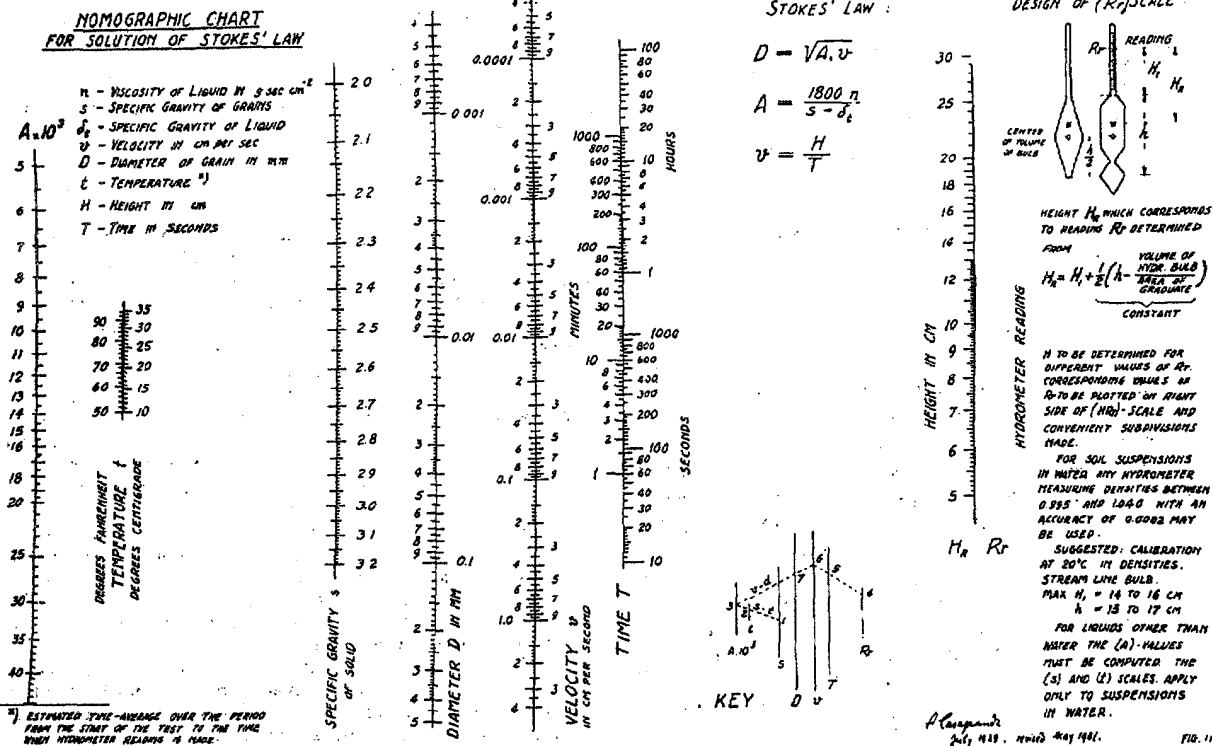


FIG. 1 Casagrande Nomographic Chart

stances, dispersion in a blender cup at maximum r/min may be considered instead of preparation with a "malted-milk mixer." (See 7.3.)

4.2 **Hydrometer**, certified with a minimum of 3 points of certification, graduated in units of specific gravity, and having a range from 0.995 to 1.038. The approximate dimensions are as follows: bulb length, 139 mm, bulb diameter 31 mm, and overall length 280 mm. Such a hydrometer is identified as a "Soil Hydrometer" in Specification E 100, and is also described in Method D 422.

4.3 **Sedimentation Cylinders**, glass, (two or more), having an inside diameter of about 65 mm and an overall height of 450 mm with a calibration mark to hold 1205 mL.⁵

4.4 Thermometer, accurate to $\pm 0.1^{\circ}\text{C}$ over a range from 15 to 35°C .

4.5 *Water Bath*, large enough to accommodate two or more of the sedimentation cylinders immersed to slightly above the 1205-mL graduation mark, and having circulating water and means for keeping its temperature throughout the bath to within $\pm 0.1^{\circ}\text{C}$ over a range from 18 to 30°C .

NOTE 2—In place of the water bath, the procedure may be carried out in a constant-temperature room controlled to the same precision as noted above.

4.6 *Time*—A stopwatch, or the equivalent, and an ordinary watch or clock.

4.7 *Balance*, sensitive to 0.01 g.

4.8 *Drying Oven*, with accurate thermostatic control to $\pm 2^{\circ}\text{C}$ at 110°C .

4.9 *Wash Bottle*, containing reagent water.

4.10 Casagrande Nomographic Chart (see Fig. 1).

5. Reagents

5.1 Purity of Water—Unless otherwise indicated references to water shall be understood to mean Type II reagent water conforming to Specification D 1193.

5.2 Dispersing Agents—The different pigments, depending on their specific surface properties, may require differing dispersant systems to effect optimum stable dispersions. Among the various common dispersing agents which have been found useful are:

5.2.1 *Tetrasodium Pyrophosphate, TSPP* ($\text{Na}_4\text{P}_2\text{O}_7$)—A freshly prepared 5 % solution of TSPP in water.

5.2.2 Sodium Hexametaphosphate (NaPO_3)₆.

5.2.3 Calgon T.¹¹

5.2.4 *Daxad* 30 (25 % active dispersant).¹²

5.2.5 *Dispex N-40* (40 % active dispersant).¹³

5.3 *Antifoaming Agent*—Pine oil, or capryl alcohol, or the equivalent.

6. Test Specimens and Sample

6.1 Sampling procedures should conform to the general practice outlined in Practice E 300.

6.2 Specimen Size—At least 50 g of well-pulverized

¹¹ Calgon T. Calgon Corp., P. O. Box 1346, Pittsburgh, PA 15230.

¹² Daxad 30, W. R. Grace, 3 Hanover Square, New York, NY 10004.

¹³ Dispex N-40, Allied Colloids Inc., 1 Robinson Lane, Ridgewood, NJ.

TABLE 1 Examples of Recommended Dispersants and Suggested Dispersant Concentrations

Pigment	Specific Gravity	Recommended Dispersant	Concentration, or Amount per 30-g Sample
Aluminum silicate	2.58	TSPP (NaPO ₃) ₆	6 mL of 5 % solution 0.15 to 0.25 g
Aluminum silicate (Predispersed)	2.58	TSPP	3 mL of 5 % solution
Magnesium silicate	2.86	TSPP	6 mL of 5 % solution
Calcium carbonate	2.71	Daxad 30	0.5 to 0.8 % "active" dispersant based on weight of dry pigment
		Calgon T	as above
Mica pigment	2.82	Calgon T	as above
TiO ₂ Pigment (Rutile, Anatase)	4.00	Calgon T TSPP	as above 4 mL of 5 % solution/20 g sample

pigment is required for each particle-size distribution determination.

7. Procedure

7.1 Determine the moisture content of the pigment by drying a 10-g sample to constant weight at 110°C essentially as described in Test Methods D 280.

7.2 Weigh out the equivalent of 30 ± 0.05 g of dry (moisture-free) pigment. (In the special case of predispersed aluminum silicate pigments, weigh out 30.10 ± 0.05 g of dry sample. For denser pigments such as TiO₂ (sp gr 4.0), the sample size should be reduced to about 20 g.)

7.3 *Preparation of the Pigment Dispersion*—Place 150 to 200 mL of distilled water in the mixing cup, start the mixer, add the pigment sample, and stir for 5 min. Then add the amount and type of dispersant (see Note 3) as suggested in Table 1, and mix for an additional 5 min. Loose agglomerates or flocs must be broken up into individual particles, but it is equally important to avoid mechanical attrition of the particles, especially in coarser pigments. For most common pigments, a 10-mm mixing period is sufficient.

7.4 Transfer the suspension into one of the graduated sedimentation cylinders, rinse out the mixing cup several times to ensure complete transfer of the sample, and finally dilute to the 1205-mL mark with distilled water adjusted to the same temperature as the water bath. If needed (but only if needed) add a drop of the anti-foaming agent. Close the mouth of the cylinder with a rubber stopper to fit (No. 13) or with the palm of the hand, and mix the suspension in the cylinder by inverting several times with shaking. Place the cylinder in the constant temperature bath (see Note 4) and stir the suspension occasionally with a glass rod to prevent sedimentation. Make a reference (blank) solution by filling the second cylinder to the mark with distilled water containing the same amount and type of dispersant as used with the pigment sample, and place along side in the bath. When both cylinders have attained the temperature of the bath, remove the cylinder with the suspension and remix by inversion and shaking as before.

NOTE 3—The accuracy of the test depends entirely on obtaining complete and stable deflocculation of the pigment so that each of its particles settles individually. The quantity and type of dispersing agent noted in Table 1 is usually satisfactory for the pigments listed when received in the usual form. Flocculation may occur due to attractive charges between the particles at which time the individual particles cease to fall independently. The pigment particles may come together to a variable degree to form flocs which descend as loosely associated groups or clusters of particles. When this occurs the apparent test results are much too "coarse." Signs of flocculation prior to or during the sedimentation test may be any or all of the following:

(a) Large clusters or pigment flocs suddenly become visibly apparent in the sedimenting suspension.

(b) An ill-defined, loosely mobile bed of sediment flocs may form at the bottom of the cylinder. If the pigment has been properly and stably dispersed, the sediment bed will be dense, solidly and tightly packed, well defined, and may appear to be graded in size if the particle size is widely distributed. Such a dispersed sediment bed is extremely difficult to resuspend by simple shaking and mixing.

(c) A very clear layer of solution may suddenly appear at the top of the cylinder, and this clear layer gradually descend with a definite demarcation between clear solution and "flocculated" sediment. If the pigment has been properly and stably dispersed, there will always be a slight residual turbidity extending to the surface of the suspension due to ultra-fine particles which remain in stable suspension even to the end of the test.

(d) Slight or partial flocculation (that is, when only a few flocs form intermittently during the settling period) is more difficult to detect. Evidence for this occurrence is also the clear zone of solution at the surface of the suspension as well as a qualitatively "looser" sediment bed. Slight or partial flocculation is particularly difficult to detect when the size distribution of the pigment is very narrow.

(e) One may be sure with confidence that flocculation is not a source of error in the test results by running a series of sedimentation tests on the same pigment sample using different dispersing agents over an appropriate range of dispersant concentrations. That sedimentation test that yields the finest distribution is the one least likely to contain flocs and most likely to represent the "actual" size-distribution. Dispersion requirements so determined then become "standard for that particular pigment type."

NOTE 4—The bath may be held at any constant temperature between 18 and 30°C, preferably only a few degrees above the normal maximum temperature of the laboratory.

7.5 Immediately after this final mixing, replace the sample cylinder in the bath, insert the hydrometer into it, and simultaneously start the timer. Immerse the hydrometer only slightly beyond the point where it floats freely.

7.6 Take hydrometer readings at such intervals that the total accumulative elapsed times form an approximate geometrical progression. A convenient series of time intervals is 1, 2, 2, 5, 10, 10, and 30 min and 1, 2, 3, 7, and 10 h, which together yield an elapsed time series of 1, 3, 5, 10, 20, and 30 min and 1, 2, 4, 7, 14, and 24 h. Read the hydrometer at the top of the meniscus, to the nearest estimated 0.001 units of specific gravity. After the first two or three time intervals, it is good practice to remove the hydrometer from the suspension, rinse it off, and float it in the reference (blank) solution. Great care must be taken to minimize any disturbance of the suspension by inserting and removing the hydrometer very slowly and evenly.

7.7 For each reading, record the elapsed sedimentation time, the temperature of the pigment suspension (or water bath) and the hydrometer readings for both the pigment suspension and the reference (blank) solution. See Table 2, which provides an example of a convenient data and computing form.

8. Calculation

8.1 *Percent of Pigment in Suspension*—Calculate for each

TABLE 2 Particle Size Distribution-Hydrometer Method—Calculation and Data Sheet[illegible]

reading the percent of dry pigment remaining in suspension at the level which the hydrometer measures the density of the suspension by the following equation:

$$P = (100\ 000/C) \times [G/(G - B)] \times [(R - B)/r]$$

where:

P = % pigment remaining in suspension,

C = original concentration of suspension in grams of pigment per litre, (for a sample of 30 g dry weight in 1205 mL of suspension, $C = 24.9$ g/L).

R_r = hydrometer reading in the pigment suspension,

B = hydrometer reading in the blank solution, and

G = specific gravity of pigment particles. (For example, in the absence of an actual determination of the specific gravity, a value of 2.6 may be taken for aluminum silicate pigment.)

8.1.1 Take the value of $(R_p - B)$ for the initial 1-min sedimentation reading to be a measure of the starting concentration. The percent remaining in suspension for each subsequent reading then becomes simply the ratio of its $(R_p - B)$ value to the initial $(R_p - B)$ value multiplied by 100.

NOTE 5—In the case of pigments reasonably free from coarse particles (larger than about 15 μm), the above calculations for the initial hydrometer readings should give values of 100 % for the percent remaining in suspension. Variation from this figure may be due to inaccurate measurements of the test specimen or volume of the suspension.

NOTE 6.—Readings taken during the first few minutes of sedimentation are more subject to possible error from several causes than are subsequent readings. These causes include settling of coarse particles on the hydrometer bulb, failure to attain complete temperature equilibrium, and the possible presence of entrained air in the suspension. Of these, the last named is perhaps the most frequent source of error. Treatment of the suspension before mixing with a small amount of an antifoam agent helps to prevent this.

8.2 Equivalent Spherical Diameter of the Particles:

8.2.1 Calculate the equivalent spherical particle diameter corresponding to the percent indicated by each hydrometer reading in accordance with Stokes' law. A particle of this equivalent spherical diameter is assumed to be at the surface

of the suspension at the beginning of sedimentation and to settle during the accumulated time to the level at which the hydrometer measures the suspension density.

8.2.2 Casagrande's nomographic solution of Stokes' law^{2,5} shown in Fig. 1 offers a convenient means for calculating equivalent spherical diameter. Its use requires calibration of the hydrometer in terms of the actual depth of its center of gravity below the surface of the suspension (H_r) for each hydrometer reading, R_r . The formula for calibration of hydrometers graduated in specific gravity units is shown in the upper right-hand corner of the chart. Calculated values of H_r for convenient intervals of R_r must be plotted on the $H_r - R_r$ scale for each individual hydrometer. Note that the hydrometer is calibrated for actual depth below the surface of the liquid, but that hydrometer readings of the density of the pigment suspension are made at the top of the meniscus, which is slightly raised above the surface of the suspension. For this reason a meniscus correction must be made. With the recommended hydrometer, it has been found that the addition of 0.0003 to the specific gravity reading made at the top of the meniscus gives values which correspond closely to the specific gravity reading at the surface level.

8.2.3 The use of the chart is explained in the key. A line through the value of S , the specific gravity of the sedimenting pigment particles and t , the temperature, gives the value for $A \times 10^3$, which is constant for a given temperature (of the bath or constant temperature room). A line through H , and T , the accumulated time, gives a value for v , the sedimentation velocity in centimetres per second; and finally, a line through $A \times 10^3$ and v gives the corresponding value for the equivalent spherical diameters, D , in millimetres.

9. Report

9.1 The report shall consist of a graph made by plotting for each hydrometer reading the percent remaining in suspension ("percent finer than") against the equivalent spherical diameter, using semilogarithmic graph paper with the particle size values on the logarithmic scale (see Fig. 2). A

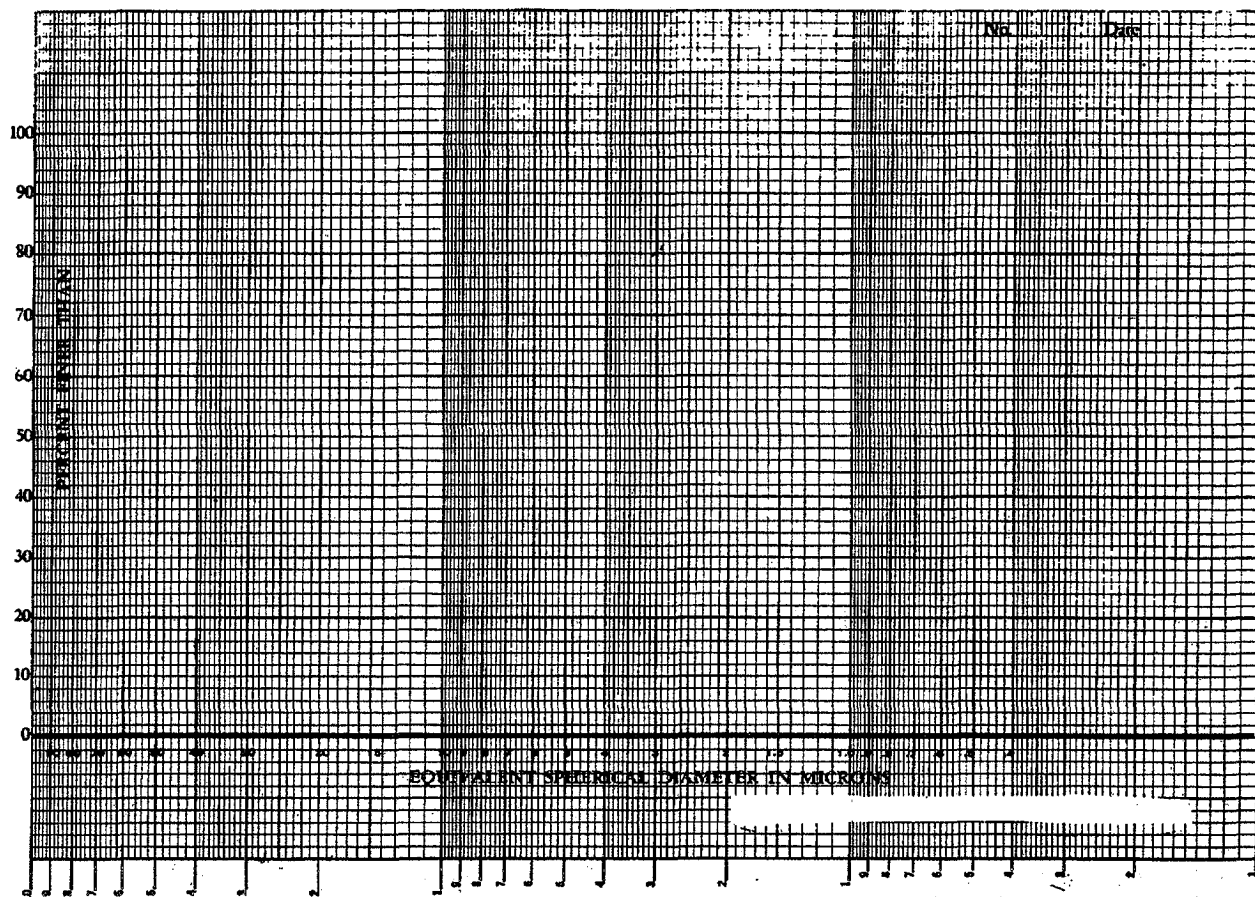


FIG. 2 Graph for Hydrometer Reading

value for the percent finer than 2 μm may be read from the graph and reported separately.

9.2 Although the entire cumulative particle size distribution curve may be of general interest, the particle size distribution criteria customarily used for extender pigments is either the weight percent less than 2 μm e.s.d., the weight median particle size, that is, the "50 % finer than" point on the distribution curve, or both. At either extreme of the particle size distribution curve, that is, the largest 10 % and the smallest 10 %, the test precision and accuracy tend to be considerably less than in the remaining 80 % mid-range of the distribution.

10. Precision

10.1 Based on interlaboratory studies of this test method

in which operators in 6 laboratories measured the particle size distribution of three extender pigments: aluminum silicate (kaolin), magnesium silicate (talc), and calcium carbonate, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

10.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 1 μm at the weight median ("50 % finer than") point, or 3 % at the percent less than 2 μm point on the cumulative distribution curve.

10.1.2 *Reproducibility*—Two results, each the mean of duplicate measurements obtained by operators in different laboratories, should not be considered suspect unless they differ by more than 2 μm at the weight median ("50 % finer than") point, or 5 % at the percent less than 2 μm point on the cumulative distribution curve.

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 Unreacted Toluene Diisocyanates in Urethane Prepolymers and Coating Solutions by Gas Chromatography¹

This standard is issued under the fixed designation D 3432; the number immediately following the designation indicates the year of original adoption 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 test method covers the determination of free toluene diisocyanates in urethane prepolymers and unpigmented coating solutions.

NOTE 1—This test method may also be useful for the determination of free isocyanate monomers of other types upon taking precautions to confirm suitability.

1.2 There is no reason to believe this test method would not work for pigmented coatings.

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 8.*

2. Referenced Documents

2.1 ASTM Standards:

D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints²

D 2698 Test Method for Determination of Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging²

E 260 Practice for Packed Column Gas Chromatography³

3. Summary of Test Method

3.1 A standard solution of the diisocyanate is analyzed by gas chromatography using an internal standard technique. The unknown specimen is then chromatographed, again using an internal standard, and a response factor is calculated from the standard solution. The unreacted diisocyanate content of the unknown is calculated from the chromatogram of the specimen.

4. Significance and Use

4.1 This test method provides a measurement of the unreacted diisocyanate content of urethane prepolymers and

coating solutions. This measurement is of basic importance to the health of the users.

5. Interferences

5.1 Under the test conditions described in this test method, other diisocyanates or impurities having identical or nearly identical elution times may interfere. It is generally necessary to know the identity of the diisocyanate being used in the specimen being tested, either by knowledge of the composition or by a separate analysis.

5.2 The temperature specifications for the injection port and column oven must be strictly followed to prevent thermal decomposition of the specimen. Decomposition can be detected by the failure of the recorder pen to return to the baseline after elution of the diisocyanate monomer.

5.3 Since resin solution specimens leave a nonvolatile residue in the injection port, the use of glass injection port liners is recommended. The glass liner should be changed daily or after each group of 10 specimens that are analyzed.

5.4 Since diisocyanates are highly reactive with water, a suitable drying cartridge should be used in the carrier gas line.

6. Apparatus

6.1 *Gas Chromatograph* equipped with flame ionization detector (FID) and capable of linear temperature programming to 300°C.

6.2 *Chromatographic Column*, 6.6 ft (2-m) by 0.125-in. (3.2-mm) outside diameter, 0.085-in. (2.1-mm) inside diameter stainless steel (or glass, 2-mm inside diameter), packed with a 10 % methyl vinyl silicone⁴ on 80/100 mesh flux-calcined diatomaceous earth.⁵ The packing is prepared by dissolving 1.0 g of the methyl vinyl silicone in 50 mL of methylene chloride. This solution is placed in an evaporating dish and 9 g of the diatomaceous earth is added with gentle stirring. The evaporating dish may be gently heated on a steam bath or evaporated at room temperature. The packing should be gently stirred, periodically, until dry and free-flowing. A newly packed column should be conditioned overnight at 200°C with a flow of dry carrier gas.

NOTE 2—Any other column, either packed or capillary, may be substituted if the required separation is obtained.

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 April 28, 1989. Published July 1989. Originally published as D 3432 - 80. Last previous edition D 3432 - 80.

² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 14.01.

⁴ UC-W-98, licensed by Union Carbide Corp., 39 Olde Ridgebury Rd., Danbury, CT 06817-0001, has been found satisfactory for this purpose and is available from most suppliers of gas chromatographic accessories.

⁵ Chromosorb W-HP, manufactured by Manville Sales Corp., Filtration and Minerals, P.O. Box 5108, Denver, CO 80217-5108, has been found satisfactory for this purpose and is available from most suppliers of gas chromatographic accessories.

NOTE 3—Other methods of packing preparation may be used, provided a free-flowing, uniformly coated packing results.

6.3 *Chromatographic Syringe*, 10 μ L.

6.4 *Analytical Balance*, accurate to 0.1 mg.

6.5 *Glassware*—All glassware used should be oven-dried to eliminate moisture.

6.6 *Recorder, Recording Integrator or Computer* with appropriate software.

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 *Ethyl Acetate* (99+ %), urethane grade, dried over a Type 5A molecular sieve.

7.3 *Standard Solution A*—Weigh about 5.0 g of 1,2,4-trichlorobenzene (TCB) to 0.1 g and dilute to 500 mL with ethyl acetate (1 mL = 10 mg TCB).

7.4 *Standard Solution B*—Weigh about 0.25 g of the diisocyanate to 1 mg into a dried 100 mL volumetric flask and add 10 mL of Standard Solution A by pipet. Dilute to 100 mL with ethyl acetate and mix well. This solution has a shelf life of one day.

7.4.1 If the unreacted diisocyanate content of the prepolymer or coating solution exceeds 1 %, then Standard Solution B should be made by diluting 0.25 g of the diisocyanate monomer, weighed to 1 mg, with Standard Solution A to 10 mL in a dried 10-mL volumetric flask. No additional solvent is added in this case.

7.5 *Toluene Diisocyanate (TDI)*, 99.5+ % assay, mixture of 80 % 2,4- and 20 % 2,6-isomers.

7.6 *1,2,4-Trichlorobenzene (TCB)*.

8. Hazards

8.1 Diisocyanates can be toxic when inhaled or adsorbed through the skin. Provide adequate ventilation and wear impermeable gloves (for example, rubber, polyethylene, etc.) when handling these materials. Consult the manufacturer's Material Safety Data Sheet (MSDS) before handling any of these materials.

9. Procedure

9.1 Prepare the gas chromatograph (refer to Practice E 260) as follows:

9.1.1 *Injection Port Temperature*—150°C. When first using the method, it may be desirable to experiment with slightly higher and lower temperatures to determine if the injection port temperature is high enough for complete volatilization of the monomer, but low enough to avoid decomposition of the polymer (see 5.2).

9.1.2 *Oven Temperature*—150°C (for TDI). For other isocyanates, isothermal or temperature programmed analysis may be necessary.

9.1.3 *Detector Temperature*—300°C.

9.1.4 *Carrier Gas*—Dry helium or nitrogen (see 5.4), flow rate adjusted to obtain proper elution time (3.7 min) of the internal standard (30 mL/min nominal).

9.2 Separation may be required for products containing pigments. Pigments may be removed using procedures given in Methods D 2372 or D 2698. Report the values for unreacted diisocyanate in the product on a pigment-free basis.

9.3 Calibrate by injecting a 1 μ L aliquot of Standard Solution B (7.4) and obtain a chromatogram.

9.4 *Specimens Having 0.1 to 1.0 % Unreacted Diisocyanate*:

9.4.1 Weigh a 5-g specimen (± 0.01 g) into a tared 25-mL (7-dram) vial. Pipet 1 mL of Standard Solution A into the vial. Add 10 mL of ethyl acetate and mix thoroughly.

NOTE 4—Additional solvent may be added if required to reduce viscosity of the specimen. Increase the specimen size to be injected proportionally.

9.4.2 Inject a 1 μ L aliquot of the test solution (Note 5). Obtain a chromatogram, internally or by temperature programming as required.

NOTE 5—One-microlitre syringes tend to "freeze" during use with polymer solutions; therefore, a 10 μ L syringe is recommended. Employ the proper technique to inject the desired specimen size.

9.5 *Specimens Having 1 to 10 % Unreacted Diisocyanate*:

9.5.1 Weigh a 5-g specimen (± 0.01 g) into a tared 25 mL (7-dram) vial. Pipet 10 mL of Standard Solution A into the vial and mix thoroughly. No additional solvent is added in this case.

9.5.2 Inject a 1 μ L aliquot of the test solution (Note 5).

9.6 The order of elution (see Fig. 1) is:

Compound	Retention Time, min
Ethyl acetate	0.4
TCB	1.9
TDI	3.7

9.7 Construct baselines under the internal standard and isocyanate peaks by drawing a straight line from valley to valley under the respective peaks. With the standard solution, the isocyanate peak may return to the baseline more promptly than with sample solutions that contain polymeric species. However, it is important that the tail of the diisocyanate peak returns to the baseline. Failure to do so indicates thermal breakdown that will result in high values. Illustrations of peak shapes are shown in Fig. 1. Due to the nonsymmetrical shape of the peaks, measure peak areas by electronic integrator or planimeter.

10. Calculation

10.1 Calculate the response factor (RF) as follows (see 9.3):

$$RF = (W_1 \times A_{TCB}) / (W_{TCB} \times A_1)$$

where:

W_1 = weight of diisocyanate in Standard Solution B,

W_{TCB} = weight of TCB in Standard Solution B,

A_1 = area of diisocyanate peak(s) (see 9.6 for measurement), and

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 Pharmacopoeia."

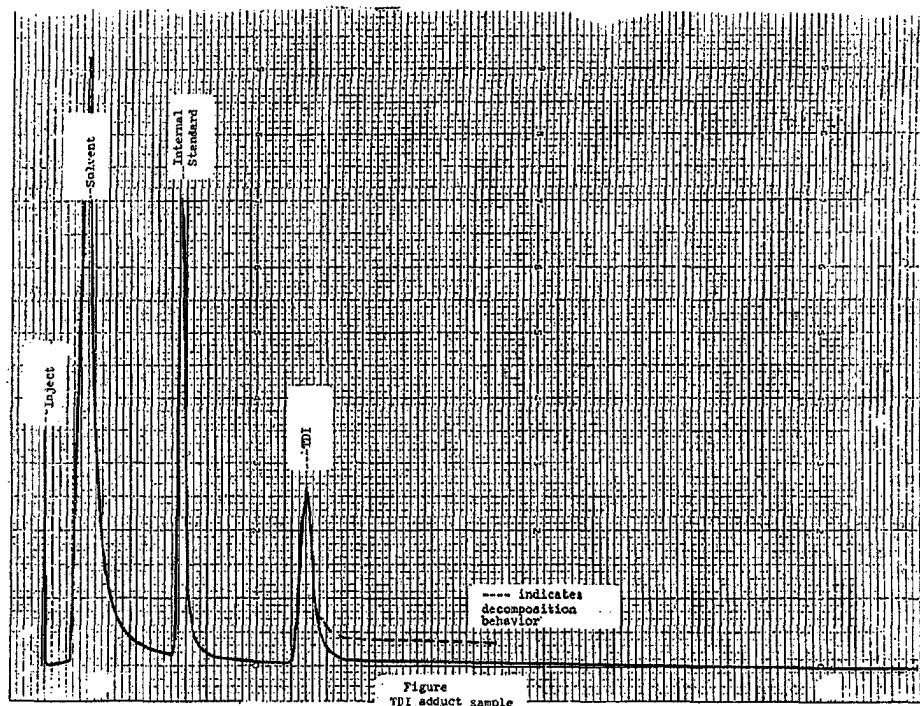


FIG. 1 TDI Adduct Sample

A_{TCB} = area of TCB peak (see 9.6).

10.2 Calculate the weight percent unreacted diisocyanate, D , as follows:

10.2.1 For specimens having 0.1 to 1.0 % unreacted diisocyanate:

$$D = (A_1 \times RF \times W_{TCB} \times 100) / (A_{TCB} \times W)$$

where:

A_1 = area of diisocyanate peak(s),

A_{TCB} = area of TCB peak,

W = specimen weight, g,

W_{TCB} = weight of TCB per millilitre in Standard Solution A, g, and

RF = response factor from 10.1.

10.2.2 For specimens having 1 to 10 % unreacted diisocyanate:

$$D = (A_1 \times RF \times W_{TCB} \times 100) / (A_{TCB} \times W)$$

11. Report

11.1 Report the results to two decimal places, expressed as

weight percent unreacted diisocyanate in the solution (example: 0.45 % unreacted TDI).

12. Precision and Bias

12.1 On the basis of an interlaboratory test of this test method in which one operator in each of six different laboratories tested three unpigmented materials covering a range of three levels of TDI in urethane coating solutions, the within-laboratory standard deviation was found to be 0.016 % and the between-laboratory standard deviation was found to be 0.085 %. Based on these standard deviations, the following criteria should be used for judging the precision of results at the 95 % confidence level:

12.2 *Repeatability*—Duplicate results by a single operator should be considered suspect if they differ by more than 0.06 % absolute.

12.3 *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 0.31 % absolute.

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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 Ashing Cellulose¹

This standard is issued under the fixed designation D 3516; the number immediately following the designation indicates the year of original adoption 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 four ashing methods for cellulose. These are intended for use on unbleached and bleached cellulose in sheeted or bulk fiber form. Each one of the test methods has advantages, so that preference applications exist for all four.

1.2 The test methods appear as follows:

	Sections
Test Method A—Ash in Cellulose at 575°C	6 to 11
Test Method B—Sulfated Ash in Cellulose at 575°C	12 to 17
Test Method C—Ashing Cellulose by Schoniger Oxidation	18 to 22
Test Method D—Wet Ashing of Cellulose for Inorganics	23 to 29

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 Note 12.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1193 Specification for Reagent Water²
- D 1348 Test Methods for Moisture in Cellulose³

3. Summary of Test Methods

3.1 *Test Method A, Ash in Cellulose at 575°C*—This test method measures the ash content of cellulose, which is defined for this test as the residue remaining after ignition at $575 \pm 25^\circ\text{C}$ until all carbon has been burned off. It is the simplest of the four test methods for the determination of ash content only, and it should not be considered as a standard preparative procedure for elemental analysis.

NOTE 1—The ash content at this ignition temperature is a reasonable measure of the mineral salts and inorganic foreign matter in the cellulose. The weight of ash obtained varies with the temperature of ignition. Higher temperatures (850°C) will convert calcium carbonate and other carbonates to the oxides and thus give lower values for ash. The composition of ash may vary with the pulping process employed for manufacture, which limits the significance of the ash determination in absolute terms.

3.2 *Test Method B, Sulfated Ash in Cellulose at 575°C*—In this ashing procedure the metal salts are converted to sulfates by treatment with sulfuric acid and ignition at $575 \pm$

25°C to overcome limitations of Test Method A. It should not be considered as a standard preparative test method for elemental analysis.

NOTE 2—Conventional dry ashing at high temperature (over 850°C) results in loss of carbon dioxide from metal carbonates, decomposition of magnesium sulfate, and loss of sodium salts. Ashing at lower temperatures (575°C) gives a better estimate of the inorganic impurities, but has the disadvantage of requiring a long ignition time and also the heat produced by the burning pulp can raise the temperature far above 575°C for a short period of time, converting calcium carbonate to the oxide in varying amounts. The sulfated ash method overcomes these objectionable features and has additional advantages. It is relatively insensitive to ignition time. Ash weight remains constant during weighing, and because the ash does not melt it will not attack the crucible.

3.3 *Test Method C, Ashing Cellulose by Schoniger Oxidation*—This test method is generally applicable to ashing cellulose for subsequent analysis of inorganic constituents. The procedure utilizes the Schoniger technique in which the cellulose is burned in a combustion flask and all combustion products are dissolved in an aqueous medium, thus avoiding the possibility of physical loss of sample such as can occur in dry ashing (Test Methods A and B). A limitation is the relatively small sample of 1 g, which can be ashed without excessively large apparatus in which to carry out the oxidation step. Small specimens may not be adequately representative where a particulate contaminate is involved.

3.4 *Test Method D, Wet Ashing of Cellulose for Inorganics*—This test method is recommended for ashing cellulose for subsequent analysis of inorganic constituents. The procedure employs saturation of the cellulose with a concentrated solution of hydrogen peroxide followed by incremental addition to a small volume of concentrated sulfuric acid. It has the advantage over Test Method C of not requiring special apparatus and can be readily applied to samples of 5 to 10 g. The test method becomes tedious and less convenient for use where larger specimens such as 50 or 100 g are required. Specimens this large, although not generally required, may be necessary for the determination of trace levels of constituents such as manganese or silica. Smaller samples may not be adequately representative where a particulate contaminate is involved.

4. Significance and Use

4.1 Ash content gives an estimation of the inorganic content of cellulose samples. The presence of high levels of ash can be expected to be detrimental to the process of making cellulose derivatives. It also provides a rough estimate of silica content which can have a significant effect on the performance of filters in cellulose derivative manufacturing facilities.

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 D01.36 on Cellulosics.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 3516 - 76. Last previous edition D 3516 - 76 (1985).

² Annual Book of ASTM Standards, Vols 06.03 and 11.01.

³ Annual Book of ASTM Standards, Vol 06.02.

5. Purity of Reagents

5.1 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.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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification D 1193.

TEST METHOD A—ASH IN CELLULOSE AT 575°C

6. Apparatus⁵

6.1 *Balance*, with an accuracy to 0.05 g is required for weighing cellulose samples.

6.2 *Analytical Balance*, with a sensitivity of 0.1 mg is required. It should be checked with Class S weights.

6.3 *Drying Oven*, with the capability of maintaining a temperature of $105 \pm 2^\circ\text{C}$.

6.4 *Desiccator*, such as silica gel, indicating drierite, or magnesium perchlorate are suitable.

6.5 *Muffle Furnace*, electric, capable of maintaining a temperature of $575 \pm 25^\circ\text{C}$, is recommended.

NOTE 3—Back to front temperatures in muffle furnaces frequently exceed the $\pm 25^\circ\text{C}$ range, so that operating limits should be established for a given furnace.

6.6 *Dish*, platinum, 100-mL capacity or larger. Porcelain crucibles are not recommended.

6.7 *Tongs*, approximately 20 in. (0.5 m) long, with nickel-chromium or platinum tips.

7. Test Specimen

7.1 The amount of cellulose selected for each ash determination depends on the ash content of the test specimen and should be varied so that the weight of the ash will be not less than 10 mg and preferably over 20 mg. Table 1 gives suggested sample sizes.

7.2 Selection of the specimen shall be in such a manner as to be representative of the lot being tested.

NOTE 4—No set sampling procedure has been established to be applicable to all samples. It will suffice to specify that the specimen shall be representative of the lot being sampled.

8. Procedure

8.1 Weigh out, to the nearest 0.05 g, sufficient cellulose to give the correct range of ash weight (see Table 1). At the same time, a separate specimen (3 to 5 g) should be weighed

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 Suitable apparatus is available from A. H. Thomas Co., Vine St. at 3rd, P. O. Box 779, Philadelphia, PA 19105.

TABLE 1 Suggested Sample Sizes

Ash, %	Moisture-Free Cellulose, g
Over 0.5	5
0.20 to 0.5	10
0.12 to 0.2	20
0.08 to 0.12	30
0.04 to 0.08	40
Less than 0.04	50

and dried at 105°C for a moisture determination.

NOTE 5—Test Methods D 1348, Method B is suitable. However, accuracy of the moisture test is not critical for the ash determination and any method capable of obtaining the percent oven dry within 0.5 % of the true value is adequate.

8.2 Heat the platinum dish in the muffle furnace at 575°C for 15 min, cool and weigh to the nearest 0.1 mg. Place the weighed cellulose in the platinum dish and place the dish on the open door of the muffle furnace, previously set and allowed to reach equilibrium at 575°C . Allow the cellulose to char without flame by gradually moving the dish into the muffle, and continue ashing with the door closed for 1 h.

NOTE 6—With some pulps, the character of the ash inhibits complete carbon removal and gray or black specks persist after 3 h of ignition. The addition of a few drops of distilled water to the ash after 8.3 followed by drying and reignition at 575°C for 1 h or more may be required. Extreme cases may require a second treatment with water.

NOTE 7—If the sample is too large, either separate ashings are required or successive additions of cellulose are made with extreme care so as not to disturb the ash. 8.2 is then repeated between each addition.

8.3 Remove the dish from the muffle furnace and allow to cool somewhat. Place it in the desiccator and allow it to cool to room temperature.

NOTE 8—Care must be taken at all times to keep drafts away from the light, fluffy ash.

8.4 Weigh the dish and ash to the nearest 0.1 mg. Reignite for a 15-min period and reweigh. Repeat as required to obtain constant weight.

9. Calculation

9.1 Calculate the ash percent of moisture-free cellulose, E , as follows:

$$E = \frac{(C - B)(100)(100)}{(A)(D)} \quad (1)$$

where:

A = weight of specimen as is, g,

B = weight of dish, g,

C = weight of dish plus ash, g, and

D = oven dry cellulose in specimen, %

10. Report

10.1 Report the results to the nearest 0.01 %. Duplicate determinations should check within approximately 5 %.

11. Precision and Bias

11.1 A precision of 8 % relative at the 95 % confidence level is found for specimens at the 0.05 % ash level.

11.2 No statement of bias can be made as no suitable reference material exists for determining bias.

TEST METHOD B—SULFATED ASH IN CELLULOSE AT 575°C

12. Apparatus and Reagent

12.1 Hot Plate.

12.2 Additional apparatus shall be in accordance with Section 6.

12.3 Sulfuric Acid—Concentrated H₂SO₄.

13. Test Specimen

13.1 The amount of cellulose selected for each ash determination depends upon the sulfated ash content of the test specimen and should be varied so that the weight of the ash will be not less than 10 mg and preferably over 20 mg. Table 1 gives suggested specimen sizes for Test Method A. The table is applicable to sulfated ash also, providing the percent ash column is taken as percent sulfated ash.

NOTE 9—The sulfated ash values will be higher than the ash obtained by ignition of the cellulose without addition of the sulfuric acid. The magnitude of the difference will depend upon the proportions of different inorganic salts present, but based on the following conversion factors for the salts commonly present, will generally be about twice the regular ash:

$$\frac{\text{MgSO}_4}{\text{MgO}} = 2.99; \frac{\text{CaSO}_4}{\text{CaO}} = 2.43; \frac{\text{MgSO}_4}{\text{MgCO}_3} = 1.43 \quad (2)$$

$$\frac{\text{CaSO}_4}{\text{CaSO}_3} = 1.36; \frac{\text{Na}_2\text{SO}_4}{\text{Na}_2\text{CO}_3} = 1.34 \quad (3)$$

13.2 Selection of the specimen shall be in such a manner as to be representative of the lot being tested (see Note 4).

14. Procedure

14.1 Weigh out to the nearest 0.05 g sufficient cellulose to give the correct range of ash weight (see Table 1). At the same time, weigh a separate specimen (3 to 5 g) and dry to constant weight at 105 ± 2°C for a moisture determination (see Note 5).

14.2 See 8.2.

14.3 Remove the dish and allow it to cool to room temperature (see Notes 7 and 8).

14.4 Moisten the ash with 1 to 2 mL of concentrated H₂SO₄ and heat on a hot plate until all the dense white fumes of sulfur trioxide (SO₃) are liberated.

14.5 Place in the muffle furnace and ignite at 575°C for 1 h. If carbon persists repeat 14.3, 14.4, and 14.5.

14.6 After the last trace of carbon has disappeared, ignite in the muffle furnace for 1 h at 575°C.

14.7 Remove the dish and allow it to cool somewhat before placing it in desiccator and cooling to room temperature.

14.8 Weigh the dish and sulfated ash to the nearest 0.1 mg. Reignite for a 15-min period and reweigh. Repeat as required to obtain constant weight.

NOTE 10—To remove the ash from the dish, clean with boiling hydrochloric acid.

NOTE 11—On igniting sulfated ash at 575°C, there should be no fusion with the dishes, even when ashing cellulose that gives an alkaline ash.

14.9 Prepare a reagent blank for the H₂SO₄ corresponding to the number of millilitres of H₂SO₄ used in 14.4 and 14.5, by the addition of the acid to a platinum dish after ignition

and true weight was determined, and carrying through 14.4 to 14.8 as they apply.

15. Calculation

15.1 Calculate the sulfated ash, *S*, as percent of moisture-free cellulose, as follows:

$$S = \frac{(C - B - R)(100)(100)}{(A)(D)} \quad (4)$$

where:

A = weight of specimen, g,

B = weight of dish, g,

C = weight of dish plus sulfated ash, g,

R = weight of H₂SO₄ reagent blank residue, g, and

D = oven-dry cellulose in specimen, %.

16. Report

16.1 Report the results to the nearest 0.01 %. Duplicate determinations should check within approximately 5 %.

17. Precision and Bias

17.1 A precision of 15 % relative is found for specimens in the 0.1 to 0.3 % ash range.

17.2 No statement of bias can be made as no suitable reference material exists for determining bias.

TEST METHOD C—ASHING CELLULOSE BY SCHONIGER OXIDATION

18. Apparatus

18.1 *Combustion Flasks*, 5000 mL, round bottom, borosilicate glass, with 65/40 ball and joint stopper. The flasks are equipped with perforated platinum specimen carriers sealed into the extended stem of the flask stopper.

18.2 *Igniter*, suitable for igniting the specimen in the flask. An infrared igniter is preferred.

18.3 *Specimen Wrappers*, black paper.

18.4 *Pipet*, 5 mL.

18.5 *Volumetric Flask*, 50 mL.

19. Reagents

19.1 *Deionized Water*, high-purity, with a conductivity <1.0 μmho/cm at 25°C.

19.2 *Absorption Solution*—0.2 N H₂SO₄ prepared from concentrated H₂SO₄.

20. Procedure

20.1 By hand, tear a 2- to 3-g air-dried test portion of the sample into 1 to 1.5-cm pieces and store in closed airtight, glass or polyethylene bottles.

20.2 Weigh a 1-g portion of the prepared sample to ± 1 mg, and wrap in a square of black ignition paper.

20.3 Weigh a second portion of sample, approximately 1 g to ± 1 mg, and determine the moisture content in accordance with Test Methods D 1348. This test method determines the weight loss on a 1-g specimen placed in a gravity convection oven at 105°C for 4 h.

20.4 Add by pipet, 10 mL of 0.2 N H₂SO₄ as an absorption solution to the combustion flask.

20.5 Place the specimen contained in the wrapper into the platinum carrier and insert into the flask.

20.6 Allow a stream of oxygen from a low-pressure, regulated supply (2 to 3 lb./in.² (14 to 21 kPa)) to flow into the flask for a period of 30 s to displace the air. Secure the stopper in the flask with the pinch clamp and place the expansion balloon on the side tube.

NOTE 12: Precaution—It is required that laboratory goggles be worn during 20.6, 20.7, and 20.8 as a safety measure.

20.7 Place the flask behind a safety shield or in a hood with a protective door.

20.8 Turn on the igniter and focus the infrared light beam on the fuse strip of the black paper wrapper until ignition occurs. Allow the specimen to burn completely and cool the flask for 1 h so that all vapors are absorbed in the solution.

20.9 Transfer the solution to a 150-mL beaker and rinse the flask with two 15-mL portions of deionized water and add to the absorption solution in the beaker.

20.10 Transfer the contents of the beaker to a 50-mL volumetric flask and rinse with 2 to 4-mL portions of deionized water. Add water to bring to 50-mL volume. Identify as *Schoniger ash solution*.

20.11 Ash a black wrapper identical in weight to one used for wrapping the specimen by following 20.4 to 20.10. Identify as *wrapper and absorption solution blank*.

21. Calculation

21.1 Calculate the concentration equivalent for oven-dry cellulose, *C*, in *Schoniger ash solution*, as follows (Note 13):

$$C = \frac{(A)(B)}{(50)(100)} \quad (5)$$

where:

A = weight of cellulose specimen, and
B = oven-dry cellulose in specimen, %

NOTE 13—Use of the solution for subsequent analysis assumes parallel treatment of both the *Schoniger ash solution* and the wrapper absorption solution blank to provide for the analytical blank correction.

NOTE 14—The purpose in specifying the calculation of equivalent oven-dry cellulose in grams per millilitre is to provide for convenient use of aliquots for subsequent elemental analyses.

22. Precision and Bias

22.1 A precision of 20 % relative is found for specimens at the 0.2 % ash content level. The operator's technique is very important in keeping the precision at a high level.

22.2 No statement on bias can be made as no suitable reference material exists for determining bias.

TEST METHOD D—WET ASHING OF CELLULOSE FOR INORGANICS

23. Summary of Test Method

23.1 A test specimen of pulp is torn into small pieces approximately 1 cm across, saturated with a solution of hydrogen peroxide, and added one piece at a time to a small volume of concentrated sulfuric acid.

23.2 The cellulose is oxidized to carbon dioxide and water. The water is volatilized by the heat of the reactions involved.

23.3 An analyst experienced with the technique can ash 30 g of pulp into an initial volume of only 10 mL of sulfuric acid in about 30 min. Total procedural time for converting a

30-g specimen to solution in a form suitable for aliquoting for metals analysis is about 2 h.

24. Apparatus

24.1 *Balance*, sensitivity to ± 0.01 g.

24.2 *Beakers*, Berzelius, 300-mL.

24.3 *Beakers*, 150-mL.

24.4 *Cylinder*, graduated-pharmaceutical, 50-mL.

24.5 *Forceps*, 20-cm stainless steel, TFE-fluorocarbon tipped.

24.6 *Hot Plate*, sensitive temperature control, ceramic-top surface or alternative suitable for use with platinum ware. Metal surfaces are not suitable.

24.7 *Pipet*, 10-mL.

24.8 *Rubber Bulb*, for filling pipet.

25. Reagents

25.1 *Hydrogen Peroxide*, 50 % H_2O_2 .

NOTE 15—The actual concentration is not critical, but the absolute minimum is 30 % below which the test fails. One should be aware that hydrogen peroxide solution tends to lose strength upon standing.

25.2 *Sulfuric Acid*, concentrated H_2SO_4 .

26. Hazards

26.1 The procedure specifies many repetitive manual additions of a strong oxidant and organic material to concentrated acid. It is imperative that the analyst wears protective equipment for the eyes and hands, and works in a fume hood free of distraction during the steps in Section 28.

27. Sampling

27.1 Select an appropriate sized sample as specified in the methods for inorganic elements to be determined, with an allowance for a moisture determination.

27.2 Tear the pulp by hand into small pieces approximately 1 cm across.

27.3 Weigh the selected weight of the specimen to 0.01 g.

27.4 Determine the moisture content on a separate specimen in accordance with Test Methods D 1348.

27.5 Correct the specimen weight (27.3) to the oven-dry basis for subsequent calculations.

28. Procedure

28.1 Pipet 10 mL of H_2SO_4 to a 300-mL tall form Berzelius beaker.

28.2 Place approximately 10 g of the weighed pulp specimen into a 150-mL beaker.

28.3 Pour approximately 50 mL of H_2O_2 into a 50-mL graduated cylinder.

28.4 Distribute approximately 30 mL of the measured portion of H_2O_2 over the 10 g of specimen allowing the pulp to saturate with it.

28.5 Add 2 to 3 mL of H_2O_2 to the 10 mL of H_2SO_4 by allowing it to run down the side of the beaker. Carefully swirl to mix, resulting in gas evolution. If mild boiling is not evident, heat gently on hot plate to induce it, then remove the beaker from the hot plate.

28.6 With stainless tongs, add the pulp saturated with H_2O_2 one piece at a time allowing for almost complete destruction between each addition.

NOTE 16—A porcelain spatula has been used in place of stainless tongs to transfer the pulp to the acid and may be preferable for specimens in powdered state. Pulp vary in their behavior to the ashing step. Thirty grams of some pulps have been ashed with only 2 or 3 additions of H_2O_2 separate from the pulp. These are required whenever the acid retains a brown or black residue after destruction of a pulp addition. With other pulps, ten or more 1 to 2 mL additions of hydrogen peroxide may be required.

28.7 After the initial 10-g portion of pulp has been ashed, repeat 28.2 to 28.6 on a second 10-g portion of the weighed specimen.

28.8 Repeat 28.7 until the total specimen is ashed.

28.9 Rinse the sides of the beaker containing ashed specimen with distilled water from a wash bottle. Use the minimum amount required.

28.10 Swirl the beaker to mix the rinse water with the acid.

28.11 Place the beaker on the hot plate and heat to the point that dense fumes of H_2SO_4 evolve.

NOTE 17—If the acid discolors, remove the beaker from the hot plate, allow it to cool, and add 2 to 3 mL of H_2O_2 to the beaker. Return the beaker to the hot plate and continue heating. Repeat this step as required to attain a colorless solution.

28.12 Continue heating to remove the excess acid.

NOTE 18—The final volume of acid is selected relative to subsequent analyses, however 2 to 3 mL has been satisfactory for most applications.

28.13 Remove the beaker from the hot plate, allow it to cool, and with extreme care add distilled water, allowing it to flow down the side of the beaker to the required dilution. Transfer it to a 25-mL volumetric flask, and adjust to volume.

NOTE 19—If an ash-determination is desired, transfer the solution to a preweighed platinum dish, take to dryness, and ignite at approximately 400°C prior to weighing. The residue may then be taken up in distilled water for elemental analysis. Some sulfates may be difficult to solubilize, particularly ferric sulfate, requiring subsequent heating.

28.14 Prepare a reagent blank corresponding to the 10 mL of H_2SO_4 and the volume of H_2O_2 used by slow addition of increments of the peroxide to the acid followed by gentle heating to remove the oxygen and water. Complete the blank in accordance with 28.12 and 28.13.

28.15 The solutions from 28.13 and 28.14 are now suitable for subsequent elemental analysis.

29. Precision and Bias

29.1 A precision of 20 % relative is found for specimens at the 0.5 % ash content level.

29.2 No statement of bias can be made as no suitable reference material exists for determining bias.

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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 Aluminum Silicate Pigments (Anhydrous)¹

This standard is issued under the fixed designation D 3619; the number immediately following the designation indicates the year of original adoption 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 covers the white pigments that consist substantially of anhydrous (calcined) natural aluminum silicates² (of the 1+1 layer type) and are restricted to those minerals which conform to the chemical compositional limits prescribed herein and which can be suitably processed to what is known as paint pigment quality.

2. Referenced Documents

2.1 ASTM Standards:

- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out³
- D 718 Test Methods for Analysis of Aluminum Silicate Pigment³
- D 1203 Test Methods for Volatile Loss from Plastics Using Activated Carbon Methods⁴
- D 1483 Test Method for Oil Absorption of Pigments by Gardner-Coleman Method³
- D 2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment³
- D 3360 Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments³
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁵

3. Composition and Properties

3.1 *Preparation*—The pigment shall be made by grinding, milling, washing, purifying, or otherwise processing, natural hydrous aluminum silicates followed by heat treatment (calcination) sufficient to reduce the residual percent loss on ignition to a maximum of 0.5 % and otherwise conforming to the composition requirements (weight percent) given in Table 1.

3.2 *pH*—The pH of a water slurry of the pigment shall be within a range from 4.5 to 5.5 unless otherwise agreed upon between the purchaser and the seller.

3.3 *Water-Soluble Matter*—The water-soluble matter shall be not more than 0.60 %.

3.4 *Wet-Sieve Residue*—The pigment shall contain no more than 0.02 % wet-sieve residue retained on a 45 μm (No. 325) sieve (grit or coarse particles) except as may be

agreed upon between the purchaser and the seller.

3.5 *Brightness (Color)*—The brightness (color reflectance) shall conform to the following requirements:

3.5.1 The brightness shall be not less than 90 % nor more than 92 % expressed as percent reflectance of standard illuminant C at 457 nm compared to a freshly smoked magnesium oxide surface by means of an accepted integrating sphere reflectance spectrophotometer⁶ or a monochromatic reflectance meter,⁶ or

3.5.2 The color may be specified by actual determination of dominant wavelength, hue, and spectral efficiency as can be calculated from the tristimulus integration of the reflectance curve, or

3.5.3 The color shall be equal, within agreed upon tolerances, to that of a reference standard agreed upon between the purchaser and the seller.

3.6 *Oil Absorption*—The oil absorption shall be within the range from 40 to 60 g/100 g as determined by the spatula rub-out test (Test Method D 281) or within a range from 60 to 80 g/100 g as determined by the Gardner-Coleman test method (Test Method D 1483) (see 5.1.4). It may be noted that reproducible test results using the spatula rub-out test method (Test Method D 281) are found to be very difficult to obtain because of a very high electrostatic charge imparted to the anhydrous pigment during the rub-out procedure. Although a spatula rub-out test result can be obtained, it has been found by experience that much more reproducible results can be obtained by use of the Gardner-Coleman test method.

3.7 *Particle Size*—Anhydrous aluminum silicate pigments may be furnished in several types or grades whose properties are dependent in part on the average particle size or particle size distribution about the average, or both; characteristic particle shape is as thin, flat, and laminated plates. The particle size or size distribution, or both, shall be within agreed upon tolerances to that of a standard agreed upon between the purchaser and the seller, but in general, the average particle size should be close to 2 μm .

4. Sampling

4.1 Two samples shall be taken 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, samples shall be taken from different packages in the ratio of two samples for each 10 000 lb, except that for shipments of less than 10 000 lb, two samples shall be taken. At the option of the

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 D01.31 on Pigment Specification.

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² Synonymous terms are kaolinite and china clay.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vol 08.01.

⁵ Annual Book of ASTM Standards, Vol 15.05.

⁶ The General Electric Reflectance Spectrophotometer (Diano Corp.), the General Electric Reflectance Meter, and the ElRefo Blue Reflectance Meter have all been found suitable for this purpose.

TABLE 1 Pigment Composition Requirements
(Percent by Weight)

NOTE—The major constituents shown in the analysis are combined as complex aluminum silicate and do not exist as free oxides.

	Ideal	Typical	Range	Max
Aluminum oxide, Al_2O_3 , %	45.91	44.4	43-49 ^A	...
Silicon dioxide, SiO_2 , %	54.09	52.3	56-50 ^B	...
Iron oxide, Fe_2O_3 , %	...	0.1	...	0.
Titanium dioxide, TiO_2 , %	...	2.0	...	2.5
Calcium oxide, CaO , %	...	...	...	0.1
Sodium oxide, Na_2O , %	...	...	...	0.1
Potassium oxide, K_2O , %	...	...	...	0.1
Other oxides, %	...	...	...	0.1
Free moisture (105°C), %	...	<0.5	...	0.5
Loss on ignition (1000°C), %	...	<0.5	...	0.5
Free silica (either amorphous or as crystalline quartz), %	...	...	...	1.0

^A Permitting up to about 5 % excess Al_2O_3 , for example, as allophane.

^B Permitting up to about 5 % excess SiO_2 , for example, but no more than 1 % of free silica (SiO_2) either amorphous or as crystalline quartz.

purchaser, the samples may be tested separately or after blending the samples from the same production unit in equal

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quantities to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods, where applicable. Test procedures not covered by ASTM test methods shall be agreed upon between the purchaser and the seller, except as follows:

5.1.1 *Hydrogen Ion Concentration (pH)*—Prepare a sample having 20 % solids, but otherwise in accordance with Test Methods D 1203. Determine the pH in accordance with Test Method E 70, except maintain sufficient agitation of the slurry such that all particles are in suspension at the time of measurement.

5.1.2 *Water-Soluble Matter*—Test Method D 2448.

5.1.3 *Coarse Particles*—Test Methods D 718.

5.1.4 *Oil Absorption*—Test Methods D 281 or D 1483.

5.1.5 *Particle Size Distribution*—Test Method D 3360.

5.1.6 *Chemical Analysis*—Test Methods D 718.



Standard Test Method for Residual Vinyl Chloride Monomer Content of Poly(Vinyl Chloride) Resins, Compounds, and Copolymers by Solution Injection Technique¹

This standard is issued under the fixed designation D 3680; the number immediately following the designation indicates the year of original adoption 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 can be used to determine small amounts of residual vinyl chloride monomer (VCM) in poly(vinyl chloride) (PVC) resins. It may be possible to use it for other resins or compounds containing vinyl chloride provided there are no other volatile components present with the same retention time as vinyl chloride monomer.

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 9.*

2. Referenced Documents

2.1 ASTM Standards:

- E 260 Practice for Packed Column Gas Chromatography²
- E 355 Practice for Gas Chromatography Terms and Relationships²

3. Terminology

3.1 Abbreviations:

- 3.1.1 VCM—vinyl chloride monomer.
- 3.1.2 PVC—poly(vinyl chloride) resin.
- 3.1.3 RVC—residual vinyl chloride monomer.
- 3.1.4 DMAC—dimethyl acetamide.
- 3.1.5 THF—tetrahydrofuran.

4. Summary of Test Method

4.1 The resin, compound, or copolymer specimen is dissolved in a suitable solvent and is then analyzed by conventional gas chromatographic (GC) techniques. Analysis time can be decreased by utilizing a backflush mode to eliminate the solvent.

5. Significance and Use

5.1 Poly(vinyl chloride) resins must contain a minimum possible amount of unreacted, or free vinyl chloride monomer.

5.2 Under optimum conditions approximately 1 ppm

VCM can be detected in PVC resins.

6. Interferences

6.1 Any material that elutes at approximately the same retention time as VCM will cause erroneously high RVC results.

6.2 Special care must be exercised in interpreting the RVC results obtained from the analyses of PVC copolymers or compounds. Many ingredients are added to PVC resins to obtain compounds with desired properties for each particular end use. These additives, or components in them, could possibly have the same retention time as VCM. In general, if a PVC compound apparently has a much larger VCM content than the starting resin, then the presence of some interfering component can be suspected as the probable cause of the larger than expected amount in the compound.

7. Apparatus

7.1 *Gas Chromatograph*, with dual flame ionization detector, and heated injection port with removable glass inserts.

7.2 *Recorder*, 0 to 1-mV full scale.

NOTE 1—Electronic integrators, or computer systems can be used to obtain peak areas and make the required calculations.

7.3 *Analytical Balance*, capable of weighing to ± 0.0001 g.

7.4 *Soap Film Flowmeter*.

7.5 *Stopwatch*.

7.6 *Pressure Regulators*, for all the required gas cylinders.

7.7 *Filter Dryer Assemblies*, for each of the GC gas cylinders.

7.8 *Vials*, 6-dram, with polyethylene-lined screw caps.³

NOTE 2—Caps with wax liners should not be used for sample dissolution.

NOTE 3—Vials and caps should be used only once and discarded.

7.9 *Serum Bottles*, 60-mL, and *Stoppers*.⁴

7.10 *Liquid Sampling Syringes*, 10 and 50- μ L. *GC Liquid Sampling Syringes*, 10 and 50- μ L.

7.11 *Gas Syringes*, 0.1 and 1-mL,⁵ equipped with valve

³ Vials #60957-L, available from Kimble Glass Co., Div. Owens Corning, Inc., One Sea Gate, Toledo, OH 43666, or equivalent, has been found satisfactory for this purpose.

⁴ Fisher No. 3-220C bottles and 3-225 stoppers, available from Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219, or equivalent, have been found satisfactory for this purpose.

⁵ Series A-2 syringes, available from Precision Sampling Corporation, 16 Tech Circle, Natick, MA 01760, or equivalent, have 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 D01.33 on Polymers and Resins.

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² *Annual Book of ASTM Standards*, Vol. 14.01.

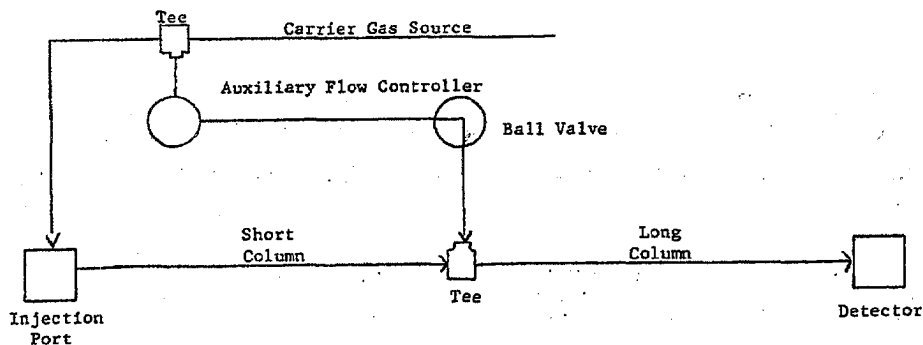


FIG. 1 Schematic of Backflushing Method

locks.

7.12 *Ball Valve*, two-way, stainless steel.⁶

7.13 *Flow Controller*.⁷

7.14 *Screw Cap*, 6-dram, with *Needle Seal Septa*.⁸

7.15 *Reciprocating or Wrist-Action-Type Shaker*.

7.16 *Column and Packings*—Any of the following may be used:

7.16.1 $\frac{1}{8}$ in. outside diameter by 1 ft plus $\frac{1}{8}$ in. outside diameter by 6 ft stainless steel packed with 80/100 mesh Poropak S or Poropak Q-S.⁹

7.16.2 $\frac{1}{8}$ in. outside diameter by 20 ft stainless steel packed with 20 % Tergitol E-35¹⁰ on 60/80-mesh Chromosorb W-AW.¹¹

7.16.3 $\frac{1}{8}$ in. outside diameter by 3 ft plus $\frac{1}{8}$ in. outside diameter by 6 ft with 0.2 % Carbowax 1500¹² on Carbowax "C".¹³

7.16.4 $\frac{1}{8}$ in. outside diameter by 20 ft packed with 20 % Apiezon L¹⁴ on 60/80-mesh Gas-Chrom Q.¹⁵

NOTE 4—The above-mentioned column lengths and packing have been used successfully for this analysis. However, any column that will elute VCM in reasonable lengths of time (1 to 4 min) can be used provided any interfering components are not eluted at the same time as VCM.

⁶ Catalog No. 4152-A-316, available from Whitey Co., 318 Bishop Rd, Highland Heights, OH 44143, or equivalent, has been found satisfactory for this purpose.

⁷ Model 8744A, Brooks Instrument Co., Div. Emerson Electric Co., 407 W. Vine St., Hatfield, PA 19440, or equivalent, has been found satisfactory for this purpose.

⁸ Precision Sampling Corp. Catalog No. 614063 and No. 644350, or equivalent, has been found satisfactory for this purpose.

⁹ Poropak is a trademark for porous polymer beads developed by the Dow Chemical Co., 2020 Dow Center, Midland, MI 48674, and marketed for gas chromatographic use by Waters Associates, Inc.

¹⁰ "Tergitol E-35," a trademark of the Union Carbide Corp., 39 Old Ridgebury Rd, Danbury, CT 06817-0001, or equivalent, has been found satisfactory for this purpose.

¹¹ "Chromosorb W-AW," a trademark of Manville Products Corp., 717 17th St., P.O. Box 5108, Denver, CO 80217, or equivalent, has been found satisfactory for this purpose.

¹² "Carbowax 1500," a trademark of Union Carbide Corp., or equivalent has been found satisfactory for this purpose.

¹³ Carbowax, a trademark of Supelco Inc., Supelco Park, Bellefonte, PA 16823, or equivalent, has been found satisfactory for this purpose.

¹⁴ "Apiezon," a trademark of the James F. Biddle Co., 510 Township Line Rd., Blue Bell, PA 19422, or equivalent, has been found satisfactory for this purpose.

¹⁵ "Gas-Chrom Q," a trademark of Applied Science Laboratories, 111 Bullard, Parkway Suite 208, P.O. Box 16118, Tampa, FL 33687, or equivalent, has been found satisfactory.

7.17 *Jars*, 2-oz (60-mL), with aluminum or polyethylene-lined screw caps.

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,¹⁶ where such specifications are available. 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 *Air*, breathing or water-pumped.

8.3 *N,N-Dimethyl Acetamide (DMAC)* (Note 5).

NOTE 5—Either solvent (DMAC or THF) may be used. However, they must be free of any contaminants that have the same retention time as VCM or any internal standard that may be used. To determine this, inject 10 μ L of solvent and obtain a chromatogram under the conditions of Section 11.

8.4 *Hydrogen*, prepurified or zero gas.

8.5 *Nitrogen*, oxygen-free.

NOTE 6—Helium may also be used as the carrier gas.

8.6 *Pentane*, at least 99 %.

8.7 *Tetrahydrofuran (THF)* (Note 5).

8.8 *Vinyl Chloride Monomer*, polymerization grade.

9. Hazards

9.1 In addition to other precautions, conduct all operations including the preparation of standards, transfer of solvents, and column backflushing (if the technique described in 11.1 is used) in a properly functioning fume hood.

10. Sampling and Storage

10.1 Keep all resin samples in tightly sealed jars. Samples should be analyzed within 24 h. If 24 h are exceeded, report the age of the sample. Use 2-oz (60-mL) bottles with aluminum or polyethylene-lined screw caps, completely filled, and seal with electrical tape.

¹⁶ "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."

10.2 Compound samples can be kept in the same type jars. The remaining precautions are not required as long as the jar is tightly sealed.

11. Preparation of Chromatograph

11.1 Install the chromatographic column and condition at least overnight at the temperature limit for the packing being used. Do not connect the exit end of the column to the detector while conditioning. Turn off hydrogen and air to the detector while the column is disconnected. One method of achieving the backflushing of the solvent is shown schematically in Fig. 1. The two columns are separated by a "Tee" fitting. Connect the short column between the injection port and one end of the "Tee." Connect the longer column between the other end of the "Tee" and the detector. Connect a line from the outlet of the ball valve to the center inlet of the "Tee." This auxiliary line may be filled with a suitable column packing if desired to act as a pressure buffer when switching to the backflush mode. Insert "Tee" into the carrier gas supply line to supply the auxiliary flow controller.

11.2 Adjust the carrier gas flow as follows (Note 7):

11.2.1 Close off the auxiliary flow with the ball valve.

11.2.2 Set flow at 30 mL/min with the chromatograph flow control valve.

11.2.3 Remove the septum from the injection port.

11.2.4 Open the ball valve.

11.2.5 Again adjust the flow to 30 mL/min with the auxiliary flow control valve.

11.2.6 Close the auxiliary flow with the ball valve.

11.2.7 Replace the septum on the injection port.

11.2.8 The system is now ready for an analysis.

NOTE 7—If possible with the chromatograph being used, adjust the carrier flow at the analysis temperature.

11.3 Set detector air and hydrogen flows and pressures at the optimum conditions for the make and model of chromatograph being used.

11.4 Set temperatures as follows:

11.4.1 Optimize oven (chromatograph column) temperature to elute VCM between 1 and 4 min after injection.

11.4.2 Injection Port—200°C.

11.4.3 Detector—At least 20°C above maximum column temperature.

NOTE 8—Other systems of backflushing have been evaluated. However, none of them have been very successful. This is probably due to the fact that a small amount of solvent seems to remain in the column and injection port. This small amount of solvent is always detected at the extreme detector sensitivity required for this analysis. It may then take as long as 30 to 40 min to return to a stable baseline. Two alternative methods of flushing solvent from the column are described in Annex A1.

NOTE 9—A trial-and-error method is used to determine the optimum time to remove and replace the septum holder for backflushing the solvent. The objective is to wait as long as possible to be sure that the VCM has passed through the short column but still to retain all, or most, of the solvent in the short column before removing the septum to backflush it, then to replace the septum after the solvent has backflushed out through the injection port.

12. Calibration by External Standard

12.1 Prepare solution mixtures of varying amounts of VCM in solvent to cover the expected parts per million range of concentration. These can be prepared as described for the

internal standard method in 13.2 and 13.3 without adding the internal standards. Obtain a gas chromatogram for each calibration standard using the parameters listed in Section 11. Prepare a chart plotting the peak height (or area) from the chromatograms versus the known concentration of VCM in each calibration standard to establish a detector response curve.

13. Calibration with an Internal Standard

13.1 Prepare a polymer solvent solution containing a known amount of internal standard as follows:

13.1.1 Partially fill a 1-L volumetric flask with solvent.

13.1.2 Into a separate beaker or flask containing a tared weight of solvent, add, by means of a syringe, approximately 0.0150 g of pentane. Reweigh the container immediately and determine the exact amount of pentane added to the nearest 0.1 mg.

13.1.3 Immediately upon reweighing, quantitatively add the contents of the container to the 1-L volumetric flask. Do so by rinsing the container with aliquot portions of the solvent. Bring to the mark with fresh solvent. Mix thoroughly and calculate the grams of pentane per 20 mL of solution as follows:

$$\text{Grams pentane/20 ml} = \text{weight of pentane/50}$$

13.1.4 Store the internal standard solution, thus prepared, in an amberized bottle. Label the bottle Solution A.

13.2 Weigh 50.00 ± 0.01 g of the solvent into a tared, serum bottle to the nearest 0.1 mg. Immediately seal the vial. Record the weight of the solvent.

13.3 Using a 0.1-mL Pressure-Lok¹⁷ syringe, add about 0.025 mL of liquid vinyl chloride monomer to the sealed bottle. Reweigh to the nearest 0.1 mg. Determine the weight of vinyl chloride added and the total weight of the solution. Calculate the grams of vinyl chloride per gram of solution. Identify this as Solution B.

$$\text{Grams VCM/gram Solution B} = \frac{\text{grams VCM}}{\text{grams VCM} + \text{grams solvent}}$$

13.4 Pipet 20.0 mL of Solution A into a vial equipped with a screw cap and a TFE-fluorocarbon "Mininert"¹⁸ valve. Immediately cap, weigh, and record the weight of the solution and the sealed container.

13.5 Using the 1-mL syringe, inject approximately 1 mL of Solution B into the contents of the vial. Reweigh and determine the weight of Solution B that was added to Solution A. Once prepared, this standard is not to be opened. Prepare a new standard each time a fresh Solution A is prepared.

13.5.1 Calculate the amount of vinyl chloride in the prepared standard as follows:

$$\text{weight of VCM} = \text{weight of Solution B} \times \text{grams VCM/grams of Solution B (from 13.3)}$$

13.6 Using the operating conditions described in Section

¹⁷ Pressure-Lok syringes, manufactured by Precision Sampling Corp., are made specifically to maintain liquids and gases under pressure within the syringe.

¹⁸ Mininert valves and caps are manufactured by the Precision Sampling Corp. The caps are designed so that the contents of the vial can be removed with a syringe without removing the cap. The valves attach to the lever fitting of a syringe to permit sampling or storage under pressure.

11 and the procedure described in Section 14, inject 10 μ L of this standard solution containing known amounts of pentane and VCM into the chromatographic system. Determine a Response Factor (RF) as outlined below from the ratio of peak areas of the vinyl chloride and pentane and their known weights:

$$RF = \frac{\text{weight of pentane} \times \text{peak area VCM}}{\text{weight of VCM} \times \text{peak area pentane}}$$

13.6.1 Example:

	Weight	Area
Pentane	0.0002940	6471
VCM	0.0005190	8921

$$RF = \frac{0.0002940 \times 8921}{0.0005190 \times 6471} = 0.781$$

NOTE 10—For highly accurate determinations of the response factor, it is advisable to run the calibration standards a minimum of five times. The average of these runs is then used. The stability of the response factor must be verified at least once per week.

14. Sample Analysis Procedure

14.1 Sample Preparation:

14.1.1 Mix the sample to be analyzed completely so that the portion selected will be as representative as possible. Weigh 1 g of the sample to be analyzed into a tared 6-dram vial and recap the vial and contents and determine the specimen weight to the nearest 0.1 mg.

14.1.2 By means of a pipet, add 20.0 mL of solvent or Solution A (see Section 13) containing the solvent and internal standard to the specimen vial.

NOTE 11—Twenty millilitres of solvent yields a 5 weight % solution. Ten millilitres of solvent will yield a 10 weight % solution.

14.1.3 Place the vial on a shaker until the specimen is completely dissolved. Once it is visually in solution, the specimen must be shaken for at least one additional hour. Leave all specimens on the shaker until they are analyzed.

14.2 Inject 10 μ L of the prepared specimen.

NOTE 12—A larger volume of prepared specimen (greater than 10 μ L) may be utilized when analyzing for very low levels of vinyl chloride. Care must be taken, however, to back flush the extra solvent from the system before injecting subsequent specimens. Also with larger injection volumes (20 to 40 μ L) there is sometimes a possibility that some of the solvent does not volatilize. For cases in which backflush capability does not exist, the use of a smaller specimen size and another solvent, for example, acetone, has been found to be acceptable.

NOTE 13—The syringe used to inject specimens into the chromatographic system must be rinsed with solvent between each specimen injection.

NOTE 14—The glass insert in the injection port will catch and retain the solid particles from the specimen that would gradually foul the column. Loosely pack the $\frac{1}{4}$ in. at the outlet end of the glass tube with glass wool. Change this trap after every specimen. Change the septum at the beginning of each shift. Do not force the septum tightly against the glass tubing as this will restrict flow to the column.

14.3 At the predetermined time (see Note 9) remove the septum and actuate the ball valve to backflush the solvent.

14.4 At the end of the predetermined time (see Note 9) first actuate the ball valve to stop the backflush flow, then replace the septum on the injection port.

14.5 Allow at least 2 min for the baseline to equilibrate. Allow the equipment to sit longer if necessary until there is

minimal baseline drift at the attenuation to be used for the analysis.

NOTE 15—After running the last specimen prior to a break in the analysis routine (of a limited time span) leave the system in the backflushing mode. An example of this would be a lunch break. When the down time will be of a longer nature, such as an entire shift, perform the normal backflushing operation after the last specimen and then leave the equipment with carrier flow in the forward position.

15. Calculation—External Standard Method

15.1 Determine the monomer content of the specimen from the calibration chart (see section 12). Compare the peak height (or area) of the VCM peak of the chromatogram to the calibration curve and read the corresponding concentration in parts per million (ppm).

NOTE 16—The use of the external standard technique is extremely dependent on specimen size. Amount of specimen injected must be as much the same as it is possible to obtain for each injection.

NOTE 17—The concentration value obtained from the calibration curve is the total VCM in solution. In order to convert the VCM concentration to a specimen weight basis, if a 10 weight % specimen solution was prepared, the chart value must be multiplied by a factor of 10. If a 5 weight % specimen solution was made, then the multiplying factor is 20.

16. Calculation—Internal Standard Method

16.1 Calculate weight percent VCM as follows:

$$\text{Weight \% VCM} = \frac{\text{area VCM} \times \text{weight pentane in sample} \times 100}{\text{area pentane} \times \text{sample weight} \times \text{RF}}$$

16.2 Calculate parts per million VCM as follows:

$$\text{ppm VCM} = \frac{\text{area VCM} \times \text{weight pentane in sample} \times 10^6}{\text{area pentane} \times \text{sample weight} \times \text{RF}}$$

NOTE 18—See 13.6 for calibration instructions for determining the Response Factor (RF).

16.3 Example:

$$\begin{aligned} \text{Weight pentane in specimen} &= 20 \text{ ml} \times 0.0000147 \text{ g/ml} = 0.0002940 \text{ g} \\ \text{RF} &= 0.781 \\ \text{Specimen weight} &= 0.9385 \text{ g} \\ \text{Area VCM} &= 1211 \\ \text{Area pentane} &= 11868 \\ \text{Weight \% VCM} &= \frac{1211 \times 0.0002940 \times 100}{11868 \times 0.9385 \times 0.781} \\ &= 0.0041 \% \text{ VCM, and} \\ \text{ppm VCM} &= 0.004 (\times 10\ 000) = 4 \text{ ppm VCM.} \end{aligned}$$

17. Precision and Bias

17.1 *Precision*—The following values were determined for the coefficients of variation of this test method, using an internal standard, on the basis of an interlaboratory test program (1976) involving twelve laboratories reporting averages of duplicate analyses.

17.1.1 Precision for Resin Samples:

Intralaboratory	8.3 %
Interlaboratory	24.8 %

17.1.2 Precision for Compounds:

	At 2 ppm	At 30 ppm
Intralaboratory	5.8 %	5.8 %
Interlaboratory	28.8 %	10.3 %

17.2 *Bias*—Since no absolute method is available for comparison, no statement of accuracy can be presented for this test method.

ANNEX

(Mandatory Information)

A1. POLYMER SOLVENT REMOVAL BY ALTERNATIVE CARRIER GAS FLOW SYSTEMS

A1.1 Alternative carrier gas flow systems for removing the polymer solvent from the column system are shown schematically in Figs. A1.1 through A1.4 on the following pages. Valve actuating times, both to eliminate the solvent and to

return to baseline equilibrium conditions must be determined experimentally for each individual column system being used.

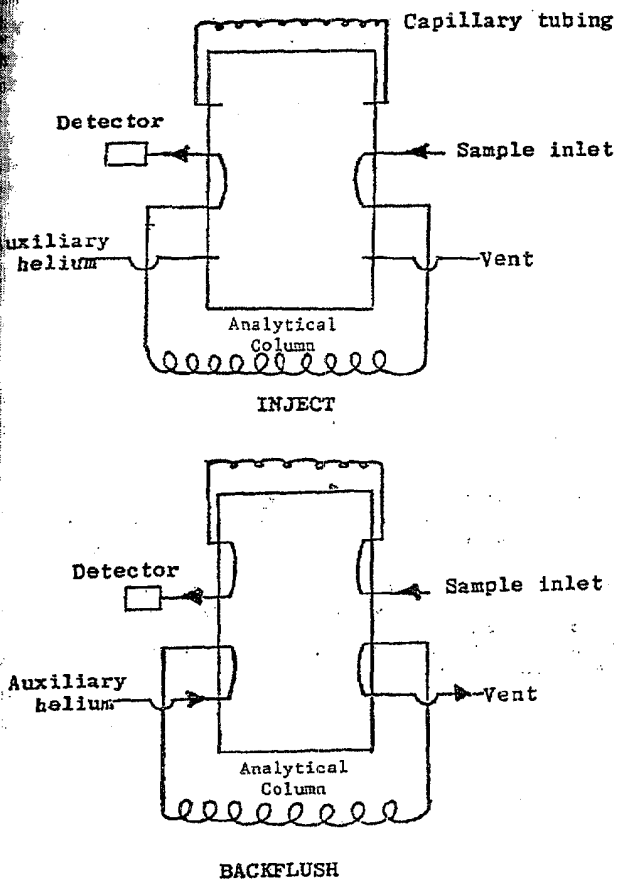


FIG. A1.1 Schematic of Flow System Utilizing an 8-Port Sliding Plate Valve with a Single Column

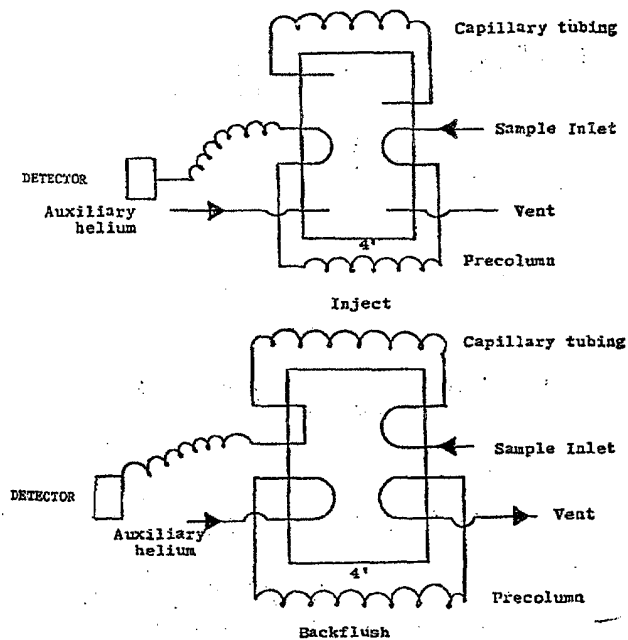


FIG. A1.2 Schematic of Flow System Utilizing an 8-Port Sliding Plate Valve with Two Columns

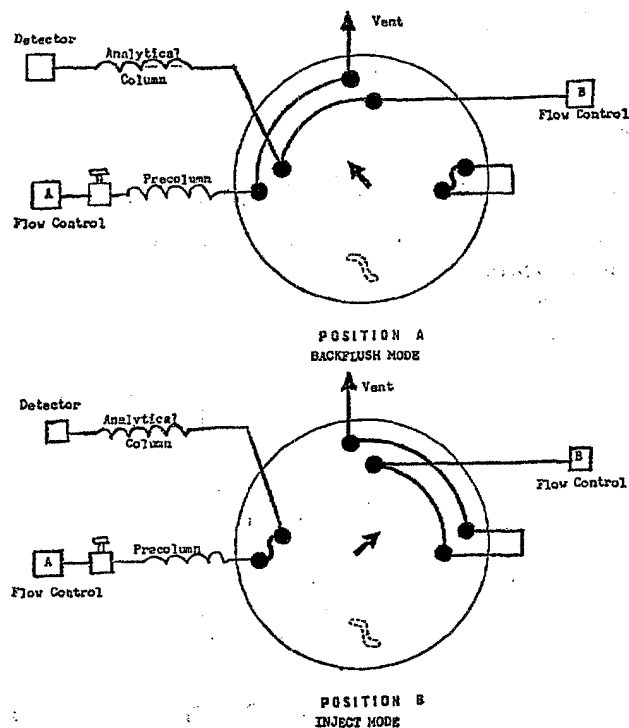
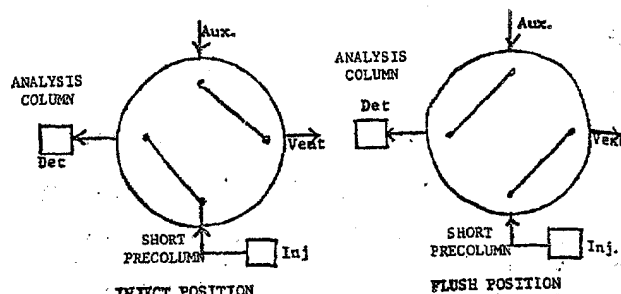


FIG. A1.3 Schematic of Flow System Utilizing a 6-Port Rotary Valve with Two Columns



NOTE—At a predetermined time, after the monomer peak has passed through the precolumn, the valve is activated to flush the solvent out the injection port and short precolumn.

FIG. A1.4 Schematic of Flow System Utilizing a 4-Port Rotary Valve with Two Columns

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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 Ratio of Anatase to Rutile in Titanium Dioxide Pigments by X-ray Diffraction¹

This standard is issued under the fixed designation D 3720; the number immediately following the designation indicates the year of original adoption 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 ratio of anatase to rutile in titanium dioxide pigments. The method is also applicable to pigment mixtures and pigmented coatings containing titanium dioxide.

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 Test Method for Chemical Analysis of White Linseed Oil Paints²

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

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings²

3. Summary of Test Method

3.1 The X-ray diffraction pattern obtained from a material is characteristic of that material. The intensity of a diffraction peak entirely due to one component of a mixture is dependent upon the amount of that substance in the mixture. To a minor extent the peak intensity of the component is also dependent on the mass absorption coefficient of other materials present. Since the test method utilizes the ratio of the intensities of diffraction peaks of two chemically similar materials, it is expected that the effects of other constituents will be the same for both materials.

3.2 The intensity of the diffraction maxima for anatase and rutile is measured by X-ray diffractometry. The intensity of the anatase peak is converted to anatase content relative to rutile and the rutile content is determined by difference.

3.3 The X-ray diffraction measurement is made on single pigments, pigment mixtures, films of pigmented coatings, and films prepared from liquid coatings, if interfering materials are not present. When interfering materials are

present, the pigment is separated from the redissolved (or ignited film, or from the liquid coating and treated to isolate the titanium dioxide.

4. Significance and Use

4.1 This test method is used by titanium dioxide pigment manufacturers and users for process control and product acceptance.

5. Interferences

5.1 Calcium sulfate interferes, but its effect is eliminated by chemical removal (see Test Method D 215). It is desirable to assure by analysis that any residual CaSO_4 is considerably less than the level of anatase being sought. The insoluble residue after removal of calcium sulfate should be ignited above 700°C. Chrome yellow and the valentinite form of antimony trioxide also interfere if not removed. High amounts of iron render analysis difficult due to increased background (see Note 1). Additives, such as antimony and zinc, and impurities, such as niobium and zirconium, are generally present in solid solution and thus would not have interfering diffraction peaks. Surface treatments such as silica and alumina do not interfere. Extreme differences in particle size between the anatase and rutile portions affect the results.

NOTE 1—Background scatter due to high iron levels in a sample may be reduced by use of a cobalt or molybdenum target tube in place of the copper target tube. The background may be eliminated for all practical purposes by use of a curved crystal monochromator equipped with a graphite crystal in conjunction with a copper target tube.

5.2 In the calculation of converting the ratio to percent it is implicitly assumed that the sum of anatase and rutile is 100 %, an assumption normally made in TiO_2 pigment systems. Other materials present would interfere to the extent that they dilute the sample. The third polymorphic form of TiO_2 , brookite, would have such an effect. However, it reportedly does not occur in commercial titanium dioxide pigments.

6. Apparatus

6.1 *X-Ray Diffractometer*—The principle components of this instrument are: (a) X-ray generator, (b) copper target X-ray tube, (c) goniometer, (d) detector, (e) electronic circuit panel, (f) computer (if used), and (g) strip chart recorder or printer.

6.2 *Operating Conditions*—The X-ray tube voltage and filament current and other settings are selected to record X-ray diffraction peaks of weak intensities.

6.2.1 *Nickel Filter*, to remove Cu K beta radiation if a monochromator is not used. Cu K beta radiation will produce a diffraction line from the rutile phase of TiO_2 that

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 3720 - 78. Last previous edition D 3720 - 84.

² Annual Book of ASTM Standards, Vol 06.01.

appears at the same 2θ angle as the anatase analytical line.

6.3 Typical Apparatus Conditions:

6.3.1 *High-Voltage Power Supply*—Select X-ray tube voltage, filament current, and other settings so that 0.1 % anatase generates a signal with intensity at least four times the noise level.

6.3.2 *Detector*—Scintillation detector operating at optimum voltage.

6.3.3 *Pulse Height Analyzer*—Settings will depend on instrumentation used.

6.3.4 *Slits*—Receiving slit, 0.15°; others, 1°.

6.3.5 *Chart Speed*, 1/2 in. (12.5 mm)/min.

6.3.6 *Goniometer Speed*, 1/4°/min, scanning or 0.02° steps with 4.8 s per step.

6.3.7 *Time Constant*, 5.

6.3.8 *Scanning Range*, $2\theta = 28$ to 24° .

6.3.9 *Scale Factors*—For samples of low anatase content a scale factor of 100 counts full scale is normally used for the $2\theta = 26$ to 24° range (anatase diffraction maximum), and a scale factor of 5000 counts full scale is normally used for the $2\theta = 28$ to 26° range (rutile diffraction maximum). The scale factors may be changed depending on the level of anatase expected in the sample.

7. Reagents

7.1 *Purity of Reagents*—Untreated rutile and anatase grades of titanium dioxide pigments are used to make synthetic standards for calibration. The crystalline structures of the reagents must be 100 % rutile and 100 % anatase as determined by examining these materials by X-ray diffraction to assure this purity.

7.2 *Mixing Reagents*—A series of rutile standards of varying rutile content are prepared to cover the range of interest by thoroughly mixing known amounts of 100 % rutile and 100 % anatase together. The pigments are dispersed in a solvent such as isopropyl alcohol and then mixed, followed by air drying. This dry cake is ground up using a mortar and pestle to ensure homogeneity.

8. Hazards

8.1 X-ray producing equipment can be dangerous to both the operator and persons in the immediate vicinity unless safety precautions are strictly observed. Refer to the manufacturer's instruction manual. Exposure to excessive quantities of X-radiation may be injurious to health. 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 has passed 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" and other standard publications on the subject. Inquiries should be made of state agencies as to existing requirements.

9. Specimen Preparation

9.1 Powder Samples:

9.1.1 Packing the specimen in the specimen holder to obtain a planar surface is one of the most important phases in X-ray analysis. Ripples or indentures in the specimen surface cause variations in the test because of an error of eccentricity and a change in the intensities of the peaks. The chance of preferred orientation of pigmentary TiO_2 is remote because of its small particle size.

9.1.2 Pack the specimen in a die suitably constructed to accommodate the specimen holder for the goniometer. Apply constant pressure with a hydraulic press (see Note 2). Alternatively, place the specimen holder on a flat, smooth firm surface and pack the specimen into the opening by applying constant and firm pressure with a flat blade. The possibility of variations among different bags of the same lot should be considered.

NOTE 2—Use of a set procedure with a hydraulic press will improve specimen preparation uniformity compared to hand-packed specimens. This is particularly true where more than one person is involved in specimen preparation.

9.2 Coatings on Metal Panels:

9.2.1 Cut the coated panel into the proper shape and size to fit an adjustable specimen holder and analyze without further preparation. This assumes that interferences are not present (see Section 5).

9.3 Liquid Coating Samples:

9.3.1 Liquid coating samples without interfering materials present (see Section 5) are analyzed as cast films of the total paint on aluminum or other suitable substrate. A 1 to 2-mil (25 to 50- μm) dry film thickness is adequate.

9.3.2 Liquid paint samples with interfering materials present (see Section 4) must not be analyzed until the interfering components are removed. This can be accomplished by centrifuging (Methods D 2371 or D 2698) to separate the total pigment. The separated pigment is treated according to 19.2.1 of Method D 215 to obtain interference-free TiO_2 . Quantitative results are not required for either separation procedure.

10. Procedure

10.1 Record the X-ray diffraction maxima of the anatase and of the rutile peak by scanning the range between $2\theta = 26$ to 24° and $2\theta = 28$ to 26° , respectively. Scan each range twice.

10.2 Draw the baseline between the lowest points of the trace on each side of the peak. Measure the peak height above the baseline expressed in chart units, centimetres, counts per second, or other convenient units for the anatase and rutile peaks. Average the net peak heights of the duplicate runs.

10.3 Since only ratios are used, the peak heights need only be corrected for differences, if any, in the scale factors used for the two peaks. Any consistent units can be used, not just counts per second.

10.4 Prior to and after analyzing the unknown sample, analyze one of the standards to verify instrument stability.

11. Calculations

11.1 Determine the value of the constant, K , as follows

$$K = (I_a/I_r)/(W_a/W_r)$$

where:

I_a = net intensity of the anatase diffraction peak,

I_r = net intensity of the rutile diffraction peak,

W_a = weight of anatase, and

W_r = weight of rutile.

11.1.1 K is equal to the slope of a calibration plot obtained when the intensity ratio I_a/I_r is plotted as a function of W_a/W_r , using data derived from a series of standards that span the concentration range of interest. Routine calibration procedures are followed.^{3,4}

NOTE—The value of K is influenced by the instrument conditions selected in Section 5. Data for standards and test samples must be collected using the same conditions.

11.2 Calculate the anatase level relative to rutile from the peak intensities above background as follows:

$$\text{Percent anatase} = \frac{1}{1 + K \frac{I_r}{I_a}} \times 100$$

³ Myers, H. and Spurr, R., *Analytical Chemistry*, Vol 29, 1957, p. 760.

⁴ Zingaro, P. W., *Norelco Reporter*, Vol 5, No. 5-6.

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12. Precision

12.1 On the basis of an interlaboratory test of this test method in which four operators in four laboratories tested two materials with composition approaching 100 or 0 % anatase or rutile, the within-laboratory standard deviation was found to be 0.06 % for a single determination. The between-laboratories standard deviation was found to be 0.07 % absolute for results each the mean of two determinations. Based on these standard deviations, the following criteria should be used for judging the precision of results at the 95 % confidence level.

12.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.18 % absolute for materials approaching 100 or 0 %.

12.1.2 *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 0.22 % absolute for materials approaching 100 or 0 %.

13 Keywords

13.1 anatase pigment analysis; rutile; titanium oxide analysis; X-ray diffraction.



Standard Specification for Synthetic Red Iron Oxide Pigment¹

This standard is issued under the fixed designation D 3721; the number immediately following the designation indicates the year of original adoption 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 specification covers synthetic red iron oxide for use in paints and coatings manufactured by any of the following:

- 1.1.1 Calcination of iron salts.
- 1.1.2 Precipitation from iron salts.
- 1.1.3 Calcination of synthetic iron oxide.
- 1.1.4 Product of organic reduction.

2. Referenced Documents

- 2.1 *ASTM Standards*:
 - D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
 - D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
 - D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
 - D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
 - D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 The pigment shall conform to the following requirements:

Fe ₂ O ₃ , min, %	93.0
Moisture and other volatile matter, max, %	1.0
Organic coloring matter	none
Total sulfates expressed as SO ₃ , max, %	2.0
Coarse particles (total residue retained on a 45- μ m (No. 325) sieve) max, %	1.0

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 D01.31 on Pigment Specifications.

Current edition approved March 25, 1983. Published July 1983. Originally published as D 3721 - 78. Last previous edition D 3721 - 78.

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

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

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Matter soluble in water, max, %
pH value, min

0.2
5.0

3.2 Inasmuch as iron oxide pigments are available in a range of colors, the mass color and, if desired by the purchaser, the character of the tint formed by mixture with a white pigment shall be within mutually agreed upon limits of a reference sample mutually agreed upon between the seller and the purchaser. This sample should be tested in accordance with Test Method D 387.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Total Iron Oxide, Sulfur, and Organic Coloring Matter*—Methods D 50.

5.1.2 *Moisture and Other Volatile Matter*—Test Methods D 280, Method A.

5.1.3 *Matter Soluble in Water*—Test Methods D 1208.

5.1.4 *Coarse Particles in Dry Pigments*—Test Methods D 185.

5.1.5 *pH Minimum*—Test Methods D 1208.

5.1.6 *Mass Color and Tinting Strength*—Test Method D 387.

6. Keywords

6.1 iron oxide; manganese; pigments

Standard Specification for Natural Red and Brown Iron Oxide Pigments¹

This standard is issued under the fixed designation D 3722; the number immediately following the designation indicates the year of original adoption 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.

Scope

1.1 This specification covers dry and wet ground naturally occurring iron oxide, dry and wet ground calcined naturally occurring iron oxide, and mixtures of these with synthetic iron oxides. These pigments are suitable for use in paints or coatings.

Referenced Documents

2.1 ASTM Standards:

- D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²
- D 1208 Test Methods for Common Properties of Certain Pigments²

3. Composition and Properties

3.1 The pigments shall conform to the following requirements:

Total iron as Fe ₂ O ₃ , min, %	70.0
Moisture and other volatile matter, max, %	1.0
Organic coloring matter	none
Total sulfates expressed as SO ₃	2.0
Matter soluble in water, max, %	2.0

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 D01.31 on Specifications for Pigments.

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² Annual Book of ASTM Standards, Vol 06.02.

³ Annual Book of ASTM, Vols 06.01 and 06.02.

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Matter soluble in water, max, % (primer pigments)	1.0
Coarse particles (total residue retained on a No. 325 (45-μm) sieve) max, %	2.0

3.2 Inasmuch as iron oxide pigments are available in a range of colors, the mass color and, if desired by the purchaser, the character of the tint and the tinting strength shall be within mutually agreed upon limits of a reference sample acceptable to both the seller and the purchaser when tested in accordance with Test Method D 387.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately, or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Total Iron Oxide, Sulfur, and Organic Coloring Matter*—Methods D 50.

5.1.2 *Moisture and Other Volatile Matter*—Test Method D 280, Method A.

5.1.3 *Matter Soluble in Water*—Test Method D 1208.

5.1.4 *Coarse Particles in Dry Pigments*—Test Method D 185.

6. Keywords

6.1 iron oxide; natural brown; pigments



Standard Specification for Synthetic Brown Iron Oxide Pigment¹

This standard is issued under the fixed designation D 3724; the number immediately following the designation indicates the year of original adoption 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—Paragraph 4.1 was editorially changed in May 1987.

1. Scope

1.1 This specification covers the pigments commercially known as synthetic pure brown iron oxides and blends. These pigments are suitable for use in paints and coatings. Methods of manufacture are:

1.1.1 Precipitation of iron salts.

1.1.2 Calcination of precipitated iron oxides.

1.1.3 Blends of synthetic red, yellow, and black iron oxides.

1.1.4 Blends of synthetic red, yellow, and black iron oxides plus the addition of carbon black to a maximum of 5.0 %. Small amounts of carbon black are added to obtain tinting colors not obtainable when synthetic brown iron oxide or carbon black are used singly.

2. Referenced Documents

2.1 ASTM Standards:

D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²

D 387 Test Method for Color and Strength of Color Pigments with a Mechanical Muller²

D 1208 Test Methods for Common Properties of Certain Pigments²

D 3872 Test Method for Ferrous Iron in Iron Oxides²

E 350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron⁴

E 351 Test Methods for Chemical Analysis of Cast Iron—All Types⁴

3. Composition and Properties

3.1 The pigment shall be a manufactured iron oxide or a

blend. It shall be a soft dry finely pulverized pigment and shall conform to the following requirements:

FeO	0 to 10 %
Fe ₂ O ₃	83 to 98 %
Carbon black, max, %	5.0 %
Moisture and other volatile matter, max, %	1.0 %
Organic coloring matter	none
Total sulfates expressed as SO ₃ , max, %	2.0 %
Coarse particles (total residue retained on No. 325 (45- μ m) mesh sieve, max, %	1.0 %
Matter soluble in water, max, %	0.5 %

3.2 Inasmuch as synthetic brown iron oxides are available in a wide range of shades. The mass color and character of the tint and the tinting strength formed by a mixture with a white pigment shall be within mutually agreed upon limits of a standard acceptable to both the purchaser and the seller when tested by Test Method D-387.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from different packages in the ratio of two samples for each 5 tons (inch-pound or SI), except that for shipments of less than 10 000 lb two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the appropriate ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.1.1 *Total Iron Oxide, Sulfur and Organic Coloring Matter*—Methods D 50.

5.1.2 *Ferrous Iron Content*—Test Method D 3872.

5.1.3 *Moisture and Other Volatile Matter*—Test Method D 280—Use Method A for pigments containing less than 8 % ferrous iron oxide. Use Method B for pigments containing more than 8 % ferrous iron oxide.

5.1.4 *Matter Soluble in Water*—Test Method D 1208.

5.1.5 *Coarse Particles in Dry Pigment*—Test Method D 185.

5.1.6 *Carbon Black Content*—Test Methods E 350 or E 351.

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 D01.31 on Pigment Specifications.

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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 03.05.

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Standard Test Method for Silicon Content of Silicone Polymers and Silicone-Modified Alkyds by Atomic Absorption¹

This standard is issued under the fixed designation D 3733; the number immediately following the designation indicates the year of original adoption 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 August 1984.

1. Scope

1.1 This test method covers the determination of the silicon content of silicone polymers and silicone modified alkyds when present in the nonvolatile portion of polymers, resins, or liquid coatings to the extent of 1 % or more.

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:

D 1153 Specification for Methyl Isobutyl Ketone²

D 1193 Specification for Reagent Water³

D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints⁴

D 2698 Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High Speed Centrifuging⁴

D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings⁴

3. Significance and Use

3.1 The silicon content of silicone modified alkyds has a direct relationship to the cost and performance characteristics, especially heat resistance of coatings prepared from them.

4. Summary of Method

4.1 The polymer solution or separated coating vehicle is diluted with methyl isobutyl ketone, and the silicon content is determined by atomic absorption spectroscopy.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of an atomizer and a single-slot burner; gas pressure-regulating and -metering devices for nitrous oxide (N_2O) and acetylene; a silicon hollow-cathode lamp with a regulated constant current

supply; a monochromator and associated optics; a photosensitive detector connected to an electronic amplifier; and a read-out device.

5.2 Centrifuge.

5.3 *Pipets*, 5, 10, 15, and 20-mL capacity.

5.4 *Volumetric Flasks*, 25, 50, 100, and 1000-mL.

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.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.

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 *Octaphenylcyclotetrasiloxane*.⁶

6.4 *Methyl Isobutyl Ketone* (MIBK), complying with Specification D 1153.

6.5 *Silicon, Standard Stock Solution* (100 $\mu\text{g/mL}$)—Weigh exactly 0.709 g of octaphenylcyclotetrasiloxane and quantitatively transfer to a 1-L flask. Dilute to volume with MIBK.

7. Calibration and Standardization

7.1 Prepare the following standard solutions from the 100 $\mu\text{g/mL}$ silicon standard stock solution, diluting to the indicated volumes with MIBK:

Stock Solution, mL	Dilute to, mL	Concentration, $\mu\text{g/mL}$
20	25	80
15	25	60
10	25	40
5	25	20
5	50	10
5	100	5

7.2 Operational instructions for atomic absorption spectrophotometers vary with different models. Consult the

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Material and is the direct responsibility of Subcommittee D 01.33 on Varnish and Resins, Including Shellac.

Current edition approved Oct. 27, 1978. Published December 1978.

² *Annual Book of ASTM Standards*, Vol 06.03.

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

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."

⁶ Available as Standard Reference Material No. 1066a from Office of Standard Reference Materials, Room B-314, Chemistry Building, National Bureau of Standards, Washington, DC 20234.

manufacturer's literature for establishing optimum conditions for the specific instrument used.

7.3 Turn the instrument on and set the wavelength to the 251.6-nm silicon line. Apply the recommended current to the silicon 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 in accordance with instructions.

NOTE 1—An acetylene pressure of 7 psi (48 kPa) and a nitrous oxide pressure of 30 psi (207 kPa) have been found to be satisfactory for most instruments.

7.4 While aspirating MIBK, carefully adjust fuel and oxidant flow rates as well as aspiration rate, until a clean, steady flame is obtained and no detectable carbonization occurs on the burner head. Aspirate the 5 µg/mL standard solution and make any necessary readjustments in instrument parameters to obtain maximum absorption. Record optimized fuel and oxidant flow rates for future reference and use for calibration and sample analyses.

7.5 Aspirate MIBK. When the recorder, meter, or other read-out device reaches a constant value, adjust it to zero immediately.

7.6 Aspirate each of the appropriate standard solutions in ascending silicon concentrations, ending with the 100 µg/mL standard. Record the corresponding instrument readings. Aspirate MIBK between each standard, and after the last standard.

7.7 Construct a calibration curve on linear graph paper by plotting the absorbance versus concentration (micrograms per millilitre) for each standard solution.

NOTE 2—Complete calibration and standardization (7.3 to 7.7) immediately prior to sample analysis.

8. Procedure

8.1 If the sample is a pigmented coating, remove all traces of pigment by centrifuging in accordance with Methods D 2372 or D 2698. Determine the nonvolatile content of the vehicle or polymer solution in accordance with Guide D 2832.

8.2 Prepare at least two replicate specimens by weighing by difference from a dropping bottle or syringe, a quantity of the vehicle or polymer solution equivalent to 60 to 240 mg (see Note 3) of solids directly into 100-mL volumetric flasks. Dilute to volume with MIBK and mix thoroughly.

NOTE 3—If the approximate silicon content is known, use the following specimen weights to optimize analytical results:

Expected Silicon Content, %	Optimum Weight of Solids, mg
8	70
4	150
2	300
1	600

8.3 Aspirate each specimen solution and determine the absorbance in the same manner in which the instrument was calibrated. Determine the concentration of silicon in micrograms per millilitre from the calibration curve. If the absorb-

ance is above the range covered by the calibration curve, dilute an aliquot of the sample solution to a suitable volume with MIBK. If the absorbance is below the range covered by the calibration curve, repeat the analysis using a larger specimen size.

NOTE 4—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.

9. Calculation

9.1 Calculate the mean concentration of silicon in the nonvolatile portion of the material under test as follows:

$$\text{Silicon, \% in nonvolatile} = \frac{C \times F}{NV \times S}$$

where:

C = concentration of silicon in the aspirated test solution, µg/mL.

F = dilution factor from 8.3 (volume diluted to volume of aliquot),

NV = percent nonvolatile of material under test, and

S = specimen weight, g.

9.2 To calculate the equivalent amount of silicon dioxide in the sample, multiply the percent silicon found by 2.139.

9.3 If the type of silicone polymer present in a varnish or coating vehicle is known, separate analysis of the silicone polymer allows the determination of a conversion factor that may then be used to calculate the percent of silicone polymer present in the vehicle.

10. Report

10.1 Report the percent of silicon, silicon dioxide equivalent, or silicone polymer present in the nonvolatile content of the material under test.

11. Precision⁷

11.1 In an interlaboratory study of the method, the within-laboratory coefficient of variation was found to be 4.11 % relative at 21 degrees of freedom and between laboratories coefficient of variation was 5.27 % relative at 6 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 12.1 % 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 18.2 % relative.

⁷ Supporting data are available on loan from ASTM Headquarters. Request RR: D 01 - 1009.

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Standard Test Method for Ferrous Iron in Iron Oxides^{1,2}

This standard is issued under the fixed designation D 3872; the number immediately following the designation indicates the year of original adoption 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 January 1991.

1. Scope

1.1 This test method covers the quantitative determination of ferrous oxide (FeO) by oxidation of ferrous iron (Fe⁺⁺) in an acid solution to the ferric state (Fe⁺⁺⁺) and titration with potassium dichromate using diphenylamine as the indicator.

1.2 This test method is applicable to synthetic black iron oxide, natural black iron oxide, magnetite or brown iron oxide where part of the iron content is present in the ferrous state (Note 1). It is applicable to iron oxides where the ferrous iron content ranges from 50 to 0.20 %.

NOTE 1—Natural iron oxides and magnetite may contain traces of metallic iron that will be combined with and analyzed as FeO.

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 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese³
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 769 Specification for Black Synthetic Iron Oxide³
- D 1193 Specification for Reagent Water⁴
- D 3722 Specification for Natural Red and Brown Iron Oxide Pigments³
- D 3724 Specification for Synthetic Brown Iron Oxide Pigment³

3. Significance and Use

3.1 This test method may be used for production quality control or specification acceptance.

4. Apparatus

4.1 The digestion apparatus consists of a standard 500-mL Erlenmeyer flask fitted with a two-hole rubber stopper and glass tubing, as shown in Fig. 1, to provide for the introduc-

tion of the inert gas and vent for gas and digestion fumes.

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.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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

5.3 *Diphenylamine Indicator*—Dissolve 1 g of diphenylamine in 100 mL of concentrated (H₂SO₄) (sp gr 1.84).

5.4 *Hydrochloric Acid (1+1)*—Dilute concentrated hydrochloric acid (HCl, sp gr 1.19) with an equal volume of reagent water.

5.5 *Iron Ore, Standard, Mesabi No. 27-D*.⁶

5.6 *Potassium Dichromate, Standard Solution (0.1 N)*—Dissolve 4.904 g of K₂Cr₂O₇ in water and dilute to 1 L. Standardize against National Bureau of Standards standard sample No. 27-D of Mesabi iron ore. Calculate the Fe factor in grams per millilitre for the solution as:

$$\text{Fe} = W/V_1 \quad (1)$$

where:

W = weight of Fe in the standard iron ore (not the solution, as there is none), and

V₁ = K₂Cr₂O₇ required for titration of standard, mL.

5.7 *Sulfuric-Phosphoric Acid Solution*—Prepare by carefully adding 600 mL of concentrated sulfuric acid (H₂SO₄) (sp gr 1.84) to 800 mL of water while stirring, then add 600 mL of phosphoric acid (85 to 87 %) mixing thoroughly. Cool and store.

6. Preparation of Sample (Note 2)

6.1 Mix the sample thoroughly and take a representative portion for the analysis. Exercise care at all times to prevent oxidation of the Fe⁺⁺ iron to the Fe⁺⁺⁺ state (Note 2). Grind

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.

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² This method is equivalent to Method 7161 of U.S. Federal Test Method Standard 141.

³ Annual Book of ASTM Standards, Vol 06.02.

⁴ Annual Book of ASTM Standards, Vols 06.03 and 11.01.

⁵ "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 May be secured from National Bureau of Standards, Department of Commerce, Washington, DC 20234. Other recognized primary iron standard may be substituted.

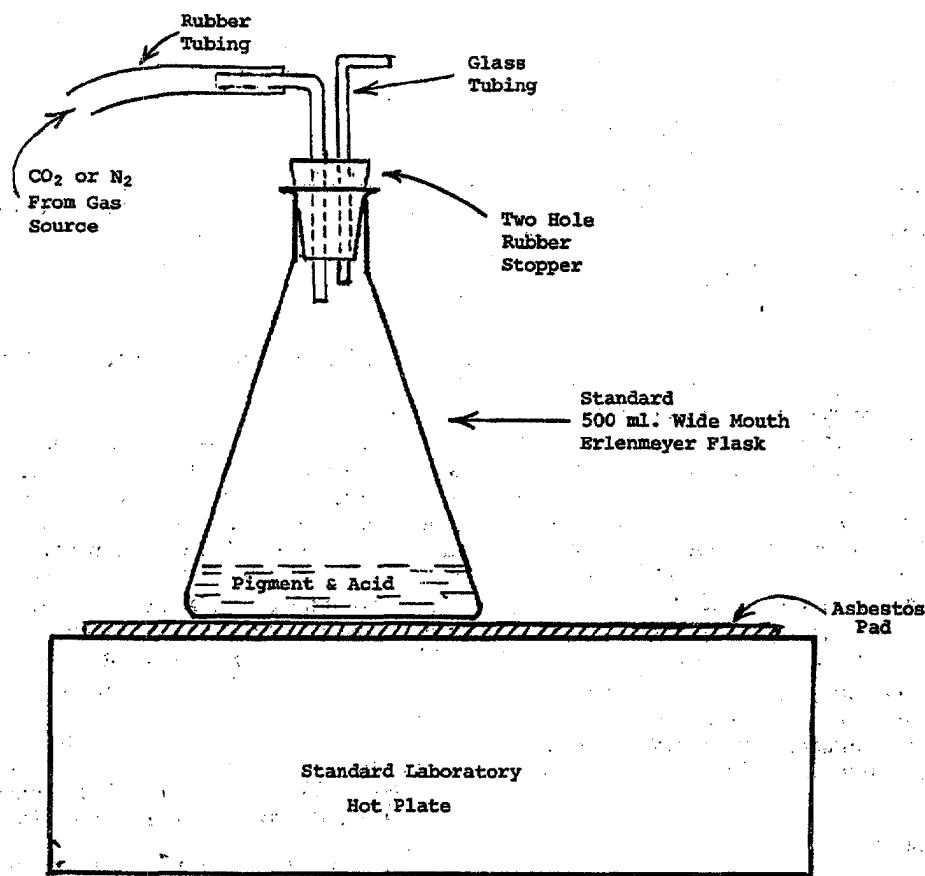


FIG. 1 Digestion Apparatus

natural iron oxides or natural magnetites to 100 % minus 100 mesh.

6.2 Finely divided synthetic black iron oxides if not carefully dried can oxidize so that part of the Fe^{++} iron is converted to Fe^{+++} . For this reason moisture should be determined on a separate specimen or by Test Methods D 280, Part B. Vacuum drying at temperatures not exceeding 65°C is recommended.

NOTE 2—Coarsely ground natural iron oxides and magnetite are very slow to dissolve. Exercise care in grinding oxides containing ferrous iron to avoid generating heat, as additional grinding could oxidize ferrous to ferric iron.

7. Procedure

7.1 Weigh 0.5 g of oxide to 1 mg and transfer to the 500-mL Erlenmeyer flask. Wash down the sides of the flask with 15 mL of water.

7.2 Attach the stopper with funnel and the gas delivery tube to the flask. Open the CO_2 valve until a gentle stream of gas flows through the flask. Nitrogen may be substituted.

7.3 Add 15 mL of 1+1 HCl (1+1). Digest on an asbestos pad on the hot plate at just below boiling temperature until completely dissolved or there is no further reaction (Note 3). Finely divided synthetic black or brown oxides usually require 5 to 10 min but natural oxides or magnetites may require considerably longer.

NOTE 3—Synthetic black iron oxides may contain small amounts of carbon black to increase tint strength. Carbon black will not dissolve during the digestion step and should not be confused with the soluble iron oxide.

7.4 Remove the flask from the hot plate and add 30 mL of sulfuric-phosphoric acid solution. Add 60 mL of water and cool in the water bath for 5 min.

7.5 Remove the CO_2 apparatus and add 5 drops of the diphenylamine indicator. Titrate with the standard potassium dichromate solution. The end point is sharp with a color change from green to purple. Avoid going past the end point.

8. Calculations

8.1 The percent FeO or Fe^{++} in the unknown sample are calculated by using Eq 2 or 3.

where:

W = weight of Fe in standard solution, g, and

P = potassium dichromate solution, mL.

$$\% \text{FeO} = \frac{V \times F \times 1.2865 \times 100}{S} \quad (2)$$

where:

F = Fe value, and

S = specimen weight, g.

$$\% \text{Fe}^{++} = \frac{V \times F \times 100}{S}$$

$$1.2865 = \frac{\text{molecular weight of FeO}}{\text{molecular weight of Fe}} = \frac{71.85}{55.847}$$

8.2 Report results to 0.1 %.

9. Precision and Bias

9.1 The precision statements are based on an interlaboratory study of the method in which operators in seven laboratories analyzed in duplicate on two days two commercial synthetic black iron oxides containing 20 and 23 % ferrous iron oxide. The within-laboratory standard deviation was found to be 0.235 for the first material and 0.05 for the second, each with 7df. The between-laboratory standard deviations were 0.061 and 0.058, respectively, each with 6 df.

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(3) Based on the pooled 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 0.5 % absolute at FeO contents of 20 to 25 %.

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 0.8 % absolute at FeO contents of 20 to 25 %.

9.2 *Bias*—Bias has not been determined.

10. Keywords

10.1 ferrous iron content; iron oxides, ferrous iron content; pigment, iron oxides



Standard Test Method for Methoxyl and Hydroxypropyl Substitution in Cellulose Ether Products by Gas Chromatography¹

This standard is issued under the fixed designation D 3876; the number immediately following the designation indicates the year of original adoption 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 October 1989.

1. Scope

1.1 This test method is applicable to the determination of methoxyl and hydroxypropyl substitution content in cellulose ether products by a Zeisel-gas chromatographic technique.

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 5 and Notes 2 and 3.*

2. Referenced Documents

2.1 ASTM Standards:

D 1347 Test Methods for Methylcellulose²

D 2363 Test Methods for Hydroxypropyl Methylcellulose²

3. Apparatus

3.1 *Gas Chromatograph*,³ with thermal conductivity detector and heated injection port.

3.2 *Electronic Integrator*,⁴

3.3 *Glass Tubing*,⁵ 6.4 mm in outside diameter, 4 mm in inside diameter, and 1829 mm long, packed with reagent (4.8). Two are required.

3.4 *Syringes*, 10 and 100 μ L.

3.5 *Reaction Vials, Caps, and Heating Block*.⁶

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 Chem-

ical 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.

4.2 *Adipic Acid*, M.P. 151 to 153°C.

4.3 *o-Xylene*, ACS.

4.4 *Toluene*, ACS.

4.5 *Iodomethane*, 99% min.

4.6 *2-Iodopropane*, 97% min.

4.7 *Hydriodic Acid* (sp. gr. 1.69 to 1.70), 57%.

4.8 *Acetone*.

4.9 *10 % Methyl Silicone Oil*,⁸ UCW 982 on 100/120 mesh solid support.

5. Hazards

5.1 Safety precautions must be taken for handling of hydriodic acid.

5.2 During the reaction, the glass vials are under pressure. Exercise caution in handling the hot vials.

6. Summary of Test Method⁹

6.1 When methyl cellulose or hydroxypropyl methyl cellulose is reacted with hydriodic acid in the presence of adipic acid, 1 mol of methyl iodide and 1 mol of isopropyl iodide are liberated for each mole of methoxyl and hydroxypropoxyl that is substituted on the cellulose chain. The methyl iodide and isopropyl iodide are extracted in situ with *o*-xylene and quantitated by gas chromatography using an internal standard technique.

7. Significance

7.1 This test method determines the methoxyl and hydroxypropoxyl content of cellulose ethers by a Zeisel-gas chromatographic technique.

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 D01.36 on Cellulosics.

Current edition approved Dec. 28, 1979. Published February 1980.

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

³ Hewlett-Packard Model 5700, available from Hewlett-Packard, Route 41, Starr Rd., P.O. Box 900, Avondale, PA 19311, has been found satisfactory for this purpose.

⁴ Hewlett-Packard Model 3380 has been found satisfactory for this purpose.

⁵ Tubing from Alltec Associates, P.O. Box 117, Rockford, IL 61105 has been found satisfactory for this purpose.

⁶ Reacti-therm Heating module, Reacti-Block Reacti-vials and Mininert valve tops from Pierce Chemical Co., Box 117, Rockford, IL 61105 have 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 Chromosorb WHP brand packing from Applied Science Laboratories, Box 440, State College, PA 16801, has been found satisfactory for this purpose.

⁹ Reference for methoxyl and hydroxypropoxyl content are in Dow Method No. MC-36 "Determination of Methoxyl and Hydroxyl-propoxyl Substitution in Cellulose Ether Products by a Zeisel Gas Chromatographic Technique."

Sampling

8.1 A specific sampling method is currently under study by the subcommittee.

Apparatus Preparation and Conditioning

9.1 *Column*¹⁰—Pack the two pieces of glass tubing (3.3) with a reagent (4.9) under vacuum and mechanical vibration using silanized glass wool to contain the packing. Install each in the gas chromatograph to facilitate on-column injection and allow it to come to equilibrium over a 12-h period under the following conditions:

Oven temperature	100°C isothermal
Injection port temperature	200°C
Detector temperature	200°C
Detector current	170 mA
Attenuation	1
Carrier gas	helium
Column A	20 mL/min
Column B	20 mL/min

NOTE 1—The conditions used here were determined to be optimum for the column used. Optimum conditions should be determined for each column on an individual basis.

9.2 Integrator:

9.2.1 Settings¹⁰

Attenuation	1
Start delay	0.5
Chart	auto
Chart speed	0.50 cm/in.
Area reject	off
Slope sensitivity	must be determined

9.2.2 Approximate component retention times

Minutes	Component
3.00	methyl iodide
5.00	isopropyl iodide
7.00	toluene (internal standard)
13.00	<i>o</i> -xylene

10. Preparation of Standard Solutions

10.1 *Internal Standard Solution (25 mg toluene/ml *o*-xylene)*:

10.1.1 Weigh a 100-mL volumetric flask containing 10 mL of *o*-xylene to the nearest 0.01 g.

10.1.2 Add 2.50 ± 0.01 g of toluene.

10.1.3 Dilute with *o*-xylene to 100 mL.

10.2 *Calibration Standard Solution*:

10.2.1 Weigh 130 to 140 ± 0.1 mg of adipic acid into a 3-dram vial.

10.2.2 Add 4.0 mL of 57 % hydriodic acid.

10.2.3 Pipet 4.0 mL of the internal standard solution into the vial and cap with a serum stopper or septum top.¹¹

10.2.4 Weigh vial and contents to nearest 0.1 mg.

10.2.5 Add 30 μ L of isopropyl iodide to the vial through the septum top with a syringe. Weigh and record the amount of isopropyl iodide added to nearest 0.1 mg.

10.2.6 Add 90 μ L of methyl iodide to the vial with a syringe. Weigh and record the amount added to nearest 0.1 mg.

10.2.7 Mix the contents well.

¹⁰ These settings were used with the Hewlett-Packard Model 3380 Integrator. Other units may require different settings.

¹¹ Mininert valve tops from Pierce Chemical Co., Box 117, Rockford, IL 61105 have been found satisfactory for this purpose.

10.2.8 Convert the alkyl iodides into their respective alkoxy equivalents using the following equations:

$$\text{mg methoxyl} = \text{g methyl iodide} \times \left(\frac{31 \times 1000}{142} \right) \quad (1)$$

$$\text{mg hydroxy propoxyl} = \text{g propyl iodide} \times \left(\frac{75 \times 1000}{170} \right) \quad (2)$$

$$\text{mg toluene} = \text{internal standard solution concentrated} \times 4 \text{ ml} \quad (3)$$

11. Calibration of Electronic Integrator¹²

11.1 Inject 2 μ L of the upper layer of the prepared standard solution (10.2) into the gas chromatograph and start the electronic integrator.

11.1.1 Calibrate in accordance with the manufacturer's instructions.

11.2 In the event an electronic integrator is not available the peak areas can be measured manually and a factor determined for each component can be obtained using the following equation:

$$F = \frac{A \times B}{C \times D} \quad (4)$$

where:

A = weight of the component in the standard solution, mg,

B = peak area of the internal standard solution, toluene from the standard run,

C = peak area of the component from the standard run,

D = weight of the internal standard solution, mg, and

F = component response factor.

12. Procedure

12.1 Sample Preparation:

12.1.1 Dry the sample at 105°C (221°F) for 30 min and store in a desiccator.

12.1.2 Weigh 60 to 70 ± 0.1 mg into a clean 5-mL reactor-vial.

12.1.3 Weigh into the reactor vial an amount of adipic acid equal to the specimen weight. (See Note 4).

12.1.4 Add 2.00 ± 0.01 mL of internal standard solution (10.1).

12.1.5 Add 2.00 ± 0.05 mL of 57 % hydriodic acid.

NOTE 2: Warning—Use a hood, goggles, and other appropriate safety equipment. Hydriodic acid can cause systemic damage.

12.1.6 Immediately cap tightly and weigh the vial. The cap should be tightened firmly to prevent leakage.

12.1.7 Shake the specimen for approximately 30 s.

12.1.8 Place the reactor-vial into a 150°C (302°F) heated block for 1 h, with removal for shaking after 20 min. Replace the specimen for the duration of the heating time.

NOTE 3: Warning—A possible safety hazard exists because the vials contain a hot corrosive acid under pressure.

12.1.9 After 1 h heating time, remove the specimen and place in the hood to cool for about 45 min. The specimen will separate into two layers.

12.1.10 Reweigh to determine any loss due to leakage. If

¹² The Hewlett-Packard 3380 has been found satisfactory for this purpose. Other electronic integrators may require a different calibration technique.

loss is greater than 0.0100 g the specimen should be discarded.

NOTE 4—The weight ratio of adipic acid to the specimen can range from 0.9 to 2.0. Excessive adipic acid may cause low methoxyl values.

13. Analysis

13.1 Enter into the integrator the milligrams of specimen and toluene internal standard used in the preparation of the specimen.

13.1.1 This can be calculated from the concentration of the internal standard solution.

13.2 Inject 2-μL of the *upper* layer of the specimen into the gas chromatograph and immediately start the integrator.

14. Calculation

14.1 The integrator printout records the methoxyl or hydroxypropoxyl substitution, or both, in weight percent.

14.2 If an electronic integrator is not available the peak areas can be measured manually and the alkoxy substitution may be calculated using the following equation:

$$\% = \frac{G \times F \times H \times 100}{I \times J} \quad (5)$$

where:

G = peak area of the component from the specimen run,

F = component response factor obtained in 11.2,

H = weight of the toluene internal standard in the specimen, mg,

I = peak area of the internal standard from the specimen run, and

J = specimen weight, mg.

15. Precision

15.1 The data using an electronic integrator show an average relative precision of 1.7% for methoxyl substitution (26% level) and 3.4% for hydroxypropyl substitution (0.3 to 10% level) at the 95% confidence limit 2σ .

15.2 *Interlaboratory Test Data.*¹³

¹³ Available from ASTM Headquarters. Request RR:D23-1000.

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Standard Test Method for Percent Solids in Titanium Dioxide Slurries¹

This standard is issued under the fixed designation D 3926; the number immediately following the designation indicates the year of original adoption 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 weight percent of solids in aqueous slurries of titanium dioxide pigments.

1.2 *This test 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:*
D1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 Slurry is weighed by difference into a tared aluminum foil dish, dried at 105°C in an oven for 1 h, cooled in a desiccator, and weighed.

4. Significance and Use

4.1 This test method is intended as a quick and reliable procedure for measuring the titanium dioxide pigment content of aqueous slurries. Included with the pigment content in the percent solids are the various nonvolatile additives used in preparing a stable slurry. Because the oxide modifiers on some titanium dioxide pigments may change somewhat with prolonged drying, in this method the solids of the slurry are considered dry after heating at 105°C for 60 to 65 min.

5. Apparatus

5.1 *Oven*—Laboratory oven capable of maintaining a temperature of 105 ± 2°C (Note 1). The oven may be a gravity-convection type or an oven with a low velocity, forced draft. An oven with a high-velocity, forced-draft air change, commonly used for baking finishes, is not suitable.

NOTE 1—The temperature in the oven must be constantly monitored. Many older ovens will no longer maintain ± 2°C; some will maintain this tolerance for a while but occasionally the thermostat will “stick” and the temperature will vary considerably.

5.2 *Balance*—Laboratory analytical balance, accurate to 0.1 mg, with 1-g optical readout range for fast weighing.

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.

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² Annual Book of ASTM Standards, Vols 06.03 and 11.01.

NOTE 2—Periodically check the accuracy of the 1-g optical scale of the balance by use of a known 1-g weight; adjust the balance if needed. The zero adjustment of the optical scale needs to be checked at least every hour routinely and immediately if there is any possibility of a spill having occurred on the balance.

5.3 *Desiccator*—Standard laboratory desiccator utilizing an indicating drying medium.

6. Reagents and Materials

6.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

6.2 *Aluminum Foil Dish*—Disposable aluminum foil dishes, approximately 60-mm diameter by 18 mm high, 1 to 2 g in weight.

6.3 *Disposable Syringe or Dropper*—Disposable 2 or 3-mL syringes or 2 to 5-mL droppers.

7. Procedure

7.1 Weigh two new, empty aluminum dishes each to 0.1 mg. For each dish, this is W_1 . With a syringe add 2 mL of water to each dish.

7.2 Shake or stir the slurry sample until it is homogeneous and free of any settled material. If the container is transparent or translucent, the absence of settled material sticking to the bottom of the container can be ascertained visually. Otherwise insert a spatula or the like to make sure there is no settled material.

7.3 Immediately withdraw 0.4 to 0.8 g of slurry in a new, empty disposable syringe or dropper. The specified amount can be estimated by a prior trial in another syringe or dropper. Wipe off the slurry from the outside of the syringe or dropper with a clean, absorbent paper. Cover the sample bottle. Weigh the syringe or dropper and slurry to 0.1 mg. This is W_2 .

7.4 Transfer the contents of the dropper into one of the weighed aluminum dishes. Add the slurry dropwise, gently shaking the dish to disperse the test specimen in the water. When no additional slurry can be transferred from the dropper, reweigh to 0.1 mg the dropper with any residual material inside. This is W_3 .

7.5 Shake the sample of slurry briefly and withdraw a second 0.4 to 0.8 g of slurry in another new dropper. Weigh and transfer contents as in 6.3 and 6.4 to the second weighed aluminum dish.

7.6 Place the two dishes and contents directly onto the metal shelf in the oven at 105 ± 2°C for 60 to 65 min. Do not dry in the oven longer than the specified 65 min.

7.7 Remove the dishes from the oven, cool in the desiccator for 10 to 60 min, remove one dish at a time, and weigh

immediately to the nearest 0.1 mg. This is W_4 for each dish. Do not cool longer than 60 min since there is a slight possibility of an equilibrium moisture exchange between the drying medium and some dried titanium dioxide pigments.

8. Calculation

8.1 For each of the duplicate measurements calculate the percent solids to two decimal places as follows:

$$\% \text{ Solids} = \frac{(W_4 - W_1)100}{W_2 - W_3}$$

where

W_1 = weight of empty aluminum dish, g,

W_2 = weight of dropper plus slurry, g,

W_3 = weight of dropper after discharging slurry into dish, and g,

W_4 = weight of dish and slurry after drying, g.

Example:

$W_1 = 1.4431$

$W_2 = 2.0894$

$W_3 = 1.4905$

$W_4 = 1.8158$

$$\begin{aligned} \% \text{ solids} &= \frac{(1.8158 - 1.4431)100}{2.0894 - 1.4905} \\ &= 62.23 \end{aligned}$$

8.2 Calculate the mean value of the duplicate measure-

ments to two decimal places.

9. Report

9.1 Round the calculated mean value to the nearest 0.1 % and report as percent solids. This rounded mean value is considered to be one result.

Example: solids = 62.2 %

10. Precision

10.1 On the basis of an interlaboratory test of this method in which 17 operators in 15 laboratories analyzed 8 materials with solids contents at different levels, the within-laboratory standard deviation was found to be 0.12 % and the between-laboratory standard deviation was found to be 0.23 %. 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 Two results, each the mean of duplicate determinations, obtained by the same operator at different times should be considered suspect if they differ by more than 0.34 % absolute.

10.1.2 Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.65 % absolute.

11. Keywords

11.1 slurries, nonvolatile content; solids in titanium pigment slurries; titanium dioxide slurries.

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Standard Test Method for Dichloromethane-Soluble Matter in Cellulose¹

This standard is issued under the fixed designation D 3971; the number immediately following the designation indicates the year of original adoption 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 dichloromethane-soluble matter in cellulose and is applicable to dissolving-type cellulose pulps prepared from cotton or wood.

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 1348 Test Methods for Moisture in Cellulose²

3. Summary of Test Method

3.1 A sample is extracted with dichloromethane in a Soxhlet apparatus as a measure of the waxes, fats, resins, and oils present.

4. Significance and Use

4.1 Dichloromethane-soluble materials are typically referred to as extractives. These extractives are comprised of organic materials that originated in the wood or cotton. The measure is an indication of the efficiency of removal of these substances during pulping and bleaching. The extractive level is of concern to dissolving pulp users since the presence of large amounts of extractives could inhibit the processing of cellulose into the desired derivative.

5. Apparatus

5.1 *Extraction Apparatus*³, Soxhlet, (Corning 3840 or 3880)—consisting of a 250-mL flask (Corning 4100), an extractor tube with standard taper 45/50 top joint (Corning 3740), and a Allihn standard taper 45/50 (Corning 3840), or Friedrichs standard taper 45/50 (Corning 3880) type condenser.

5.2 *Extraction Thimble*, either (1) standard thickness paper, 43 by 123 mm, (2) Alundum⁴, 34 by 100 mm,

medium porosity, or (3) glass with coarse-porosity fritted-glass disk, 45 by 130 mm.

5.3 *Heating Device*—Steam bath or heating mantle.

5.4 *Oven*, maintained at $105 \pm 3^\circ\text{C}$.

5.5 *Dish*, evaporating, disposable, light aluminum, 63 mm in diameter by 17.5 mm deep.

5.6 *Desiccator* with efficient desiccant.

6. Reagent

6.1 *Dichloromethane*, ACS, 99 % CH_2Cl_2 , having a residue after evaporation of less than 0.002 %.

7. Procedure

7.1 Weigh 8 to 12 g of loose pulp, to the nearest 0.01 g, into an extractor thimble that has previously been extracted with dichloromethane. For sheet pulp, cut a sample into strips about 10 mm wide and 70 mm long, and weigh about 20 g into a thimble. Weigh a separate portion for a moisture determination in accordance with Test Methods D 1348.

7.2 Place the extraction thimble with sample in the extractor and connect the flask. Pour 250 mL of the dichloromethane into the body of the extractor. Connect the assembled extractor to the condenser and place the flask in the heating device. Turn on the cooling water to the condenser and adjust the heating rate to cause siphoning 6 to 8 times per hour. Continue the extraction for 5 h.

7.3 Heat the evaporating dish in the oven at 105°C for 30 min, cool in a desiccator, and weigh to the nearest 0.1 mg.

7.4 When the extraction is complete, disconnect the flask at a time when most of the solvent has collected in the extractor. Partially evaporate the solvent in the extraction flask to a volume of 15 to 20 mL. Transfer the extract to the tared weighing dish by washing with three 5-mL portions of fresh solvent.

7.5 Place the dish on a steam hot plate and evaporate just to dryness. Then place the dish in the oven at 105°C for 1 h, cool in a desiccator, and weigh to the nearest 0.1 mg.

NOTE: Caution—Care must be taken to keep the steam hot plate surface clean in order to prevent contamination of the bottom of the aluminum dish and a subsequent unmeasured increase in tare weight.

7.6 Run a blank determination with the solvent used in the test. Evaporate 250 mL of the solvent to dryness and weigh the residue to the nearest 0.1 mg. Correct the weight of the dried extract by the weight of residue found.

8. Calculation

8.1 Calculate the percent of dichloromethane-soluble

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 D01.36 on Celluloses.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 3971 - 80. Last previous edition D 3971 - 80 (1985).

² Annual Book of ASTM Standards, Vol 06.02.

³ Available from the Corning Glass Works, Corning, NY 14832.

⁴ Alundum is a registered trademark of Norton Co., P.O. Box 350, Akron, OH 44309, and can be obtained from any laboratory supply house.

⁵ Fisher Scientific Catalog No. 08-732-5C available from the Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219 has been found satisfactory for this purpose.

matter, D , on the dry basis as follows:

$$D = (W_e - W_b) / W_s (100 - M) / 100 \times 100$$

where:

W_e = oven-dry weight of extract, g,

W_b = oven-dry weight of solvent blank determined in 7.6, g,

W_s = weight of sample, g, and

M = moisture in sample, % (Test Methods D 1348).

nearest 0.01 %.

9. Report

9.1 Report the dichloromethane-soluble matter to the

10. Precision and Bias

10.1 Results of a round robin study by five laboratories showed that for a content of 0.20 % extractives the relative standard deviation was 10 %. For samples at the <0.15 % level the relative standard deviation was <7 %.

10.2 No statement of bias can be made as no suitable reference material exists for determining bias.

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Standard Test Method for Metals in Cellulose by Atomic Absorption Spectrophotometry¹

This standard is issued under the fixed designation D 4085; the number immediately following the designation indicates the year of original adoption 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 iron, copper, manganese, and calcium content of cellulose pulp from wood or cotton.

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 1193 Specification for Reagent Water²

D 1348 Test Methods for Moisture in Cellulose³

D 3516 Practices for Ashing Cellulose³

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁴

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁵

3. Summary of Method

3.1 The sample is ashed in accordance with Practices D 3516.

3.2 This method is dependent on the fact that metallic elements in the ground state will absorb light of the same wavelength they emit when excited. When radiation from a given excited element is passed through a flame containing ground-state atoms of that element, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground-state element in the flame. A hollow cathode lamp whose cathode is made of the element to be determined provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the specimen into an oxidant-fuel flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation.

4. Significance and Use

4.1 Manganese in pulp acts as a catalyst in oxidizing cellulose.

4.2 Iron in pulp can cause yellowness in rayon fibers and influence cellulose acetate plastics color. Iron also causes problems in photographic and blueprint papers.

4.3 Copper in pulp can act as a retardant in oxidizing cellulose and can affect viscose ripening. Copper interferes with the dye level of rayon fibers and influences cellulose acetate plastics color.

4.4 Calcium in pulps can cause problems in processing into acetate, rayon, cellophane, etc. Calcium can create undesirable deposits in viscose spinning and film casting operation. Calcium can influence viscosity control during cellulose acetate manufacture.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of an atomizer and burner, suitable pressure-regulating devices capable of maintaining constant oxidant and fuel pressure for the duration of the test, a hollow cathode lamp for each metal to be tested, an optical system capable of isolating the desired line of radiation, an adjustable slit, a photomultiplier tube or other photosensitive device, and a read-out mechanism for indicating the amount of absorbed radiation.

5.2 *Oxidant*—Air, which has been cleaned and dried through a suitable filter to remove oil, water, and other foreign substances, is the usual oxidant.

5.3 *Fuel*—Acetylene, commercially available, is the usual fuel. Acetone, always present in acetylene cylinders, can be prevented from entering and damaging the burner head by replacing a cylinder that has a gage pressure of only 3.5 kPa (50 psi) remaining.

5.4 *Volumetric Flasks*, 25, 100, and 1000-mL.

NOTE 1—In listing the apparatus for this method, the items required for the ashing step are not listed. For these items refer to Practices D 3516.

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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water should be understood to mean reagent water conforming to the requirements in Specification D 1193, Type I.

6.3 *Hydrochloric Acid (1+1)*—Add 50 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 50 mL of water.

6.4 *Hydrochloric Acid (1+99)*—Dilute 10 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 1000 mL with water.

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 D01.36 on Cellulosics.

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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 14.02.

⁵ *Annual Book of ASTM Standards*, Vols 06.03 and 14.02.

6.5 *Nitric Acid* (1+1)—Add 50 mL of concentrated nitric acid (HNO₃, sp gr 1.42) to 50 mL of water.

6.6 *Sulfuric Acid* (1+1)—Add 50 mL of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) to 50 mL of water.

6.7 *Standard Solutions*—Dilute the solutions that follow with HCl (1+99) to prepare the standards to be used for calibration. Store all solutions in polyethylene bottles.

6.7.1 *Iron* (1 mL = 1.0 mg Fe)—Dissolve 1.000 g of pure iron in 100 mL of H₂SO₄ (1+1) with the aid of heat. Cool and dilute to 1 L.

6.7.2 *Copper* (1 mL = 1.0 mg Cu)—Dissolve 1.000 g of electrolytic copper contained in a 250-mL beaker in 30 mL of nitric acid (1+1). Slowly add 4 mL of H₂SO₄ (1+1) and heat until SO₃ fumes evolve. Cool and dilute to 1 L.

6.7.3 *Manganese* (1 mL = 1.0 mg Mn)—Dissolve 3.076 g of manganous sulfate monohydrate (MnSO₄·H₂O) in a mixture of 10 mL of HCl and 100 mL of water. Dilute to 1 L.

6.7.4 *Calcium* (1 mL = 1.0 mg Ca)—Weigh 2.497 g of calcium carbonate (CaCO₃) and transfer it to a 500-mL Erlenmeyer flask. Add 10 mL of water. Pour 10 mL of HCl slowly down the side of the flask. Add an additional 200 mL of water and heat until solution is complete. Cool and dilute to 1 L.

NOTE 2—Acceptable standard solutions are available commercially from any laboratory supply house.

6.8 *Lanthanum Solution* (50 g/L)—Weigh 58.65 g of lanthanum oxide (La₂O₃) with water. Add slowly 250 mL of HCl to the mixture. When dissolved dilute to 1 L with water.

7. Preparation of the Sample

7.1 Select a representative sample in the amount of 30 g, 5 g for the determination of moisture and 25 g for the determination of the metals.

8. Procedure

8.1 Weigh about 25 g of pulp to the nearest 0.01 g. At the same time weigh out a separate sample for oven-dry cellulose determination. Ash the sample by Method D in Recommended Practices D 3516. Determine moisture content in accordance with Method D 1348.

NOTE 3—Method D in Recommended Practices D 3516 has been listed as the preferred one because it minimizes opportunity for sample loss during ashing, especially for the iron determination. However for calcium, or manganese greater than 1 mg/kg, low results may occur from sulfate interferences and Method A in Recommended Practices D 3516 may be preferred.

8.2 Calibration and Standardization of Atomic Absorption Spectrophotometer.

8.2.1 The method of operation varies with different models of atomic absorption spectrophotometers. Therefore, no attempt is made here to describe in detail the steps for placing an instrument into operation.

8.2.2 Prepare working standard solutions daily from those described in 6.7. Make the final calcium dilutions to contain 1 % lanthanum.

8.2.3 Atomize the standards and calibrate the spectrophotometer for the element of interest.

8.3 Analyze the sample solutions prepared from 24.13 and 24.14 of Method D in Recommended Practices D 3516 in accordance with 8.2. Iron, copper, and manganese nor-

TABLE 1 Precision Data from Interlaboratory Testing Program Using Dry Ashing

Metal	Average, mg/kg	2S, mg/kg	2S	D2S, mg/kg	I(R), mg/kg
Iron	4.0	1.7	41	2.4	28
Copper	0.22	0.25	110	0.34	0.45
Manganese	2.0	0.7	34	1.0	1.3
Calcium	201	42	21	59	59

mally can be run without further dilutions. Dilutions for calcium should be made 1 % in lanthanum.

NOTE 4—If Method A in Recommended Practices D 3516 was used, digest the ash from 7.4 with 5 mL HCl (1+1) on a steam bath, cool and dilute in a 25-mL volumetric flask to volume with water. Further dilutions for calcium should be made 1 % in lanthanum.

8.3.1 A reagent blank should be used to zero the atomic absorption spectrophotometer before taking sample readings.

9. Calculations

9.1 Calculate the concentration of the metallic ion, in milligrams per litre, using the calibration determined in 8.2.3.

$$M = \frac{C \times V}{W}$$

where:

M = metal content, mg/kg,

C = amount of material determined in sample solution, mg/L,

V = final dilution volume of sample, mL, and

W = weight of oven-dry cellulose, g.

10. Precision and Bias

10.1 *Precision*—An interlaboratory test of this procedure was conducted using a single cellulose pulp. Four laboratories conducted tests using both dry ashing and wet ashing procedures; one additional participating laboratory used wet ashing only. The results of this study are presented in Table 1 (dry ashing) and Table 2 (wet ashing).

10.1.1 Data in the tables include the overall average value, two-sigma limits (2S), two-sigma limits in percent (2S %) and difference-two-sigma limits (D2S) as defined in Recommended Practice E 177. An estimate of the reproducibility interval, *I*(*R*) as defined in Practice E 691, is included. These statistics apply only to testing carried out in a manner similar to the study on which they are based and should be considered as guidelines rather than exact mathematical quantities.

10.1.2 The within-laboratory, single operator, same-day, single-machine precision of this method is represented by the two-sigma limits (2S) in the tables. The corresponding relative precision, two-sigma limits in percent (2S %), is also

TABLE 2 Precision Data from Interlaboratory Testing Program Using Wet Ashing

Metal	Average, mg/kg	2S, mg/kg	2S %	D2S, mg/kg	I(R), mg/kg
Iron	5.9	2.9	49	4.1	6.3
Copper	0.91	1.02	112	1.4	1.7
Manganese	1.9	0.7	24	1.0	1.6
Calcium	193	23	12	34	34

given. It is not likely that all sources of error in this procedure are directly proportional to the test level. Therefore, the indexes in percent may not apply at levels far removed from those in the tables.

10.1.3 In most experiences less than 5 % of all random pairs of measurements will not differ in absolute value by more than the difference two-sigma limits (D2S) shown in the tables. This is also equivalent to the repeatability interval, $I(r)$, as defined in Practice E 691.

10.1.4 On the basis of test error alone (including both within- and between-laboratory components) the difference,

in absolute value, of two test results obtained in different laboratories on the same material will be expected to exceed the reproducibility interval, $I(R)$, only about 5 % of the time. This index is only a guideline, as noted in Practice E 691, and may change at test levels appreciably different from those used in the interlaboratory test.

10.2 *Bias*—In the absence of a suitable reference material, no accuracy determination is possible. It should be noted, however, that dry ashing methods are subject to loss of some metals during ignition, yielding lower results than wet ashing procedures. This may have been the case for the iron and copper results in the interlaboratory test.

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Standard Guide for Determining Volatile and Nonvolatile Content of Pigments¹

This standard is issued under the fixed designation D 4139; the number immediately following the designation indicates the year of original adoption 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 July 1991.

1. Scope

1.1 This guide is intended to aid in the selection of the proper ASTM test method for determining the volatile and nonvolatile content of pigments.

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:

Standard	Section	ASTM Designation
Inert or low hiding pigments	4.1	D 280
White pigments	4.2	D 280
Black pigments	4.3	D 280 D 1509
Aluminum and zinc pigments	4.4	D 280 D 480
Blue pigments	4.5	D 280 D 1135
Green pigments	4.6	D 280
Yellow, orange, brown pigments	4.7	D 280 D 3724 D 763
Red pigments	4.8	D 280
Miscellaneous	4.9	

2. Referenced Documents

2.1 ASTM Standards:

- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²
- D 480 Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes²
- D 763 Specification for Raw and Burnt Umber Pigments²
- D 1135 Test Methods for Chemical Analysis of Blue Pigments²
- D 1509 Test Method for Carbon Black—Heating Loss
- D 3724 Specification for Synthetic Brown Iron Oxide Pigment²

3. Significance and Use

3.1 The nonvolatile content of raw materials may be used to determine the total nonvolatile content (solids) of paint and related coatings. Such information may be useful to coatings producers and users for the determination of the total solids available for film formation and for the estimation of the volatile organic content.

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 Paint Materials.

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² Annual Book of ASTM Standards, Vol 06.02.

4. Procedure

4.1 Inert or Low Hiding Pigments:

4.1.1 Test Methods D 280 contain Method A for pigments that do not decompose at 110°C and uses a time of 2 h at 105 to 110°C. Method B is for pigments that decompose at 110°C and use a vacuum to remove the volatile material.

4.1.1.1 Test Methods D 280 are applicable to anhydrous and hydrous aluminum silicate, calcium carbonate, magnesium silicate, pumice, and wet ground mica pigments for determination of hygroscopic moisture and other matter volatile under the test conditions.

4.2 *White Pigments*—Test Methods D 280 are applicable to titanium dioxide and zinc sulfide pigments.

4.3 Black Pigments:

4.3.1 Test Methods D 280 are applicable to synthetic black iron oxide pigment.

4.3.2 Test Method D 1509 is used to determine heating loss in carbon black pigment.

4.4 Aluminum and Zinc Pigments:

4.4.1 Test Methods D 480 cover the determination of nonvolatile matter in aluminum paste.

4.4.2 Test Methods D 280 are applicable to zinc dust (metallic zinc powder).

4.5 Blue Pigments:

4.5.1 Test Methods D 280 are applicable to copper phthalocyanine blue and ultramarine blue pigments.

4.5.2 Test Methods D 1135 cover the determination of moisture in iron blue pigments by the Brabender moisture tester and by toluene distillation.

4.6 *Green Pigments*—Test Methods D 280 are applicable to pure chrome green, chrome oxide green, and phthalocyanine green pigments.

4.7 Yellow, Orange, Brown Pigments:

4.7.1 Test Methods D 280 are applicable to zinc yellow (zinc chromate), chrome yellow, chrome orange, cuprous oxide, natural red and brown iron oxide, molybdate orange, ochre, raw and burnt sienna, and raw and burnt umber pigments.

4.7.2 Specification D 3724 specifies methods for the determination of moisture and other volatile matter in synthetic brown iron oxide pigment, requiring use of Test Methods D 280, Method A for pigments containing less than 8 % iron and Method B for pigments containing more than 8 % iron.

4.7.3 Specification D 763 specifies 105°C for determination of moisture and other volatile matter in raw and burnt umber. Additional loss at temperatures above 110°C is considered to be ignition loss.

4.8 *Red Pigments*—Test Methods D 280 are applicable to

natural red and brown iron oxide, synthetic red iron oxide, pure para red toner, red lead, and pure toluidine red toner pigments.

4.9 Miscellaneous—No recommended methods are available for pigments not specifically listed in 4.1 through 4.8. Procedures to be used for determination of volatile and nonvolatile content of pigments not specified in 4.1 through 4.8 should be agreed upon between the producer and user.

5. Precision

5.1 None of the references standards contain precision statements.

6. Keywords

6.1 nonvolatile content of pigments; volatile content of pigments

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Standard Guide for Testing Epoxy Resins¹

This standard is issued under the fixed designation D 4142; the number immediately following the designation indicates the year of original adoption 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 methods for testing epoxy resins as listed in Table 1. All of the methods were tested by interlaboratory participation in accordance with usual ASTM guidelines. Each method specifies a recommended amount of sample for starting a separate analysis, but several of the procedures can be conducted on the same starting material if so desired. For example, viscosity, color, and density could be run on the same specimen.

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 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)³
- D 1259 Test Methods for Nonvolatile Content of Resin Solutions⁴
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁵
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)⁶
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁶
- D 1639 Test Method for Acid Value of Organic Coating Materials⁵
- D 1652 Test Methods for Epoxy Content of Epoxy Resins⁴
- D 1726 Test Method for Hydrolyzable Chlorine Content of Liquid Epoxy Resins⁴
- D 1847 Test Methods for Total Chlorine Content of Epoxy Resins⁴

3. Significance and Use

- 3.1 This guide directs the user to test methods which

determine properties generally accepted as standard test items for classification of epoxy resins.

4. Epoxy Content

4.1 The epoxy content of epoxy resins is determined by reacting a solution of the resin with a standard solution of hydrogen bromide in glacial acetic acid. The quantity of acid consumed is a measure of the epoxy content. Test Methods D 1652 was found to have a repeatability of 2 % of the epoxy content and a reproducibility of 6 % of the epoxy content.

5. Hydrolyzable Chlorine

5.1 Test Method D 1726 covers the determination of the easily hydrolyzable chlorine content of liquid epoxy resins in concentrations below 1 weight %. The specimen is refluxed with a known amount of a standard alcoholic potassium hydroxide solution. The amount of hydroxide consumed is measured by titration and corresponds to the hydrolyzable chlorine content of the resin. By interlaboratory testing, the repeatability was found to be 0.02 %, and the reproducibility was found to be 0.05 %, both absolute.

6. Total Chlorine

6.1 Organic and inorganic chlorine compounds are determined in epoxy resins in accordance with Test Methods D 1847. The resin specimen is oxidized by combustion in a bomb containing oxygen under pressure. The chlorides formed are dissolved in a sodium carbonate solution and then either titrated or determined gravimetrically. The absolute repeatability and reproducibility for the titration method are 0.02 and 0.05 weight %, respectively. For the gravimetric method, the repeatability and reproducibility are both reported to be 0.05 weight %, absolute.

7. Viscosity

7.1 Kinematic viscosity is determined by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer in accordance with Test Method D 445. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid. The limits of precision at the three sigma level of confidence were found to be 0.35 % relative within one laboratory and 0.70 % relative between laboratories.

7.2 It is also possible to measure viscosity by the bubble time method described in Test Method D 1545. The rise time of a bubble in the resin contained in an ASTM viscosity tube is measured. The rise time in seconds is approximately equal to the viscosity in stokes. In the range from 4.5 to 440 St, duplicate runs by one operator should be considered suspect if they differ by more than 4.9 % relative. Two results, each the mean of duplicates obtained by different

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.

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² Annual Book of ASTM Standards, Vol 05.01.

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

⁴ Annual Book of ASTM Standards, Vol 06.02.

⁵ Annual Book of ASTM Standards, Vol 06.01.

⁶ Annual Book of ASTM Standards, Vols 06.01, 06.02, and 06.03.

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Coatings

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solids. Test Method D 1475 is a general method for determining the density of fluid formulated coatings and components thereof and is therefore equally applicable for determination of the density of lattices.

8. Viscosity

8.1 Lattices are non-Newtonian materials having rheological properties that can be characterized by apparent viscosity measurements made using rotational-type viscometers. Test Method D 2196 describes techniques using a Brookfield viscometer by which apparent viscosity can be measured and relative shear rate and time dependence of apparent viscosity can be assessed.

9. Consistency

9.1 Consistency of formulated fluid paints, lacquers, varnishes, and their individual fluid components, including high-viscosity lattices, are frequently measured using the Stormer viscometer. Test Method D 562 describes operation of the Stormer viscometer for measuring the consistency of such materials either with or without a stroboscopic timer.

10. Water Content

10.1 Although the major component of the volatile material in a latex is usually water, other volatile components may be present. Test Method D 3792 was developed for determining the water content of liquid latex coatings by direct

TABLE 1 Methods for Testing Latex Vehicles

Test Method	Section	ASTM Designation
Latex sampling methods	3	D 3925
Nonvolatile content	4	Method under development
Minimum film formation temperature (MFT)	5	D 2354
Qualitative polymeric analysis	6	D 3188
Density	7	D 1475
Viscosity	8	D 2196
Consistency	9	D 562
Water content	10	D 3792; D 4017
pH	11	E 70
Surface tension	12	D 1417

injection into a gas chromatograph. This method is equally suitable for determining water content of lattices. Test Method D 4017 is based on the Karl Fischer method and is also applicable for determining the water content of lattices.

11. pH

11.1 The pH of a latex can be measured by the general procedure described for aqueous solutions in Test Method E 70.

12. Surface Tension

12.1 The surface tension of latex may be determined by the technique given in Test Method D 1417. It is useful as a quality control/assurance test, and as an indicator of the wetting characteristics.

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Standard Practice for Determining Volatile and Nonvolatile Content of Cellulosics, Emulsions, Resin Solutions, Shellac, and Varnishes¹

This standard is issued under the fixed designation D 4209; the number immediately following the designation indicates the year of original adoption 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 is intended to serve as a guide to the selection of the proper ASTM test method for determining the volatile and nonvolatile content of cellulosics, emulsions, resin solutions, shellac, and varnishes. Unless otherwise specified, the test methods referenced appear in Volume 06.02 of the *Annual Book of ASTM Standards*.

NOTE—Standards for determining the composition of the volatile fraction are not covered by this practice.

1.2 The standards referenced in the practice are as follows:

Classification	Section	ASTM Standard
Cellulosics	5.1	D 871
		D 914
		D 1347
		D 2369
Emulsions	5.2	D 1259
Resin Solutions	5.3	D 1490
		D 29
Shellac	5.4	D 1650
Varnishes	5.5	D 115
		D 1644

2. Referenced Documents

2.1 ASTM Standards:

- D 16 Terminology Relating to Paint, Varnish, Lacquer, and Related Products²
- D 29 Test Methods for Sampling and Testing Lac Resins³
- D 115 Test Methods for Varnishes Used for Electrical Insulation⁴
- D 360 Specification for Shellac Varnishes³
- D 871 Methods of Testing Cellulose Acetate³
- D 914 Test Methods For Ethylcellulose³
- D 1259 Test Methods for Nonvolatile Content of Resin Solutions³
- D 1347 Test Methods for Methylcellulose³
- D 1490 Test Method for Nonvolatile Content of Urea-Formaldehyde Resin Solutions⁵
- D 1644 Test Methods for Nonvolatile Content of Varnishes⁶

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 Paint and Paint Materials.

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² *Annual Book of ASTM Standards*, Vols 06.01, 06.02, and 06.03.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ *Annual Book of ASTM Standards*, Vol 10.01.

⁵ *Annual Book of ASTM Standards*, Vol 15.06.

⁶ *Annual Book of ASTM Standards*, Vol 06.01.

D 1650 Test Methods for Sampling and Testing Shellac Varnish³.

D 2369 Test Method for Volatile Content of Coatings⁶

D 4758 Test Method for Nonvolatile Content of Latexes³

3. Terminology (see Terminology D 16)

3.1 Definitions:

3.1.1 *cellulose esters*—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced by acyl groups.

3.1.2 *cellulose ethers*—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced by alkyl groups.

3.1.3 *cellulose nitrates (nitrocellulose)*—derivatives of cellulose in which one or more of the hydroxyl hydrogens have been replaced by nitrate groups.

3.1.4 *emulsion vehicle*—an emulsion of binder in water. The binder may be oil, oleoresinous varnish, resin, or other emulsifiable liquid.

3.1.5 *latex*—a stable aqueous dispersion of synthetic resin, produced by emulsion polymerization, as the principal constituent of the binder.

3.1.6 *shellac varnish*—a solution or “cut” of a specified type and grade of dry lac resin in a suitable alcohol.

3.1.7 *varnish*—a liquid composition that is converted by oxidation or thermal cross-linking to a transparent or translucent solid film after application as a thin layer.

4. Significance and Use

4.1 The nonvolatile content of raw materials may be used to determine the total nonvolatile content (solids) of paint and related coatings. Such information may be useful to coatings producers and users for the determination of the total solids available for film formation and for the estimation of the volatile organic content.

5. Procedure

5.1 Cellulosics:

5.1.1 Test Methods D 1644, Method A, should be used for determining the amount of alcohol present in nitrocellulose shipments. This method may also be used for other cellulose derivatives if mutually agreed upon by producer and user.

5.1.2 Moisture in three different cellulose derivatives may be determined using test methods given in the following standards:

Cellulose Derivative	ASTM Standard	Section
Cellulose acetate	D 871	3-4
Ethylcellulose	D 914	4-6
Methylcellulose	D 1347	3-4

5.2 Emulsions and Latices:

5.2.1 Test Method D 2369, Procedure B may be used to determine the nonvolatile and volatile content of most emulsions. Important considerations are the presence of volatile plasticizers and surfactants and the presence of oxidizable components in oleoresinous vehicles and drying oil modified alkyds.

5.2.2 Test Method D 4758 should be used to determine the nonvolatile and volatile content of synthetic latex samples.

5.3 Resin Solutions:

5.3.1 Test Methods D 1259, Method A is recommended for solutions of non-heat reactive resins that remain stable and release solvents under the test conditions. Examples include alkyds and rosin esters.

5.3.2 Test Methods D 1259, Method B is recommended for solutions of heat reactive resins that undergo condensation or other reactions or both under the influence of heat. Examples include the formaldehyde reaction products of urea, melamine, and phenols. Method B is also recommended for resin solutions that release solvent slowly, for example, epoxy resin and vinyl resin solutions when these contain high boiling solvents.

5.3.3 Method A and Method B of Test Methods D 1259 differ primarily in the longer drying time specified for Method B (2 h versus 30 min). In both methods, a weighed specimen of resin solution is spread under pressure between two weighed sheets of aluminum foil, which are then separated and dried at 105°C. The method is unique in that it provides for drying of very thin films of resin, minimizing chances for volatiles to be trapped.

5.4 Shellac:

5.4.1 Sections 14 and 15 of Test Methods D 29 describe

two methods for the determination of volatile matter (moisture) in lac resins. Method A (Section 14) is recommended for orange shellac, button lac, garnet lac, and dry bleached lac. Method B (Section 15) is recommended for bleached lac in the form of hanks, bars, and crushed fresh ground.

5.4.2 Sections 14 to 16 of Methods D 1650 are recommended for the determination of the nonvolatile and volatile content of shellac varnishes.

5.5 Varnishes:

5.5.1 Test Methods D 1644 describes two methods for the determination of the nonvolatile and volatile content of varnishes. Method A requires heating a 1.2 g specimen at 105°C for 3 h in an oven, while Method B requires heating a smaller specimen at 149°C for 10 min on a hotplate. Method A may give high results due either to incomplete elimination of volatile matter or to absorption of oxygen by oxidizing-type varnishes.

5.5.2 Sections 18 to 22 of Methods D 115 is 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. 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 user.

6. Precision

6.1 Some of the referenced ASTM standards have precision limits. Reference to the individual standards for precision statements is recommended.

7. Keywords

7.1 nonvolatile content of cellulose, emulsions; volatile content of cellulose, emulsions

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Standard Guide for Testing Amino Resins¹

This standard is issued under the fixed designation D 4277; the number immediately following the designation indicates the year of original adoption 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 3 and 4 were renumbered in October 1988.

1. Scope

1.1 This guide covers test methods suitable for testing heat-reactive nitrogen resins, particularly urea-formaldehyde and melamine-formaldehyde resins. The test methods used are listed in Table 1.

2. Referenced Documents

2.1 ASTM Standards:

- D 883 Definitions of Terms Relating to Plastics²
- D 1013 Test Method for Determining Total Nitrogen in Resins and Plastics³
- D 1198 Test Method for Solvent Tolerance of Amine Resins³
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)⁴
- D 1259 Test Methods for Nonvolatile Content of Resin Solutions³
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁵
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁶
- D 1639 Test Method for Acid Value of Organic Coating Materials⁵
- D 3278 Test Methods for Flash Point of Liquids by Setaflash Closed-Cup Apparatus⁷

3. Terminology

3.1 **Definition**—The following definition is taken from Definitions D 883:

3.1.1 *amino resin*—a resin made by polycondensation of a compound containing amino groups such as urea or melamine with an aldehyde, such as formaldehyde, or an aldehyde-yielding material.

4. Significance and Use

4.1 This guide summarizes the currently available ASTM test methods useful for testing amino resins. These test methods may be used by producers and users to characterize

TABLE 1 Methods for Testing Nitrogen Resins

Test Method	Section	ASTM Designation
Total nitrogen in resins and plastics	5	D 1013
Solvent tolerance of amino resins	6	D 1198
Color of clear liquids	7	D 1209
Nonvolatile content resin solutions	8	D 1259
Density	9	D 1475
Viscosity	10	D 1545
Acid value	11	D 1639
Flash point	12	D 3278

a variety of properties of nitrogen resins related to their quality and safe use.

5. Total Nitrogen

5.1 Test Method D 1013 is a standard Kjeldahl procedure for nitrogen determination, directly applicable to amino resins and solutions thereof.

6. Solvent Tolerance

6.1 The amount of hydrocarbon that an amino resin tolerates is measured in accordance with Test Method D 1198. The end point of this titration, a defined degree of turbidity, is sensitive to both the alkylation and degree of polymerization of the amino resin.

7. Color

7.1 The color of solutions of amino resins can be measured using the platinum-cobalt scale according to the procedure in Test Method D 1209.

8. Nonvolatile Content

8.1 The nonvolatile content of amino resins is measured using Test Methods D 1259, Method B, which applies to heat reactive resin solutions and requires a longer drying time. A very thin film, created by pressing a resin specimen between sheets of aluminum foil, is dried for 2 h and weighed. There may be some reaction of amino resins during the drying procedure, but the repeatability (0.7 % absolute) and reproducibility (1.7 % absolute) are acceptable.

9. Density

9.1 Test Method D 1475 is a general-purpose test method for determination of density using a pycnometer or cup. It is fully applicable to amino resins.

10. Viscosity

10.1 Viscosity of amino resins is determined in bubble seconds, approximately equal to stokes, using standard

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.

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ *Annual Book of ASTM Standards*, Vols 06.01 and 06.03.

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

⁶ *Annual Book of ASTM Standards*, Vols 06.01, 06.02 and 06.03.

⁷ *Annual Book of ASTM Standards*, Vol 06.03.

viscosity tubes as described in Test Method D 1545.

11. Acid Value

11.1 Test Method D 1639, which describes titration of a resin specimen with standard potassium hydroxide, is suitable for determining the acid value of amino resins.

12. Flash Point

12.1 Test Methods D 3278 provide for either a flash/no flash or finite flash point determination using the Setaflash closed tester. It is applicable only when the material to be tested has a flash point between 0 and 110°C and a viscosity lower than 150 St at 25°C.

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Standard Specification for Calcium Borosilicate Pigments¹

This standard is issued under the fixed designation D 4288; the number immediately following the designation indicates the year of original adoption 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 specification covers three grades of pigment commercially known as calcium borosilicate composite pigment. The two types differ in chemical composition while the two classes of Type I differ in oil absorption.

2. Referenced Documents

2.1 ASTM Standards:

- C 958 Method for Determination of Particle Size Distribution of Alumina or Quartz by X-Ray Monitoring of Gravity Sedimentation²
- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints³
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out⁴
- D 1366 Practice for Reporting Particle Size Characteristics of Pigments⁴
- D 3360 Test Method for Particle Size Distribution by Hydrometer of the Common White Extender Pigments⁴
- D 4487 Test Methods for Analysis of Calcium Borosilicate⁴

3. Composition and Properties

3.1 All types of pigment shall consist of a complex composite of alkaline earth silicates and borates and shall conform to the requirements of Table 1.

3.2 The mass color of the dry pigment is white. The shade and tinting strength when specified shall be equal to that of a reference sample mutually agreed upon between the purchaser and the seller.

3.3 Particle Size:

3.3.1 All types are characterized by major amounts of particles in the 1 to 10- μ m range. Coarse particles retained on a 45- μ m (No. 325) sieve shall be less than 0.3 %. The maximum specific surface diameter (SSD) shall be 2.5.

3.3.2 Where closer control within a grade or type is required, the fineness requirements shall be as agreed upon between the purchaser and the seller.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Chemical Analysis*—Test Methods D 4487.

5.3 *Ignition Loss*—Test Methods D 4487.

5.4 *Particle Size*—To be determined by Test Method D 3360 or equivalent instrumental technique.⁵

5.5 *Specific Surface Diameter*—Particle Size by Sedimentation Methods section of Practice D 1366.

5.6 *Oil Absorption*—Test Method D 281.

5.7 *Coarse Particles*—The Insoluble Dry Pigments section of Test Methods D 185.

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 D01.31 on Pigment Specifications.

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² *Annual Book of ASTM Standards*, Vol 15.02.

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

⁴ *Annual Book of ASTM Standards*, Vol 06.02.

⁵ The Micromeritics Sedigraph 5000D Particle Size Analyzer, manufactured by Micromeritics Instrument Corp., 568 Goshen Springs Rd., Norcross, GA 30093, has been found satisfactory for this purpose. See Method C 958 for operating instructions. Other instruments having a similar slurry density measurement function may also be used.

TABLE 1 Requirements for Pigments

Compositional Requirements	Weight %			
	Type 1, 10% B ₂ O ₃		Type 2, 17% B ₂ O ₃	
	Min	Max	Min	Max
Calcium oxide (CaO)	41	46	41.5	45.5
Boron oxide (B ₂ O ₃)	9	11	15	17.5
Silica (SiO ₂)	38	42	32	38
Moisture and other volatile matter	...	0.75	...	0.75
Ignition loss at 450°C	2.5	6.0	2.5	6.0
Class A		Class B		
	min	max	min	max
Properties:				
Oil absorption (lb/100 lb pigment)	35	41	23	29
Coarse particles			37	47
Total residue retained on 45-μm (No. 325) sieve, %	0.3			0.3

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Standard Test Method for Total Chlorine in Epoxy Resins and Compounds¹

This standard is issued under the fixed designation D 4301; the number immediately following the designation indicates the year of original adoption 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 10 and Note 2 were added editorially in April 1990.

1. Scope

1.1 This test method describes a procedure for the determination of total chlorine in epoxy resins and glycidyl ethers.

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 Method

3.1 The material, dissolved in dimethoxyethane (DME) or other suitable inert solvent, is reacted with sodium biphenyl to convert bound organic chlorine to the water soluble chloride. The excess reagent is decomposed with isopropyl alcohol. The chloride ion is then titrated potentiometrically with silver nitrate.

4. Significance and Use

4.1 The presence of residual chlorine in epoxy resins is deleterious to final product properties. This test method has been found to be applicable to resins or ethers with chlorine contents ranging from 50 ppm to 35 % by weight. Other halogen compounds react with the reagent but are distinguished from chlorine by the final potentiometric titration. Epoxy and other functional groups will consume reagent but do not affect the results.

5. Apparatus

5.1 Potentiograph.

5.2 Beaker, of appropriate size.

5.3 Buret, of appropriate size.

5.4 Stirrer, magnetic or paddle.

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 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 Sodium Biphenyl Reagent⁴ (in ether solution).

NOTE 1—Store in a refrigerator when not in use to prevent loss of activity.

6.4 Nitric Acid (HNO_3) (1+1), aqueous solution.

6.5 1,2-Dimethoxyethane.

6.6 *Silver Nitrate* (AgNO_3), 0.1 and 0.01 *N* standardized solutions.

6.7 *Methyl Red Indicator* (0.2 % alcohol solution), dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

6.8 Isopropyl Alcohol.

7. Procedure

7.1 Pipet 5 mL of 1,2-dimethoxyethane into a clean, dry 250-mL beaker. Add a weighed amount of sample. (If the material is anticipated to be low in total chlorine, use 0.40 g of sample. For materials high in chlorine, weigh 0.1 to 0.2 g of sample.) Gently swirl to dissolve.

7.2 Add about 15 mL (one bottle) of sodium biphenyl reagent and mix thoroughly. If the solution loses its dark blue or green color within 5 min, add another 15 mL of reagent. Allow to react for 5 min.

7.3 Add 100 mL of isopropanol to decompose the excess reagent and to serve as the titrating medium. Add 3 to 5 drops of methyl red solution and neutralize with HNO_3 (1+1). Add 5 to 8 drops of acid in excess; start the stirrer.

7.4 Titrate the solution potentiometrically using standard AgNO_3 solution. (For low chlorine content, titrate with 0.01 *N* AgNO_3 and for high chlorine content use 0.10 *N* AgNO_3). The scale of the titration curve will depend on the instrument used but should give a curve of the approximate scale of 0.25 mL/cm and 30 mV/cm. The end point is selected as the middle of the steepest portion of the curve.

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 Jan. 5, 1984. Published July 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 This reagent is available in small bottles (15-mL) from Southwestern Analytical Chemicals, P. O. Box 485, Austin 63, TX. It may also be prepared as described in *Analytical Chemistry*, Vol 26, p. 748.

NOTE 2—It may be preferable to use the first or second derivative to determine the end point providing appropriate equipment is available.

7.5 Repeat the determination and also run a blank using all reagents but omitting the specimen.

Calculation

8.1 Calculate the weight percent of chlorine, C , as follows:

$$C = \frac{(V - B) \times N \times 3.546}{S}$$

where:

V = titration of specimen, mL,

B = titration of blank, mL,

N = normality of AgNO_3 solution, and

S = weight of specimen, g.

8.2 Calculate the mean of the two runs.

9. Precision

9.1 On the basis of an interlaboratory study of this test method in which one operator in each of three laboratories analyzed in duplicate on two different days five materials containing 0.1 to 0.3 % total chlorine; for a total of 60 determinations, the within-laboratory standard deviation was found to be 5 % relative and the between-laboratories standard deviation 7 % relative. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.2 *Repeatability*—Two results, each the mean of two runs, obtained by the same operator should be considered suspect if they differ by more than 15 % relative.

9.3 *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.

10. Keywords

10. chlorine; glycidyl ethers; liquid epoxy resins

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Standard Test Method for Lead and Chromium in Air Particulate Filter Samples of Lead Chromate Type Pigment Dusts by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 4358; the number immediately following the designation indicates the year of original adoption 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 14 was added editorially in May 1990.

1. Scope

1.1 This test method determines amounts of lead and chromium in residues obtained from air sampling of lead chromate and lead silico-chromate type pigment dusts. It is not applicable to all pigment dusts or to paint overspray samples of any kind.

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 Standard:

D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The filter containing the air particulate sample is dissolved with nitric acid followed by treatment with 50 % aqueous ammonium acetate solution. The solution is transferred to a volumetric flask and lead and chromium determined by atomic absorption.

4. Significance and Use

4.1 This test method covers only the analysis of the pigment dusts on filters, the results being expressed as micrograms of lead and of chromium. Sampling procedures are not a part of this test method, nor are those calculations that are required to convert the results to concentration of lead and chromium in the sampled air, as required by the U.S. Occupational Safety and Health Act (OSHA).

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of an atomizer and burner; gas pressure regulating and metering devices for air, acetylene, and nitrous oxide; provision for hollow cathode source lamps with regulated constant current supply; a monochromator with associated optics; a photodetector; an amplifier; and digital or analog readout system.

5.2 *Lead Hollow Cathode Lamp* or electrodeless discharge source lamp.

5.3 *Chromium Hollow Cathode Source Lamp*.

5.4 *Volumetric Flasks*, 10-mL.

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 shall conform to 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.

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 *Ammonium Acetate Solution* (50 % W/V aqueous)—Dissolve 250 g of ammonium acetate ($\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$) in water and dilute to 500 mL.

6.4 *Nitric Acid* (sp gr 1.50)—Concentrated nitric acid (HNO_3).

6.5 *Nitric Acid*, (1+3)—Dilute 1 part concentrated nitric acid with 3 parts water.

6.6 *Lead, Standard Stock Solution* (1000 $\mu\text{g/mL}$)—Dissolve 0.1599 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 50 mL of water containing 20 mL of concentrated HNO_3 and 10 mL of 50 % ammonium acetate solution, and dilute to 100 mL with water.

6.7 *Chromium, Standard Stock Solution* (1000 $\mu\text{g/mL}$)—Dissolve 0.3734 g of potassium chromate (K_2CrO_4) in 50 mL water containing 5 mL of concentrated HNO_3 and dilute to 100 mL with water.

6.8 *Filter Blank*—Millipore 0.8- μm pore size, 37-mm diameter, mixed esters of cellulose, Type AA white, plain, or equivalent.

6.9 *Hydrogen Peroxide*—30 % (H_2O_2).

7. Calibration and Standardization

7.1 Good atomic absorption practice requires that the 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 D01.21 on Chemical Analysis of Paints and Paint Materials.

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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."

solutions being analyzed be aspirated into the flame interspersed between or at least contiguous to aspiration of the calibration standards. The formal separation of calibration and analysis is for clarity of the methodology only and such separation should not be made in practice.

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 Insert the lead source lamp into the appropriate holder. Turn on the instrument and apply the current recommended by the manufacturer to the source lamp. Allow the lead source lamp a 10-min warmup. Adjust the wavelength to 283.3 nm, set the slit to 0.7-nm bandpass, and maximize the transmitted energy by fine adjustment of the wavelength.

7.4 Prepare fresh standard solutions each day containing 0.5, 1.0, 2.0, 5.0, and 10.0 ppm lead by appropriate dilutions of the 1000 µg/mL stock standard. 20 mL of concentrated HNO₃ and 2 mL of 50 % ammonium acetate should be added in each case per 100 mL of final dilution.

7.5 Adjust the air and acetylene pressure or flow rates and ignite the burner according to the instruction manual. Adjust the acetylene to give a rich flame. Allow a few minutes to equilibrate.

7.6 Aspirate water to rinse the atomizer chamber. Aspirate the 10 ppm standard and make any necessary readjustment in instrument parameters to obtain maximum absorption.

7.7 Aspirate each standard solution and record the corresponding instrument readings. Aspirate 1+3 HNO₃, then water between each standard.

7.8 Construct a calibration curve for lead on linear graph paper by plotting the absorbance versus concentration (micrograms per millilitre) for each standard solution.

7.9 Replace the lead source lamp with the chromium source lamp, adjust the applied current to that recommended and allow to warm up for 10 min. Adjust the wavelength to 357.9 nm, set the slit to 0.7-nm bandpass, and maximize transmitted energy by fine wavelength adjustment.

7.10 Prepare fresh standard solutions each day containing 0.1, 0.2, 0.5, 1.0 and 2.0 ppm chromium by appropriate dilutions of the 1000 µg/mL stock standard. Add 20 mL of concentrated HNO₃ and 2 mL of 50 % ammonium acetate in each case per 100 mL of final dilution.

7.11 Turn on air and acetylene, adjust the flow rates and ignite according to the instruction manual. Adjust the acetylene to give a rich flame. Allow a few minutes to equilibrate. (Note 1).

NOTE 1—The National Institute for Occupational Safety and Health (NIOSH) recommends the use of a nitrous oxide-acetylene flame, which reduces iron and nickel interference in the chromium determination. However, chromium sensitivity in the nitrous oxide flame is also much lower, so that air/acetylene is specified here. Iron, aluminum, titanium as well as SO₂ interference may be reduced by adding 10 % of W/V KHSO₄ to both standard and sample solution, employing an air/acetylene flame. The comparison of the experimental lead to chromium ratio noted can be of some use here. (See Section 11.)

7.12 Aspirate water into the flame. Then aspirate the 2.0 ppm standard and adjust burner position and flame composition to give maximum absorption.

7.13 Aspirate all standards into the flame, aspirating water between each standard and the next. Construct a calibration curve for chromium on linear graph paper by plotting the

absorbance versus concentration (micrograms per millilitre) for each standard solution.

8. Procedure

8.1 With each batch of samples a filter blank (minimum of 1 filter blank for every 10 filter samples) from the same membrane lot must be analyzed. The filter blank is carried through all steps of the analysis along with the sample(s).

8.2 Carefully open the filter cassette containing the air particulate sample. Remove the filter using forceps and deposit it in a 50-mL beaker.

8.3 Add 6 mL of 1+3 HNO₃, cover the beaker with a watch glass, and keep just under boiling by placing on a hot water bath. Heat for 10 min. Add 2 mL of 50 % ammonium acetate solution and continue to heat, adding water as needed to keep the volume at about 5 to 6 mL. The solution *must not* be allowed to go to dryness. When analyzing lead silicochromate-type pigment dusts, add 0.5 mL of 30 % hydrogen peroxide (H₂O₂) after the 2 mL of 50 % ammonium acetate solution, and continue heating until cellulose filter completely dissolves.

8.4 Continue heating for 1 h. If the cellulose filter is not dissolved, continue the digestion until the solution is clear. Cool and transfer to a 10.0-mL volumetric flask. Dilute to volume with water and determine lead and chromium.

8.5 Prepare the atomic absorption instrument for lead as noted in 7.3, 7.5, and 7.6.

8.6 Aspirate the test solution into the flame. Record the reading. Aspirate 1+3 HNO₃, then water between test solutions.

8.7 Aspirate the appropriate standards, usually one above and below the test solution concentration. Aspirate 1+3 HNO₃, then water between each standard and the next. Record readings.

8.8 If the absorption of any test solution is greater than that of the 10 ppm lead standard, make an appropriate dilution (matching the standard matrix), aspirate and measure the absorbance of the diluted solution. It is important that the diluted test solution be aspirated with the flame not being shut off or altered following aspiration of the standards.

8.9 Prepare the atomic absorption instrument for chromium as noted in 7.9, 7.11, and 7.12.

8.10 Repeat steps 8.6 through 8.8, in this case for chromium except that, between each standard or sample and the next, aspirate only water.

8.11 From the calibration curves determine the lead and chromium concentrations in the aspirated solutions.

9. Calculation

9.1 Calculate the micrograms of lead, *A*, on the filter as follows:

$$A = (L)(V_1)(F) \quad (1)$$

where:

L = lead read from calibration curve (8.11), µg/mL,
*V*₁ = final volume of lead solution which is measured (8.8 or if no dilution is necessary, 8.4), and
F = [volume to which originally diluted (8.4) divided by millilitres of aliquot of original dilution taken for final dilution].

9.2 Calculate the micrograms of chromium, B , on the filter as follows:

$$B = (C)(V_2)(F) \quad (2)$$

where:

C = chromium read from calibration curve (8.11), $\mu\text{g/mL}$,
 V_2 = final volume of chromium solution which is measured (8.10 or if no dilution is necessary, 8.4), and
 F = [volume to which originally diluted (8.4) divided by millilitres of aliquot of original dilution taken for final dilution].

10. Lead to Chromium Ratio

10.1 If the workplace dust involves a single, known lead chromate or lead silicochromate type pigment, the determined lead to chromium ratio found (9.1 and 9.2) may be compared with the theoretical ratio (or actual ratio) of the bulk pigment as an internal check.

11. Report

11.1 Report the micrograms of lead and the micrograms of chromium present on the filter.

12. Precision for Lead Chromate Type Pigment Dust

12.1 *Lead Determination*—On the basis of an interlaboratory study of the test method in which operators in eight laboratories analyzed sixteen air particulate samples containing from 16 to 80 μg of lead and from 3 to 15 μg of chromium, the within-laboratory coefficient of variation of the lead determination was found to be 9 % relative at 21 df and the between-laboratory coefficient of variation was 15 % 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.

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 26 % relative for lead.

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 40.4 % relative for lead.

12.2 *Chromium Determination*—In the same interlaboratory study of the test method, the within-laboratory coefficient of variation of the chromium determination was found to be 11 % relative at 20 df and the between-laboratory coefficient of variation was 19 % 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.

12.2.1 *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 33 % relative for chromium.

12.2.2 *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 54 % relative for chromium.

13. Precision for Lead Silicochromate Type Pigment Dust

13.1 *Lead Determination*—On the basis of an interlaboratory study of the test method in which operators in five laboratories analyzed sixteen air particulate samples containing from 37 to 116 μg of lead and 2 to 8 μg of chromium, the within-laboratory coefficient of variation of the lead determination was found to be 2.7 % relative at 15 df and the between-laboratory coefficient of variation was 9.4 % relative at 12 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 8.2 % relative for lead.

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 28.4 % relative for lead.

13.2 *Chromium Determination*—In the same interlaboratory study of the test method, the within-laboratory coefficient of variation of the chromium determination was found to be 2.3 % relative at 12 df and the between-laboratory coefficient of variation was 23.6 % relative at 9 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

13.2.1 *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 6.8 % relative for chromium.

13.2.2 *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 68.8 % relative (Note 2) for chromium.

NOTE 2—Error appears large due to the low range of chromium studied (2 to 8 μm per filter) leading to poor reproducibility.

14. Keywords

14.1 air particulates; air sampling; atomic absorption spectroscopy; chromium; lead; pigment dust

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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 Poly(Vinyl Chloride) Resins¹

This standard is issued under the fixed designation D 4368; the number immediately following the designation indicates the 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 covers test methods for poly(vinyl chloride) resins and vinyl chloride copolymers for use in coatings applications. The test methods listed in Table 1 were tested by interlaboratory participation in accordance with ASTM guidelines. Also included are methods useful to test the suitability of resins for use in fluid nonaqueous vinyl dispersions.

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 1243 Test Method for Dilute Solution Viscosity of Vinyl Chloride Polymers²
- D 1705 Test Method for Particle Size Analysis of Powdered Polymers and Copolymers of Vinyl Chloride³
- D 1755 Specification for Poly(Vinyl Chloride) Resins³
- D 1823 Test Method for Apparent Viscosity of Plastisols and Organosols at High Shear Rates by Extrusion Viscometer³
- D 1824 Test Method for Apparent Viscosity of Plastisols and Organosols at Low Shear Rates by Brookfield Viscometer³
- D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials³
- D 1921 Test Method for Particle Size (Sieve Analysis) of Plastic Materials³
- D 2196 Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer⁴
- D 2222 Test Method for Methanol Extract of Vinyl Chloride Resins³
- D 3030 Test Method for Volatile Matter (Including Water) of Vinyl Chloride Resins³
- D 3205 Test Method for Viscosity of Asphalt with Cone and Plate Viscometer⁵

D 3680 Test Method for Residual Vinyl Chloride Monomer Content of Poly(Vinyl Chloride) Resins, Compounds, and Copolymers by Solution Injection Technique⁶

D 3749 Test Method for Residual Vinyl Chloride Monomer in Poly(Vinyl Chloride) Homopolymer Resins by Gas Chromatographic Head Space Technique⁷

D 4287 Test Method for High-Shear Viscosity Using the ICI Cone/Plate Viscometer⁴

E 442 Test Method for Chlorine, Bromine, or Iodine in Organic Compounds by Oxygen Flask Combustion⁸

3. Significance and Use

3.1 The test methods listed and discussed in this guide are used to determine the purity and physical properties of poly(vinyl chloride) and vinyl chloride copolymers. This information is useful to polymer producers and coatings manufacturers.

4. Total Chlorine Content

4.1 The determination of the total chlorine in vinyl chloride homopolymers and copolymers is useful for the compositional analysis of the polymer and also to determine the amount of vinyl resin present in a coating binder, provided no other chlorine-containing ingredients are present.

4.2 Test Method E 442 involves the determination of chlorine in organic compounds by the oxygen flash technique (Schoeninger technique).

5. Dilute Solution Viscosity

5.1 The dilute solution viscosity provides a measure of the relative molecular weight of the vinyl resin. In Test Method D 1243, 0.2 g of the sample in 100 mL of cyclohexanone is employed. The flow time of the solution in a standard Ubbelohde viscometer at $30 \pm 0.5^\circ\text{C}$ is used to calculate the inherent viscosity (logarithm viscosity number).

5.2 The procedure is applicable to all vinyl chloride homopolymers and copolymers used in coatings.

6. Viscosity Measurement (of Vinyl Dispersions)

6.1 Viscosity behavior is important to the use of fine powder vinyl resins (dispersion resins) in fluid nonaqueous dispersions for coatings applications. These dispersions are called plastisols when only plasticizer(s) is(are) employed with the resin, and organosols when volatile diluents or solvents are also present.

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.

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² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 04.03.

⁶ Annual Book of ASTM Standards, Vol 06.02.

⁷ Annual Book of ASTM Standards, Vol 08.03.

⁸ Annual Book of ASTM Standards, Vol 15.05.

TABLE 1 Test Methods for Vinyl Chloride Resins

Method	Section	ASTM Designation
Viscosity, dilute solution	5	D 1243
Particle size (sieve analysis)	11	D 1705
Poly(vinyl chloride) resins, specification	6	D 1755
Viscosity, vinyl dispersions, high shear	6	D 1823
Viscosity vinyl dispersions, low shear	6	D 1824
Apparent density	10	D 1895
Particle size (sieve analysis)	11	D 1921
Methanol extract	7	D 2222
Volatile content	8	D 3030
Residual VC1 monomer-solution technique	9	D 3680
Residual VC1 monomer-head space technique	9	D 3749
Viscosity, high shear, cone and plate	6	D 4287
Total chlorine content	4	E 442

6.2 The flow behavior of the dispersions at low shear rates, which determines the relative ease of mixing and pumping (transfer) and is also related to low shear coating applications for example and dip coating, is measured in Test Method D 1824 or Test Method D 2196 with a Brookfield viscometer. The flow at high shear rates, related to high shear application such as in coil coating, is characterized in Test Method D 1823 using an extrusion rheometer. High shear flow can also be determined using a cone-and-plate viscometer, following the general procedure of Test Method D 3205 or with the equivalent Test Method D 4287.

6.3 The preparation and testing of the resin in a standard vinyl plastisol, which contains 60 parts of di(2-ethylhexyl phthalate) and 100 parts of the test vinyl dispersion resin, are described in Specification D 1755.

7. Methanol Extract

7.1 The amount of nonvolatile, methanol-soluble material present in the vinyl resin, such as surface active agents, plasticizers, and other alcohol-soluble additives, is obtained in Test Method D 2222 by weighing the material soluble in refluxing methanol.

7.2 This procedure provides a relative measure of the polymeric purity of the resin.

7.3 With some low molecular weight vinyl copolymer

resins with moderate comonomer content, the methanol extraction may also remove the low molecular fraction of the polymer.

8. Volatile Content

8.1 The volatile material present in the vinyl resin is determined from the loss in weight observed on heating a resin specimen to constant weight in an air-circulating oven at 110°C (see Test Method D 3030).

9. Residual Vinyl Chloride Monomer

9.1 Residual vinyl chloride monomer present in either vinyl chloride homopolymers or copolymers is measured in Test Method D 3680 using analysis of a solution of the test resin in tetrahydrofuran injected into a gas chromatograph.

9.2 Gas chromatography using a head space sampling technique is also applicable to poly(vinyl chloride) (Test Method D 3749).

10. Apparent Density

10.1 The apparent or bulk density of the solid resin is the weight of the powder per unit volume; that is, grams per cubic centimetre (or pounds per cubic foot). This parameter is a measure of the compactness of the powder. A cylindrical cup of 100 ± 0.5 mL is used in measuring this characteristic in Test Methods D 1895; a small quantity of powdered carbon black is used to dissipate the electrostatic charge present occasionally on vinyl resin powders or granules.

11. Particle Size

11.1 The median particle size and particle size distribution of the resin sample are determined using sieve analysis (Test Method D 1921). A small amount of carbon black is used to dissipate the static charge generated in the procedure. The lower size limit of the determination is about 38 μ m; for smaller particle sized resins, sedimentation methods are recommended.

11.2 Test Method D 1705 describes a wet sieve method that can also be used to determine the particle size and distribution.

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Standard Test Method for Analysis of Zinc Hydroxy Phosphite Pigment¹

This standard is issued under the fixed designation D 4450; the number immediately following the designation indicates the year of original adoption 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 15 was added editorially in May 1990.

1. Scope

1.1 This test method is intended for the determination of zinc oxide and phosphorous acid content of the pigment commercially known as zinc hydroxy phosphite. The zinc content is determined by EDTA titration and calculated as zinc oxide (ZnO), while the phosphorus content is determined colorimetrically and calculated as phosphorous acid (H₃PO₃).

1.2 The analytical procedures appear in the following order:

	Sections
Zinc oxide	6 through 8
Phosphorous acid	9 through 14

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:*
D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 The zinc is determined by dissolving the test sample in nitric acid, adjusting the pH of the solution to 5 to 5.5 and titrating with EDTA.

3.2 The phosphorus is determined colorimetrically with the aid of nitric acid, ammonium vanadate, and ammonium molybdate. The absorbance of the test sample is compared to a calibration curve that yields the number of milligrams of phosphorus.

4. Significance and Use

4.1 This test method may be used to confirm the stated zinc oxide and phosphorous acid content of zinc hydroxy phosphite pigment.

5. Purity of Reagents

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

TOTAL ZINC AS ZINC OXIDE

6. Reagents

6.1 *Ammonium Hydroxide* (sp gr 0.90), concentrated ammonium hydroxide (NH₄OH).

6.2 *Glacial Acetic Acid* (min 99.7 %), concentrated glacial acetic acid (CH₃COOH).

6.3 *Acid Ammonium Acetate Buffer*—Mix 400 mL of distilled water and 400 mL of reagent grade ammonium hydroxide (sp gr 0.90). Add 375 mL of reagent grade glacial acetic acid slowly while stirring.

6.4 *Cupric Sulfate Solution* (0.1 M)—Dissolve 25 g of CuSO₄·5H₂O in distilled water and dilute to 1 L.

6.5 *Disodium Ethylenediaminetetraacetate Dihydrate* (0.05 M) (EDTA Solution)—Dissolve 18.6 g of the reagent grade salt in distilled water and dilute to 1 L. Standardize the solution as follows:

6.5.1 Transfer 25 mL of lead standard (6.6) to a 400-mL beaker. Add concentrated ammonium hydroxide (6.1) dropwise until a permanent precipitate just forms. Add 25 mL of acid ammonium acetate (6.3), dilute to 200 mL, heat to boiling, add 4 drops of copper EDTA (6.7) and 12 drops of PAN (6.8), and titrate with EDTA to a clear yellow.

6.5.2 Calculate the molarity of the EDTA, *M*, as follows:

$$M = 25 \times 0.01036 / L \times 0.20721 \quad (1)$$

where *L* = EDTA solution, mL. Also 1 mL of primary standard 0.05 M lead nitrate = 0.01036 g of lead and 0.20721 is the milliequivalent weight of lead.

6.6 *Primary Standard* (0.05 M lead nitrate)—Dissolve 16.5615 g reagent grade lead nitrate in distilled water and dilute to 1 L (1 mL = 0.01036 g lead).

6.7 *Copper-EDTA Solution*—Mix equivalent amounts of cupric sulfate solution (6.4) and EDTA solution (6.5) and

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.

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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."

store in a dropping bottle. The cupric sulfate EDTA equivalence may be determined as follows:

6.7.1 Pipet 10 mL of cupric sulfate into a beaker, and add concentrated ammonium hydroxide (6.1) dropwise until the precipitate that forms just redissolves. Dilute to 200 mL with water, add two Murexide indicator tablets, crush with a stirring rod to dissolve, and titrate immediately with EDTA to purple.

6.8 *Pan Indicator* [1-(2-pyridylazo)-2-naphthol]—Dissolve 0.1 g in 100 mL of ethanol.

6.9 *Murexide Indicator Tablets*—Ammonium salt of purpuric acid.

6.10 *Nitric acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

7. Procedure

7.1 Weigh accurately 0.2 g of pigment and transfer to a 400-mL beaker. Add 5 mL of nitric acid (6.10) and 50 mL of water and boil gently for 10 min. Cool, and add ammonium hydroxide (6.1) until the pH of the solution is from 5 to 5.5 (as indicated by pH paper). Add 25 mL of acid ammonium acetate (6.3), boil 5 min, dilute to 200 mL with water, heat to boiling, add 4 drops of copper-EDTA (6.7) and 12 drops of PAN (6.8), and titrate while hot with standard EDTA to a clear yellow.

8. Calculation

8.1 Calculate the percent zinc oxide, A , as follows:

$$A = L \times M \times 0.08137 \times 100/S \quad (2)$$

where:

L = EDTA required for titration, mL,

M = molarity of EDTA solution,

S = specimen, g, and

0.08137 = milliequivalent weight of zinc oxide.

TOTAL PHOSPHORUS AS PHOSPHOROUS ACID

9. Reagents

9.1 *Standard Phosphorus Solution*—Dissolve 0.9156 g of secondary sodium phosphate (Na_2HPO_4) in about 200 mL of water. Add 20 mL of nitric acid (sp gr 1.42), dilute to 1 L, and mix. 1 mL = 0.0002 g P.

9.2 *Ammonium Vanadate Solution*—Dissolve 2.5 g of ammonium vanadate (NH_4VO_3) in 500 mL of hot water. When solution is complete, add 10 mL nitric acid (sp gr 1.42), cool, dilute to 1 L, and mix.

9.3 *Ammonium Molybdate Solution*—Dissolve 100 g of ammonium molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$) in 800 mL of water and dilute to 1 L. Filter before use.

9.4 *Bromine*, reagent grade.

9.5 *Bromine Water*, distilled water saturated with bromine.

9.6 *Nitric Acid* (sp gr 1.42), concentrated nitric acid (HNO_3).

9.7 *Nitric Acid* (2:3)—Dilute 200 mL of nitric acid (sp gr 1.42) to 500 mL with water.

10. Apparatus

10.1 *Photoelectric Colorimeter*, either a single- or double-beam spectrophotometer with a wavelength capability of 465 μm .

10.2 *Volumetric Flasks*, 100-mL.

10.3 *Tubes*, 10-mm or 20-mm.

10.4 *Filter Paper*, medium texture.

11. Calibration Curve

11.1 Transfer from a pipet 0.0, 1.0, 3.0, 7.0, 10.0, 15.0, and 20.0-mL aliquots of standard phosphorus solution (9.1) to a 100-mL volumetric flask. To each flask add 10 mL of nitric acid (9.7), 10 mL of ammonium vanadate (9.2), and 10 mL of ammonium molybdate (9.3), dilute to the mark, and mix. Allow solutions to stand 20 min and transfer to a 20-mm tube. Read the absorbance of the solution at 465 μm . Construct a calibration curve for phosphorus by plotting the absorbance versus milligrams of phosphorus in 100 mL of solution for each standard solution.

12. Procedure

12.1 Weigh accurately 0.2 g of pigment and transfer to a 150-mL beaker. Add 25 mL of bromine water (9.5), 10 mL nitric acid (9.6), and 0.5 mL bromine (9.4), and mix the solution. Allow to stand in a warm place for about 1/2 h, and then gently boil off the bromine until the solution is clear.

12.2 Cool the solution and transfer to a 100-mL volumetric flask. Make to the mark with water and mix.

12.3 Pipet 10 mL of the solution into a 100-mL volumetric flask and add 10 mL nitric acid (9.7), 10 mL of ammonium vanadate (9.2), and 10 mL of filtered ammonium molybdate (9.3).

12.4 Dilute the sample to the mark, mix, and allow to stand for 20 min, and transfer to a 20-mm tube.

12.5 Read the absorbance of the sample at 465 μm and from the calibration curve, read the number of milligrams of phosphorus in the test solution.

12.6 Run a blank with the sample.

13. Calculation

13.1 Calculate the percent phosphorus acid, C , as follows:

$$C = A - B \times 2.646/S \times 10 \quad (3)$$

where:

A = phosphorus in test solution, mg,

B = phosphorus in reagent blank, mg,

S = specimen in aliquot, g and

2.646 = gravimetric factor to convert phosphorus to phosphorous acid.

14. Precision⁴

14.1 In an interlaboratory study of this test method, in which two samples containing 18.5 to 19.0 % H_3PO_3 and 77.0 to 78.0 % ZnO were analyzed by one operator in each of four laboratories for H_3PO_3 and five laboratories for ZnO, the within-laboratory and between-laboratories pooled standard deviations, after discarding one day's results from one laboratory for both samples of ZnO (see Table 1). 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*—Two results, each the mean of two

⁴ Supporting data are available from ASTM Headquarters. Request RR:D01.1048.

TABLE 1 Standard Deviation

	Within-laboratory		Between-laboratory	
	s_w	DF	s_b	DF
H ₃ PO ₃	0.198	8	0.515	6
ZnO	0.193	8	0.42	8

runs, obtained by the same operator should be considered suspect if they differ by more than 0.65 % absolute at H₃PO₃ contents of 18 to 20 % and more than 0.63 % absolute at ZnO contents of 75 to 80 %.

14.1.2 *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 1.78 % absolute for H₃PO₃ and 1.37 % absolute for ZnO at the same contents as in 14.1.1.

15. Keywords

15.1 phosphorus acid; pigment analysis; zinc; zinc hydroxy phosphate; zinc oxide

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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 Zinc Hydroxy Phosphite Pigment¹

This standard is issued under the fixed designation D 4462; the number immediately following the designation indicates the year of original adoption 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 a zinc hydroxy phosphite pigment having the general formula $\text{ZnHPO}_3 \cdot [\text{ZnO} \cdot 2\text{Zn}(\text{OH})_2]$.

2. Referenced Documents

2.1 ASTM Standards:

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out³
- D 4450 Test Method for Analysis of Zinc Hydroxy Phosphite Pigment³

3. Composition and Properties

3.1 The pigment shall consist of zinc hydroxy phosphite and shall conform to the requirements in Table 1.

3.2 The mass color of the dry pigment is white. The shade and tinting strength, when specified, shall be within mutually agreed upon limits of a reference sample acceptable by the purchaser and the seller as tested in an agreed test procedure.

4. Sampling

4.1 Two samples shall be taken at random from different packages from each lot, batch, day's pack, or other unit of production in a shipment. When no markings distinguishing between units of production appear, samples shall be taken from 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 two samples shall be taken. At the option of the purchaser, the samples may be tested separately or after blending in equal quantities the samples from the same production unit to form a composite sample.

5. Test Methods

5.1 Tests shall be conducted in accordance with the following ASTM test methods. Test procedures not covered by ASTM test methods shall be mutually agreed upon between the purchaser and the seller.

5.2 *Chemical Analysis*—Test Method D 4450.

5.3 *Moisture Loss*—Determine the hygroscopic moisture loss by Method A of Test Methods D 280.

5.4 *Oil Absorption*—Test Method D 281.

5.5 *Sieve Residue*—Determine the residue on a No. 325 sieve in accordance with the Procedure for Insoluble Dry Pigments of Test Methods D 185.

TABLE 1 Requirements for Zinc Hydroxy Phosphite Pigment

	Min	Max
Zinc (as ZnO), %	74.7	79.7
Phosphorus (as H_3PO_3), %	18.5	20.1
Moisture, %	...	1.25
Oil absorption, lb/100 lb pigment, %	15	22
Residue, No. 325 sieve, %	...	1.0

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 D01.31 on Pigment Specifications.

Current edition approved July 16, 1985. Published October 1985.

² Annual Book of ASTM Standards, Vols 06.01 and 06.02.

³ Annual Book of ASTM Standards, Vol 06.02.

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Standard Test Methods for Analysis of Calcium Borosilicate¹

This standard is issued under the fixed designation D 4487; the number immediately following the designation indicates the year of original adoption 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 analysis of the pigment commercially known as calcium borosilicate.

1.2 The test methods appear in the following order:

Test Methods	Sections
Silicon Dioxide (SiO ₂)	6 to 9
Iron Oxide (Fe ₂ O ₃)	10 to 13
Boron Trioxide (B ₂ O ₃)	17 to 20
Calcium Oxide (CaO)	21 to 23
Moisture and Volatile Matter	24
Water of Hydration	25 to 26
Coarse Particles	27
Oil Absorption	28

1.3 Individual specimens may be used for the direct determinations of SiO₂, B₂O₃, and CaO. SiO₂ and Fe₂O₃ should be removed before the determination of the B₂O₃ and CaO.

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 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints²
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments³
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out³
- D 1193 Specification for Reagent Water⁴

3. Significance and Use

3.1 These test methods compile in one place, recommended procedures for analysis of the pigment known commercially as calcium borosilicate. This pigment is used extensively in paints and the composition is important to the user and producer.

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 II of Specification D 1193.

4.3 *Concentration of Acids and Ammonium Hydroxide*—When acids and ammonium hydroxide are specified by name or chemical formula only, it should be understood that concentrated reagents of the following specific gravity are intended:

Hydrochloric acid (HCl)	1.19
Nitric acid, (HNO ₃)	1.42
Sulfuric acid (H ₂ SO ₄)	1.84
Ammonium hydroxide (NH ₄ OH)	0.90

5. Preparation of Samples

5.1 Thoroughly mix and comminute the sample before taking portions for analysis.

SILICON DIOXIDE

6. Apparatus

- 6.1 *Evaporating Casserole*, 250-mL capacity.
- 6.2 *Hot Plate*.
- 6.3 *Porcelain Filtering Crucible*, medium porosity, 30-mL capacity.
- 6.4 *Drying Oven*, maintained at 100 ± 5°C.
- 6.5 *Muffle Furnace*.

7. Reagents

- 7.1 *Hydrochloric Acid* (1+1).
- 7.2 *Hydrochloric Acid* (1+19).
- 7.3 *Hydrochloric Acid* (1+99).

8. Procedure

8.1 Introduce a 1-g specimen, weighed to the nearest 0.1 mg into an evaporating casserole. Add 50 mL of HCl (1+1) and thoroughly mix.

8.2 Place the casserole on a hot plate and evaporate carefully to dryness.

8.3 Place the casserole in the oven at 100°C for 2 h. Do

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.

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² *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

³ *Annual Book of ASTM Standards*, Vol 06.02.

⁴ *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

⁵ "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."

not allow the oven temperature to exceed 105°C at any time. Remove the casserole and allow to cool for 10 min.

8.4 Completely wet the residue with 25 mL of HCl (1+1) and cover the casserole with a watch glass. Warm just to boiling on a hot plate and maintain for 10 min.

8.5 Add 25 mL of water, free any material from the sides of the casserole with a stirring rod, and immediately filter through a tared porcelain crucible of medium porosity.

8.6 Wash the residue with two 5-mL portions of hot HCl (1+19), one 5-mL portion of hot HCl (1+99), and finally with two 5-mL portions of hot water. Save the combined filtrates for the determination of iron oxide (Sections 10 to 12).

8.7 Place the crucible containing the precipitate in the muffle furnace from 600 to 800°C and heat to constant weight (± 4 mg). Cool in a desiccator and weigh.

9. Calculation

9.1 Calculate the percent of SiO_2 , A , as follows:

$$A = R/S_1 (100) \quad (1)$$

where:

R = weight of residue, g, and

S_1 = weight of original specimen, g.

IRON OXIDE

10. Apparatus

10.1 *Volumetric Flasks*, 250-mL and 1000-mL capacity.

10.2 *Buret*, 10-mL capacity.

11. Reagents

11.1 *Potassium Iodate* (0.01878 N)—Dry 1.0 g of KIO_3 at 120°C for 2 h in a drying oven. After cooling, weigh 0.6700 g and dissolve it in 100 mL of water. Dilute the solution to 1 L in a volumetric flask. 1 mL = 0.001500 g Fe_2O_3 .

11.2 *Potassium Iodide* (KI)—Iodate free.

11.3 *Starch Indicator Solution*—Make a homogeneous paste of 10 g of soluble starch in cold water. Add to this 1 L of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/L) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to 10°C. Prepare fresh indicator when the end point of the titration from blue to colorless fails to be sharp.

11.4 *Sulfuric acid* (H_2SO_4) (1+18).

11.5 *Sodium Thiosulfate, Standard Solution* (0.025 N)—Dissolve 1.5 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 50 mL of water and dilute to 250 mL. Standardize as follows: Pipet 10 mL of the KIO_3 solution into each of three 150-mL beakers. Dilute each to 100 mL with water, add 2 g of KI and 5 mL of H_2SO_4 (1+18), and dissolve the KI with stirring. Titrate the liberated iodine with 0.025 N $\text{Na}_2\text{S}_2\text{O}_3$ solution until the color of the solution becomes pale yellow. Add 2 mL of starch indicator and continue the titration dropwise until the color changes from blue to colorless.

$$I = 1 \text{ mL } \text{Na}_2\text{S}_2\text{O}_3 = 0.01500/V_1 \text{ g } \text{Fe}_2\text{O}_3 \quad (2)$$

where:

I = iron oxide equivalent of $\text{Na}_2\text{S}_2\text{O}_3$ solution,

V_1 = $\text{Na}_2\text{S}_2\text{O}_3$ required for titrations, mean, mL, and

0.01500 = (10.00 mL KIO_3) $\times$ (0.001500 g Fe_2O_3 /mL KIO_3).

12. Procedure

12.1 Dilute the solution obtained from the procedure in 7.6 to 100 mL with water. Add 10 mL of HCl and 5 g KI. Dissolve the KI with stirring.

12.2 Titrate with 0.025 N $\text{Na}_2\text{S}_2\text{O}_3$ solution until the color becomes a pale yellow. Add 2 mL of starch indicator solution and continue the titration until the color changes from blue to colorless.

13. Calculation

13.1 Calculate the percent of iron oxide, D , as follows:

$$D = (I \times V_2)/S_1 (100) \quad (3)$$

where:

V_2 = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration, mL, and

S_1 = weight of original specimen, g.

SOLUTION OF PIGMENT FOR THE DETERMINATION OF BORON TRIOXIDE AND CALCIUM OXIDE

14. Apparatus

14.1 *Boiling Flask*—300-mL capacity with ground glass connection.

14.2 *Büchner Funnel*, 56-mm diameter.

14.3 *Filter Paper*, 55-mm diameter.⁶

14.4 *Filter Flask*, 250-mL capacity.

14.5 *Hot Plate/Stirrer*.

14.6 *pH Meter*.

14.7 *Reflux Condenser*, with ground glass connection, water cooled.

14.8 *Sintered Glass Crucible*, 50-mL capacity, medium porosity.

14.9 *Volumetric Flask*, 250-mL capacity.

15. Reagents

15.1 *Hydrochloric Acid* (1+1).

15.2 *Hydrochloric Acid* (1+25).

15.3 *Nitric Acid* (1+1).

15.4 *Potassium Hydroxide*, pellets.

15.5 *Potassium Hydroxide Solution*, 28 g/L.

16. Procedure

16.1 Introduce 2.5 g of sample, weighed to 0.1 mg, into the 300-mL boiling flask.

16.2 Add 50 mL of HCl (1+1) and 2 drops HNO_3 (1+1). Place a magnetic stirring bar in the flask, connect the reflux condenser to the flask, and reflux for 1 h on the hotplate with constant stirring.

16.3 Remove the hotplate and place an ice bath under the flask. After the solution has cooled, wash the condenser down with a small amount of water (10 mL) and remove from the flask.

16.4 Filter the mixture through a Büchner funnel equipped with filter paper⁶ into a 250-mL filter flask. Wash the boiling flask with several 5-mL portions of hot water to transfer all the material to the filter.

16.5 Cool the filtrate in an ice bath to a temperature of approximately 10°C, add 15 g of KOH pellets and swirl to

⁶ Whatman No. 50 Filter Paper, available from Whatman, Inc., or equivalent, has been found satisfactory for this purpose.

dissolve them. Using the pH meter, neutralize the solution to a pH from 6.0 to 7.0, adding first KOH pellets and finally either KOH (28 g/L) or HCl (1+25). Be careful not to add appreciably to the volume of the solution, nor to exceed a pH of 7.0.

16.6 After neutralizing the solution, warm it gently on a hot plate to coagulate the iron precipitate. Filter off the iron precipitate with a medium-porosity sintered glass filter into a clean 250-mL filter flask.

16.7 Replace the sintered glass crucible on the original filter flask and add 10 mL of water. Dissolve the iron precipitate by the dropwise addition of HCl (1+1) and constant stirring. Filter with suction and wash the crucible with two 5-mL portions of hot water. Swirl the acidic solution around in the flask to dissolve any iron precipitate adhering to the sides of the flask.

16.8 Repeat the neutralization step (16.5) and filter the solution through the sintered glass crucible into the flask containing the first filtrate (16.7).

16.9 Wash the flask used in the neutralization with two 5-mL portions of water, and then wash the precipitate in the crucible.

16.10 Transfer the filtrate quantitatively to a 250-mL volumetric flask and dilute to volume. Use this filtrate for the determination of boron trioxide (Sections 17 to 19) and calcium oxide (Sections 20 to 22).

BORON TRIOXIDE

17. Apparatus

- 17.1 pH Meter.
- 17.2 Buret, 25-mL capacity.
- 17.3 Volumetric Flasks, 1000-mL and 100-mL capacity.
- 17.4 Drying Oven, maintained at $105 \pm 2^\circ\text{C}$.

18. Reagents

- 18.1 Hydrochloric Acid (1+19).
- 18.2 Mannitol.
- 18.3 Phenolphthalein Indicator Solution—Dissolve 0.10 g phenolphthalein in 50 mL of ethyl alcohol. Dilute to 100 mL with water.
- 18.4 Thymol Blue Indicator Solution—Dissolve 0.1 g of thymol blue in 50 mL of 0.01 N sodium hydroxide. Dilute to 100 mL with ethyl alcohol.
- 18.5 Mixed Indicator Solution—Mix 25 mL of thymol blue indicator solution with 75 mL of phenolphthalein indicator solution. Adjust the pH from 6.0 to 7.0 on a pH meter with either HCl (1+19) or NaOH (0.1 N).
- 18.6 Potassium Hydrogen Phthalate (KHP)—Acidimetric standard.
- 18.7 Sodium Hydroxide, Standard Solution (0.1 N)—Dissolve 4.0 g of NaOH pellets in 100 mL of water and dilute to 1 L in a volumetric flask. Standardize as follows: Dry 2.0 g of KHP for 2 h at 105°C in the oven and cool. Weigh 1.6420 g and dissolve in 50 mL of water. Transfer quantitatively to a 100-mL volumetric flask and dilute to volume with water (1 mL = 0.002800 g B_2O_3). Pipet 25 mL of the KHP solution into each of three 150-mL beakers. Add 3 drops of phenolphthalein indicator and titrate with 0.1 N NaOH until the color changes from colorless to red.

$$B = 1 \text{ mL NaOH} = 0.07000/V_3 \text{ g B}_2\text{O}_3 \quad (4)$$

where:

B = B_2O_3 equivalent of NaOH solution,
 V_3 = NaOH used in standardization titrations, mean, mL, and

$$0.07000 = (25.00 \text{ mL KHP}) \times (0.002800 \text{ g B}_2\text{O}_3/\text{mL KHP}).$$

19. Procedure

19.1 Pipet 50 mL of the filtrate from the procedure in 16.10, which has been diluted to volume, into each of two 150-mL beakers.

19.2 With the pH meter, adjust the pH of each solution to 6.0 using either HCl (1+19) or 0.1 N NaOH. Add 10 g of mannitol and dilute to 100 mL with water.

19.3 Add 1 mL of mixed indicator solution and titrate with 0.1 N NaOH. The end point is when the color changes from yellow to dark violet.

20. Calculation

20.1 Calculate the percent of boron trioxide, E , as follows:

$$E = B \times V_4/S_2 (500) \quad (5)$$

where:

V_4 = NaOH used in titrations, mean, mL, and
 S_2 = weight of original specimen, g.

CALCIUM OXIDE

21. Apparatus

- 21.1 Buret, 25-mL capacity.
- 21.2 Volumetric Flasks, 500-mL and 1000-mL capacity.
- 21.3 Reagents.
- 21.4 Ammonium Hydroxide (1+4).
- 21.5 Buffer Solution—Dissolve 33.75 g of ammonium chloride in 285 mL of NH_4OH , add 5 mL of concentrated ammonium sulfide, and dilute to 500 mL with water.
- 21.6 Calcium Standard—Dry 7 g of calcium carbonate (CaCO_3) at 105°C for 2 h. Weigh 6.2461 g of the dried CaCO_3 into a 500-mL flask. Slowly add HCl (1+1) until all the CaCO_3 has dissolved. Add 200 mL of water and boil for a few minutes to expel CO_2 . Cool and adjust the pH from 5 to 6 by adding NH_4OH (1+4) or HCl (1+1). Transfer solution to a 1000-mL volumetric flask and dilute to volume. 1 mL = 0.003500 g CaO.
- 21.7 Sodium Carbonate (0.2 %)—Dissolve 2 g Na_2CO_3 in water and dilute to 1 L.
- 21.8 Sodium Carbonate (10 g/L)—Dissolve 10 g Na_2CO_3 in water and dilute to 1 L.
- 21.9 Eriochrome Black T Indicator Solution—Dissolve 1.0 g of Eriochrome Black T in 30 mL of 0.2 % sodium carbonate (Na_2CO_3) solution. Dilute to 100 mL with isopropanol. Adjust the pH to 9.0 with Na_2CO_3 solution (10 g/L). Indicator shelf life: 2 months.

21.10 EDTA, Standard Solution (0.17 N)—Weigh 63.3 g of disodium ethylenediamine tetraacetate (EDTA) and 0.1 g magnesium chloride (MgCl_2), and dissolve in 500 mL of NaOH (20 g/L) solution and dilute to 1 L with water. Standardize as follows: Pipet 50 mL of calcium standard into each of three 150-mL beakers. Add 15 mL of buffer solution and 3 to 4 drops of indicator solution to each beaker. Titrate immediately with EDTA solution (0.17 N) until the color changes from red to a definite blue.

$$C = 1 \text{ mL EDTA} = 0.1750/V_5 \text{ g CaO} \quad (6)$$

where:

C = CaO equivalent of EDTA solution,
 V_5 = EDTA required for titrations, mean, mL, and
 $0.1750 = (50 \text{ mL Ca Standard}) \times (0.003500 \text{ g CaO/mL})$.

22. Procedure

22.1 Pipet 50 mL of the filtrate from the procedure in 15.10 into each of two 150-mL beakers. Add 25 mL of buffer and 3 to 4 drops of indicator solution to each beaker.

22.2 Immediately titrate with EDTA until the color changes from red to a definite blue.

23. Calculation

23.1 Calculate the percent of calcium oxide, F , as follows:

$$F = C \times V_6/S_2 (500) \quad (7)$$

where:

V_6 = EDTA required for titrations, mean, mL, and
 S_2 = weight of original specimen, g.

MOISTURE AND OTHER VOLATILE MATTER

24. Procedure

24.1 Determine moisture and other volatile matter in accordance with Method A of Test Methods D 280.

WATER OF HYDRATION

25. Apparatus

25.1 Casserole.

25.2 Muffle Furnace, preferably electrically heated maintained at a temperature of $450 \pm 10^\circ\text{C}$.

26. Procedure

26.1 Place the casserole in the muffle furnace and heat at 450°C for 30 min. Remove to a desiccator, cool, and weigh to the nearest 0.1 mg.

26.2 Remove the weighing bottle containing approximately 3 to 5 g of previously dried pigment (Section 24) from the desiccator. Transfer most of the pigment to the casserole and weigh to 0.1 mg. Subtract the weight of the dish from the total weight to obtain the specimen weight.

26.3 Place the dish containing the specimen in the muffle furnace heated at a temperature of about 250°C and raise the temperature to 450°C . Maintain temperature at 450°C for 1 h. Remove the dish from the furnace to the desiccator and allow to cool to room temperature. Weigh to the nearest 0.1

mg. Heat again at 450°C for 15 min to check the loss in weight. The two weights should not differ by more than 4 mg.

26.4 Calculate the percent water of hydration on the basis of dried pigment to 0.01 %.

COARSE PARTICLES

27. Procedure

27.1 Determine the percent of coarse particles in the pigment in accordance with Test Methods D 185.

28. Oil Absorption

28.1 Determine the oil absorption of the pigment in accordance with Test Method D 281.

29. Precision⁷

29.1 In an interlaboratory study of this test method in which operators at four locations made single analyses on two days of two pigments containing 32.9 and 39.3 % SiO_2 , 16.5 and 10.6 % B_2O_3 and 40.7 and 44.1 % CaO , the within-laboratory pooled standard deviations were found to be 0.27 % for SiO_2 , 0.13 % for B_2O_3 , and 0.31 % for CaO , each with 8 df, and the equivalent between-laboratories standard deviations 0.346 %, 0.255 % and 0.495 %, with 6 df each. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

29.1.1 *Repeatability*—Two results, each single determinations, obtained by the same operator on different days should be considered suspect if they differ in percent absolute by more than 0.88 % SiO_2 , 0.48 % B_2O_3 , and 1.01 % CaO at levels of 32 to 40 % SiO_2 , 10.5 to 16.5 % B_2O_3 and 40 to 45 % CaO .

29.1.2 *Reproducibility*—Two results, each single determinations, obtained by operators in different laboratories should be considered suspect if they differ in percent absolute by more than 1.20 % SiO_2 , 0.88 % B_2O_3 , and 1.72 % CaO at the same levels as in 29.1.1.

30. Keywords

30.1 boron trioxide; calcium borosilicate pigment analysis; calcium oxide; iron oxide; silicon dioxide; water of hydration

⁷ Supporting data are available from ASTM Headquarters. Request RR: D01-1047.

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Standard Test Method for Measuring Apparent pH of Water Insoluble Phenol-Formaldehyde Resins¹

This standard is issued under the fixed designation D 4613; the number immediately following the designation indicates the year of original adoption 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.

⁶ NOTE—Keywords were added editorially in September 1990.

Scope

1.1 This test method covers the measurement of the apparent pH of a water extract of an acetone solution of water insoluble phenol-formaldehyde resin.

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 1193 Specification for Reagent Water²

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode³

3. Summary of Test Method

3.1 Aqueous test solutions are generally used in determining pH. In this test method the resin is dissolved in acetone, water is added, and the apparent pH is measured with a pH meter.

4. Significance and Use

4.1 The hydrogen ion concentration (pH) is of critical importance due to its effect on the performance of the end product. The pH is widely used in quality control and process control.

5. Apparatus

5.1 *pH Meter*, complying with Test Method E 70.

5.2 *Beaker*, 250-mL.

5.3 *Balance*, accurate to 0.1 g.

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 *Water*, conforming to Type II of Specification D 1193.

6.3 *Acetone*, reagent grade.

6.4 *pH Standard Solutions*—4, 7, 10 for standardization of pH meter.

7. Procedure

7.1 Standardize the pH meter with the 4, 7, or 10 standard buffer in the expected testing range.

7.2 Weigh a 10 ± 0.1 g specimen of the resin under test into a 250-mL beaker.

7.3 Add 40 mL of acetone and dissolve the specimen. Add 40 mL of water and stir for 5 min at room temperature. Allow the solution to settle for 1 min. Immerse the electrode into the solution (Caution: Keep undissolved resin away from electrode) and record the result.

7.4 Wash the electrode with acetone, followed by water until clean or until the pH meter returns to the standard buffer reading.

8. Report

8.1 Report the pH to 0.1 units.

9. Precision and Bias

9.1 The precision and bias for this test method have not been determined.

10. Keywords

10.1 pH; phenol-formaldehyde resins; water-insoluble

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.

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² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ *Annual Book of ASTM Standards*, Vol 15.03.

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."

 D 4613

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Standard Test Method for Volatile Content in Phenolic Resins¹

This standard is issued under the fixed designation D 4639; the number immediately following the designation indicates the year of original adoption 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 in August 1990.

1. Scope

1.1 This test method covers the determination of matter in a solid phenolic resin that is volatile at 300°F (150°C).

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. Summary of Test Method

2.1 A specified weight of resin is added to a tared aluminum dish and weighed. The resin is then heated for 2 h in an oven at 300°F (150°C) and reweighed.

3. Significance and Use

3.1 Volatile material causes problems when phenolic resins and varnishes are heated at temperatures above 300°F (150°C). Heating a specified weight of resin for 2 h at this temperature indicates the amount of volatile material emitted when molding or curing phenolic resins.

4. Apparatus

4.1 *Analytical Balance*, capable of weighing to 0.1 mg.

4.2 *Thermometer*, glass, having a range from 1 to 200°C and accurate to 1°C.

4.3 *Circulating Oven*, maintained at 300 ± 3.5°F (150 ± 2°C).

4.4 *Aluminum Drying Dishes*, 2½-in. (63 mm) diameter, ¾-in. (20 mm) depth or equivalent.

4.5 *Desiccator*.

5. Procedure

5.1 Mark two aluminum dishes for identification of each resin being tested and weigh them on the analytical balance to 0.1 mg. Record as w_1 .

5.2 Weigh a 5-g specimen of resin to 0.1 mg into each aluminum dish. Record total weight as w_2 .

5.3 Place the dishes in the oven for 2 h at 300°F (150°C).

5.4 Remove from oven and immediately place in desiccator until they cool to room temperature. Reweigh immediately upon removal from the desiccator and record as w_3 .

6. Calculation

6.1 Calculate the volatile content, V , as follows:

$$V, \% = 100 - \left[\frac{w_3 - w_1}{w_2 - w_1} \times 100 \right]$$

where:

w_1 = weight of aluminum dish, g,

w_2 = weight of aluminum dish and specimen used, g, and

w_3 = weight of aluminum dish and specimen after 2 h in oven, g.

7. Report

7.1 Report the following information:

7.1.1 Resin identification, time, and temperature.

7.1.2 Percent of volatile matter, or volatile content (average of duplicate determinations).

8. Precision and Bias

8.1 The precision and bias for this method have not been determined.

9. Keywords

9.1 phenolic resins; volatile 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 D01.33 on Polymers and Resins.

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Standard Test Method for Determining Stroke Cure Time of Thermosetting Phenol- Formaldehyde Resins¹

This standard is issued under the fixed designation D 4640; the number immediately following the designation indicates the year of original adoption 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 September 1990.

1. Scope

1.1 This test method covers the determination of the cure rate at any specified temperature for a thermosetting phenol-formaldehyde resin. The time at that temperature required to make a fluid mixture change into a hard immovable mass indicates speed of cure.

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. Description of Terms Specific to This Standard

2.1 *stroke cure time*—the time required for a resin being held at a specified temperature on a hot plate and being stroked with a spatula, to adhere to the hot plate and not to the spatula.

3. Summary of Test Method

3.1 This test method employs the use of controlled heat and a thin layer of resin to determine the time in seconds required to change from a fluid stage to a cured immovable stage.

4. Significance and Use

4.1 This method is significant as a process control for polymerization time.

4.2 When the cure times are determined at several temperatures and plotted as time versus temperature, different resins can be compared for curing characteristics.

5. Apparatus

5.1 *Electric Hot Cure Plate*, with a smooth surface free of

rough spots with adjustable control to hold the temperature within 3.5°F (2°C) (Note 1):

5.2 *Spatula*, 3 by 7/16 in. (75 by 10 mm), stainless.

5.3 *Stopwatch*.

5.4 *Knife*, brass, putty with straight edge.

6. Procedure

6.1 Adjust the hot plate to the desired test temperature.

NOTE 1—Usual temperatures are 300°F (150°C), 320°F (160°C), and 330°F (165°C).

6.2 Place an approximately 0.5-g specimen on the hot plate and spread over an approximately 2-in.² (130-mm²) area with a clean spatula as quickly as possible. Start the stopwatch immediately upon adding the specimen to the hot plate, then stroke steadily across the specimen in alternating smoothing and patting motions.

6.3 As the resin approaches the cured stage, it becomes more viscous and stringy. Stop the stopwatch when the resin on the hot plate and the spatula no longer adhere to each other. Record the elapsed time.

6.4 Remove cured resin from hot plate immediately with the straight edged putty knife.

NOTE 2—The longer on the hot plate, the harder it is to remove.

NOTE 3—Test results can be affected by differences in construction of hot plates, the stroking and patting with spatula, and the starting and stopping of the stopwatch.

7. Report

7.1 Report the following information:

7.1.1 Stroke cure time in seconds; record to the nearest whole number.

7.1.2 Test temperature.

8. Precision and Bias

8.1 No statement is made about either the precision or bias of this method for measuring stroke cure since the result merely states whether there is conformance to the criteria for success specified in the procedure.

9. Keywords

9.1 phenol-formaldehyde resins; stroke cure time

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 Nov. 28, 1986. Published January 1987.

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Standard Test Method for Determining Qualitatively Methylol Group in Phenolic Resins¹

This standard is issued under the fixed designation D 4706; the number immediately following the designation indicates the year of original adoption 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 to determine qualitatively the presence of methylol group in phenol-formaldehyde resins.

NOTE—The values stated in SI units are to be regarded as standard.

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 This test method of qualitatively determining the presence of methylol groups in a phenol resin uses the reaction of ferric chloride with the methylol group to produce a blue color at room temperature.

3. Significance and Use

3.1 This test method provides a fast and simple method for determining the presence of methylol group for quality control testing.

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.

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4. Apparatus

- 4.1 *Test tube*, 6 by 1-in. (152 by 25-mm) glass.
- 4.2 *Acetone*, reagent grade.
- 4.3 *Ferric chloride*, 5 % aqueous solution.
- 4.4 *Balance*.

5. Procedure

- 5.1 Place 0.1 g of the specimen under test in a test tube.
- 5.2 Add 10 mL of acetone.
- 5.3 Shake and dissolve specimen.
- 5.4 Add two drops of 0.5 % ferric chloride solution and swirl.
- 5.5 Observe color, yellow or blue.

6. Report

- 6.1 Report the following information:
 - 6.1.1 Yellow equals methylol group absent.
 - 6.1.2 Blue equals methylol group present.

7. Precision and Bias

7.1 Precision and bias do not apply since this is a qualitative test method that is recorded by a color change.

8. Indexing Terms

8.1 This test method is indexed under the following terms: ferric chloride; methylol group content; phenol-formaldehyde (PF) plastics; plastics, general; and resins (subheading phenolic).



Standard Test Method for Determining Unreacted Monomer Content of Latexes Using Gas-Liquid Chromatography¹

This standard is issued under the fixed designation D 4747; the number immediately following the designation indicates the year of original adoption 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 free monomer content of acrylic latexes. Monomers that have been successfully determined by this procedure include *n*-butyl methacrylate, *n*-butyl acrylate, styrene, and methyl methacrylate. The determination of other monomers has not been evaluated, but this test method is believed to be applicable. The established working range of this test method is from 100 to 1000 $\mu\text{g/g}$, but there is no reason to believe it will not work outside of this range, provided that appropriate dilutions and adjustments in specimen size are made.

1.2 The volatile composition of acrylic latexes is expected to change with time and environmental factors. This time dependence of the determination may be seen as an artificially large deviation of results, making the method mostly applicable for in-house quality control, where sampling and analysis conditions can be better controlled.

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.* See Section 7 for specific hazard statements.

2. Referenced Documents

2.1 ASTM Standards:

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials²

E 260 Practice for Packed Column Gas Chromatography³

3. Summary of Test Method

3.1 A suitable aliquot of the latex is internally standardized with isobutyl acrylate, diluted with water, and then injected into a gas chromatographic column containing a packing material coated with a stationary phase that separates the internal standard and monomers in question from each other and from other volatile compounds.

4. Significance and Use

4.1 Excessive amounts of unreacted monomer may cause

concerns relating to toxicity and odor. This test method is designed to measure the unreacted monomer content of latexes. The results may be used to monitor the extent of polymerization during manufacture, as well as to establish maximum unreacted monomer content for regulatory purposes.

5. Apparatus

5.1 *Gas Chromatograph*, any gas-liquid chromatographic instrument having a flame ionization detector and linear temperature programming. An injection port using replaceable glass liners to facilitate periodic removal of accumulated residues is recommended.

5.2 *Column*, 2 by 2-mm inside diameter glass or 6 ft by 1/8-in. outside diameter steel tubing, packed with 10 % by weight of a 2-nitroterephthalic acid derivative of Carbowax 20M on 100/120 mesh acid washed, silane treated diatomaceous earth.⁴ A column of equivalent or superior performance may also be used.

5.3 *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 level of ± 0.03 % of full scale (see Practice E 260).

5.4 *Liquid Charging Devices*, microsyringe, 10- μL capacity or an automatic liquid sampling device.

5.5 *Dropper Pipets*, glass, disposable.

5.6 *Vials*, approximately 7-mL capacity, with caps. Open top screw cap vials fitted with polytetrafluoroethylene/silicone septa are preferred.

5.7 *Autosampler Vials*, 2-mL capacity (optional).

5.8 *Analytical Balance*, accurate to 0.1 mg.

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

⁴ Columns prepared from the following stationary phases and supports have been found suitable for this purpose:

Stationary Phases

SP-1000—Supelco, Inc., Bellefonte, PA

AT-1000—Alltech Assoc., Deerfield, IL

OV-351—Ohio Valley Chem. Co., Marietta, OH

FFAP—Various manufacturers

Supports

Chromosorb W-HP—Manville Corp., Denver, CO

Gas Chrom QII—Alltech Assoc., Deerfield, IL

Supelcoport—Supelco, Inc., Bellefonte, PA

Anakrom Q—Analabs, North Haven, CT

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.

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² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 14.01.

TABLE 1 Instrument Conditions

Detector	flame ionization
Air flow	240 mL/min
Hydrogen flow	30 mL/min
Column (suggested)	2 by 2 mm inside diameter glass, packed with 10 % of a 2-nitroterephthalic acid derivative of Carbowax 20M on 100/120 mesh acid washed, silane-treated diatomaceous earth.
Carrier gas, flow rate	Helium, 30 mL/min
Temperatures:	
Injection port	250°C
Detector block	250°C
Column	
Initial	80°C
Hold time	4 min
Program rate	8°C/min
Final	200°C, or higher as needed (see 8.1)
Final hold	10 min, or as needed
Injection volume	2 μ L

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.

6.2 *Carrier Gas*, helium of 99.995 % or higher purity. High purity nitrogen may also be used.

6.3 *Acetone*, reagent grade.

6.4 *Isobutyl Acrylate* (internal standard), 99+ % pure.

NOTE [—Isobutyl acrylate was found to be a suitable internal standard, but any other monomer not found in the sample may be substituted. The internal standard chosen should yield a clear chromatographic separation, and should be free of interferences.

6.5 *Monomers of Interest*, 99+ % pure.

7. Hazards

7.1 Acrylic and methacrylic monomers are considered hazardous. Precautions should be taken to avoid inhalation and skin or eye contact with these chemicals. All sample preparations should be done in a well-ventilated area, such as a fume hood.

8. Preparation of Apparatus

8.1 *Column Conditioning*—Attach one end of the column to the inlet side of the instrument leaving the exit end of the column disconnected. This prevents the contamination of the detector due to column bleed. Set the helium flow rate at 30 mL/min and purge the column at ambient temperature for 30 min. Program the column oven from 50 to 220°C at 2°C/min and maintain at 220°C overnight. In no case should the temperature of the column be allowed to exceed 275°C.

8.2 After conditioning, connect the exit end of the column to the detector 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 baseline. 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 rate to a constant value.

9. Calibration

9.1 Determine the retention of each component expected to be present by injecting small amounts either separately or in known mixtures. Retention times should be determined each day the method is used.

9.2 *Standardization*—Determine in duplicate the relative response of the monomers of interest to the isobutyl acrylate internal standard as follows:

9.2.1 Weigh to 0.1 mg about 0.05 g of isobutyl acrylate and each monomer of interest into a vial (5.6). Weigh approximately 5 g of acetone into the vial and mix well.

9.2.2 Weigh approximately 0.05 g of the solution (9.2.1) into another vial, add approximately 5 g of acetone and mix well.

9.2.3 Inject a 1- μ L aliquot of the solution from 9.2.2 onto the column and record the chromatogram. The elution order for acetone and each of the monomers using the conditions given in Table 1 is shown in Fig. 1.

9.2.4 Measure the peak areas, of the individual components and calculate the relative response factor, *RF*, for the monomers of interest as follows:

$$RF = (W_1 \times A_s) / (W_s \times A_1)$$

where:

RF = relative response factor for each monomer,

*A*₁ = peak area produced by the monomer,

*A*_s = peak area produced by the internal standard,

*W*₁ = weight of monomer used for calibration (9.2.1), and

*W*_s = weight of internal standard (9.2.1).

10. Procedure

10.1 If the composition of the latex is not known or if the approximate level of monomers in the latex is not known, a preliminary analysis must be performed by diluting approx-

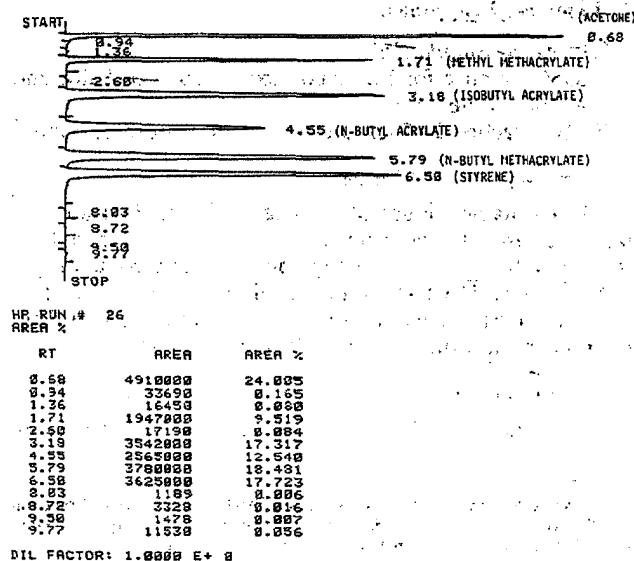


FIG. 1 Typical Chromatogram

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."

approximately 0.5 g of latex with approximately 5 g of water and injecting a 2- μ L aliquot into the chromatographic column. Using the same conditions as for standardization, record the peaks of all components at attenuation settings that provide maximum peak heights. Use the relative retention times to identify the monomers present. If the specimen has a component eluting at the same retention time as isobutyl acrylate, choose a different internal standard (Note 1).

10.2 Prepare a dilute solution of the internal standard by weighing to 0.1 mg about 0.05 g of isobutyl acrylate and 5 g of acetone into a septum vial. Take care to minimize losses due to evaporation. Prepare this solution fresh each day the method is used.

10.3 Weigh to 0.1 mg an appropriate amount of sample into a septum vial using Table 2 as a guide to specimen size. Also weigh to 0.1 mg 50 mg of the dilute solution prepared in 10.2 into the vial. Add about 3 to 5 g of water or acetone. Shake the vials on a wrist action shaker or other suitable device for 15 min.

NOTE 2—The viscosity of a number of latexes increases upon the addition of an organic solvent. If acetone (or another organic solvent) is found to be compatible with the specimen, it should be used as the diluent instead of water. It should be kept in mind that some organic solvents may interfere with the chromatographic separation.

10.4 Inject 2 μ L of the prepared solution (10.3) into the chromatographic column and record the chromatogram using the conditions as in 10.1. Measure the peak areas (Note 3) of the internal standard and relevant monomers, multiplying each area by the appropriate factor to express the peak areas on a common basis.

NOTE 3—Peak areas may be determined by any method that meets the precision requirements of Section 12. Electronic integration is recommended for best results.

10.5 Repeat procedure in 10.3 through 10.4 and calculate the mean values.

11. Calculations

11.1 The weight of the internal standard present in the diluted specimen (10.3) is calculated as follows:

$$W_4 = (W_3/W_6)W_7$$

where:

TABLE 2 Suggested Dilutions

NOTE—This table shall be used only as a guide. If the monomer concentrations are outside the range given, appropriate adjustments must be made in terms of specimen size, dilution and amount of internal standard added.

Level of Free Monomer Expected, μ g/g	Specimen Size, g	Diluent, g
250	2	3
500	1	4
750	0.7	4.3
1000	0.5	4.5

W_4 = weight of internal standard in diluted specimen in 10.3,

W_5 = weight of internal standard used to prepare solution in 10.2,

W_6 = weight of acetone plus weight of internal standard used to prepare solution in 10.2, and

W_7 = weight of the dilute internal standard solution (10.2) added to the specimen in 10.3.

11.2 Calculate the concentration, C , of each monomer present in the latex sample from the results obtained from 10.5 as follows:

$$C = [(A_3 \times W_4 \times RF)/(W_8 \times A_4)] \times 10^6$$

where:

C = concentration of free monomer, μ g/g,

A_3 = peak area produced by the monomer,

A_4 = peak area produced by the internal standard,

RF = relative response factor for each monomer (9.2.4),

W_4 = weight of internal standard (11.1), and

W_8 = specimen weight (10.3).

12. Precision and Bias⁶

12.1 *Precision*—In an interlaboratory study of the method by five laboratories using four samples, the within-laboratory coefficient of variation was found to be 16.8 % relative at 11 degrees of freedom and the between-laboratories coefficient of variation was 18.1 % relative at 8 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level (see Practice D 3980 and Note 4).

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 52 % 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 59 % relative.

NOTE 4—Variation in results may be due to the changing composition of the samples used for the study. This precision statement should be used only as a guide, since it represents only the magnitude of variation that is possible, which will vary with time depending on the latex and the particular monomers being determined.

12.2 *Bias*—Bias cannot be determined for this test method.

13. Keywords

13.1 acrylic latexes chromatography (subheading gas chromatography); gas chromatography, analysis of monomers; latex paints; latex vehicles; styrene; trace monomers; unreacted monomers in latexes

⁶ Supporting data are available from ASTM Headquarters. Request RR: D01-1055.

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Standard Test Method for Nonvolatile Content of Latexes¹

This standard is issued under the fixed designation D 4758; the number immediately following the designation indicates the year of original adoption 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 quality control test for nonvolatile content of latex vehicles. The specified time-temperature conditions (180°C, 20 min) were selected to allow test completion in 1 h or less. For latex vehicles used in certain air-dry or low-temperature bake coatings, as well as for those that contain temperature-sensitive materials, the use of a lower temperature and other bake time will more accurately reflect the effective nonvolatile content (Note 1). Alternative time-temperature conditions should be agreed upon between the producer and user.

NOTE 1—The test conditions of Test Method D 2369 (110°C, 60 min) have been found suitable for use with many latex vehicles.

1.2 This test method is not intended to be employed for determining the volatile organic content (VOC) of formulated coatings.

1.3 *This standard may involve hazardous materials, operations or 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 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 2369 Test Method for Volatile Content of Coatings²
E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens³
E 300 Practice for Sampling Industrial Chemicals⁴

3. Significance and Use

3.1 This test method is designed to quantify the weight percent of a commercial, synthetic latex product that, when incorporated into a coating product, constitutes the binder content of the applied coating.

4. Sampling

4.1 Procedures described in the Slurry Sampling section of Practice E 300 shall be used for sampling latexes.

5. Apparatus

- 5.1 *Oven*, forced-ventilation Type IIB, as specified in Specification E 145, with closed vent.
5.2 *Balance*, sensitive to 0.1 mg.
5.3 *Desiccator*.
5.4 *Weighing dish*, aluminum, approximately 57 mm in diameter by 18 mm deep.
5.5 *Syringe*, 5-mL disposable.

6. Procedure

6.1 For each latex to be tested, wash two aluminum dishes with a suitable reagent grade solvent (for example, toluene) or heat to constant weight to assure that volatile contaminants are removed. Determine the tare weight of the aluminum dishes to 0.1 mg.

6.2 Thoroughly mix the sample on a mechanical shaker or can roller to assure homogeneity.

6.3 Draw 3.5 mL of the sample into the syringe and weigh to 0.1 mg.

6.4 Transfer 1.5 ± 0.1 mL of the latex into a tared weighing dish and reweigh the syringe to 0.1 mg. Calculate the latex specimen weight, S , as follows:

$$S = w_1 - w_2$$

where:

w_1 = initial weight of syringe and latex, g,

w_2 = weight of syringe and latex remaining after discharging specimen, g.

6.5 Repeat 6.4 for the duplicate determination.

6.6 Transfer the aluminum dishes to an oven operating at $180 \pm 4^\circ\text{C}$ and heat for 20.0 ± 1 min. (Note 2). Remove from the oven, cool in a desiccator for 5 min at room temperature and weigh to 0.1 mg. Determine the weight of residue, N , in each dish as follows:

$$N = W_1 - W_2$$

where:

W_1 = weight of dish and residue, g,

W_2 = tare weight of aluminum dish, g.

NOTE 2—Add distilled water to viscous test specimen prior to baking.

7. Calculation

7.1 Calculate the mean weight percent nonvolatile material (% NVM) where Subscripts 1 and 2 refer to the individual data from the duplicate determinations:

$$\% \text{ NV} = \frac{N_1/S_1 + N_2/S_2}{2} \times 100$$

8. Report

8.1 Report the nonvolatile content to 0.01 weight %.

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.

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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 06.03 and 15.05.

9. Precision

9.1 An interlaboratory study was conducted on this test method in which one operator in each of five laboratories made duplicate determinations on each of two days on six commercial latices containing 44 and 65 % nonvolatile matter. The latex polymer types were three all-acrylics, a vinyl acetate-acrylic copolymer, and acrylic terpolymer and a vinyl acetate-vinyl chloride-ethylene copolymer. Two laboratories could not run the test on two latexes because they had gelled, and in the statistical analysis all results obtained on one latex were rejected because they differed significantly from the others. The pooled within-laboratory standard deviation was found to be 0.09 % absolute with 20 degrees of freedom and the between-laboratories standard deviation 0.16% absolute with 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:

9.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator should be considered suspect if they differ by more than 0.27 % absolute.

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 0.48 % absolute.

10. Indexing Terms

10.1 This test method is indexed under the following terms: accelerated testing; air dry coatings; baked coatings; latex paints; latex vehicles; low temperature bake coatings; nonvolatile matter content; and temperature tests (sub-heading low).

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 Determination of Ethoxyl Substitution in Cellulose Ether Products by Gas Chromatography¹

This standard is issued under the fixed designation D 4794; the number immediately following the designation indicates the 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 applicable to the determination of ethoxyl substitution in cellulose ether products by a Zeisel gas chromatographic technique.

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 5 and Note 3.*

2. Summary of Test Method

2.1 When ethyl cellulose is allowed to react with hydriodic acid, one mole of ethyl iodide is liberated for each mole of ethoxyl ether substituted on the cellulose chain. The ethyl iodide is extracted in-situ with o-xylene and quantitated by gas chromatography using an internal standard technique.

3. Apparatus

3.1 *Gas Chromatograph* with thermal conductivity detector and heated injection port.

3.2 *Electronic Integrator.*

3.3 *Column*—Glass tubing, two, 6.4-mm outside diameter, 4-mm inside diameter, 1829 mm in length, packed with the reagent in 4.7, coiled to fit injection system and instrument, or equivalent column and packing as appropriate.

3.4 *Syringe*, 10 μ L.

3.5 *Reaction Vials, Caps, and Heating Block.*²

3.6 *Syringe*, 100 μ L.

4. Reagents and Materials

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 Chem-

ical 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.

4.2 *Iodoethane*, 99 % minimum.

4.3 *O-xylene.*

4.4 *Toluene.*

4.5 *Hydriodic Acid*, 57 % (sp gr 1.69 to 1.70).

4.6 *Acetone*, technical grade.

4.7 DC550, 20 %, on 100/120 mesh Chromosorb PAW.

5. Hazards

5.1 Safety precautions must be taken for handling of hydriodic acid.

5.2 During the reaction, the glass vials are under pressure. Exercise precaution in handling the hot vials.

6. Apparatus Preparation and Conditioning

6.1 *Column* (Note 1)—The two pieces of glass tubing are packed with reagent under vacuum and mechanical vibration using silanized glass wool to contain the packing. Install each in the gas chromatograph to facilitate on column injection and allow to come to equilibrium over a 12-h period under the conditions described below:

Oven temperature, °C	100 isothermal
Injection port temperature, °C	200
Detector temperature, °C	200
Detector current, ma	170 ma
Attenuation	1
Polarity	A
Carrier gas	helium
Column A, mL/min	20
Column B, mL/min	20

NOTE 1—The conditions used here were determined to be optimum for the column. Optimum conditions should be determined for each column on an individual basis.

6.2 Integrator:

6.2.1 Settings (Note 2):

Attenuation	1
Start delay	0.5
Chart	auto
Chart speed, cm/min	0.50
Area reject	off
Slope sensitivity	must be determined for each GC

NOTE 2—These settings were used with a Hewlett-Packard 3380 Integrator. Other units may require different settings.

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 D01.36 on Cellulose and Cellulose Derivatives.

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² Reacti-Therm Heating Module, Reacti-Block, Reacti-Vials, and Minnert valve tops, trademarks of the Pierce Chemical Company P.O. Box 117, Rockford, IL 61105-9976, have been found satisfactory for this purpose.

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."

6.2.2 Approximate Component Retention Times:

Minutes	Component
1.13	methyl iodide
1.80	ethyl iodide
2.30	isopropyl iodide
3.00	toluene (internal standard)
4.45	o-xylene

7. Preparation of Standard Solutions

7.1 *Internal Standard Solution* (25 mg toluene/mL o-xylene):

7.1.1 Weigh a 100-mL volumetric flask containing 10 mL of o-xylene to the nearest 0.01 g.

7.1.2 Add 2.50 ± 0.1 g of toluene.

7.1.3 Dilute with o-xylene to 100 mL.

7.2 *Calibration of Standard Solution:*

7.2.1 Pipet 4.0 mL of 57 % hydriodic acid into a 3-dram vial.

7.2.2 Pipet 4.0 mL of the internal standard solution (prepared in 7.1) into the vial and seal with the mininert valve.

7.2.3 Weigh the vial and contents to the nearest 0.1 mg.

7.2.4 Add 90 μ L of ethyl iodide to the vial with a syringe.

7.2.5 Weigh and record the amount of ethyl iodide added.

7.2.6 Mix well.

7.2.7 Calculate the ethoxyl equivalent of the ethyl iodide using the following equation:

$$\text{mg ethoxyl} = \text{g ethyl iodide} \times [(45 \times 1000)/156]$$

$$\text{mg toluene} = \text{concentration of int. std.} \times 4 \text{ mL}$$

8. Calibration of Electronic Integrator

8.1 Inject 2 μ L of the standard solution prepared in 9.2 into the gas chromatograph and record the chromatogram.

8.1.1 Calibrate the integrator according to the manufacturer's instructions.

8.2 If an integrator is not available peak areas and response factors can be determined manually as follows:

$$F = (A \times B)/(C \times D)$$

where:

F = component response factor,

A = weight of ethoxyl equivalent in the calibration standard, mg,

B = peak area of internal standard (toluene) from standard run (8.1),

C = peak area for component from standard run (see 8.1), and

D = weight of internal standard in the calibration standard, mg.

9. Procedure

9.1 *Specimen Preparation:*

9.1.1 Dry the specimen at 105°C for 30 min and store in a dessicator.

9.1.2 Weigh 60 to 70 mg of the specimen prepared in dry 9.1.1 into a clean, tared 5-mL reaction vial (see 3.5).

9.1.3 Add 2.0 mL of the internal standard solution prepared in 7.1 to the vial.

9.1.4 Add 2.0 mL of hydriodic acid to the vial.

9.1.5 Cap tightly and weigh the vial.

9.1.6 Shake the specimen for 30 s.

9.1.7 Place the vial in a heated block (150°C) for 1 h, removing and shaking the specimen after 20 min.

NOTE 3: Warning—Vials contain a hot corrosive acid under pressure.

9.1.8 Remove the specimen and cool in the hood for 45 min. The specimen will separate into two layers.

9.1.9 Reweigh to determine any loss due to leakage. Discard any specimen with loss greater than 25 mg.

9.2 *Analysis:*

9.2.1 Enter the weight of the toluene (internal standard) and the weight of the specimen into the integrator if an integrator is being used.

9.2.2 Inject 2 μ L of the upper layer of the specimen into the gas chromatograph.

10. Calculation

10.1 The integrator reports the ethoxyl concentration in weight percent.

10.2 If an integrator is not available, calculate the peak areas and concentrations manually using the following equation:

$$E = (G \times F \times H \times 100)/(I \times J)$$

where:

E = ethoxyl concentration, weight %,

G = peak area of component,

F = response factor for component (10.2),

H = weight of toluene (mg) in the specimen, mg,

I = peak area of toluene, and

J = specimen weight, mg.

11. Precision and Bias⁴

11.1. *Precision*—The relative precision was found to be 0.64 % at the 95 % confidence level.

11.2 *Bias*—No justifiable statement can be made on the bias of the procedure in this test method because no suitable reference material for determining the bias exists.

⁴ Supporting data is available from ASTM Headquarters. Request RR: D01-1058.

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Standard Test Method for Nitrogen Content of Soluble Nitrocellulose—Alternative Method¹

This standard is issued under the fixed designation D 4795; the number immediately following the designation indicates the year of original adoption 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 Test Methods D 301 for measuring nitrogen content in nitrocellulose by nitrometer are the accepted standard. However, the glassware is specialized and the precision is dependent on the development of a high level of skill by the operator. The ferrous-sulfate titration of nitrate is a classical procedure. By controlling critical variables and automating the actual titration, precision equivalent to the nitrometer can be achieved with nitrocellulose. This test method describes such a procedure.

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 8.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
 - D 301 Test Methods for Soluble Cellulose Nitrate²
 - D 1193 Specification for Reagent Water³
- 2.2 *Military Standards:*⁴
 - MIL-STD M-244A
 - MIL-STD 286 208.1.3

3. Summary of Test Method

3.1 A weighed specimen of nitrocellulose is dissolved in sulfuric acid and titrated automatically with ferrous sulfate. The nitrogen content of the specimen is calculated using the equivalence factor of the ferrous sulfate.

4. Significance and Use

4.1 This test method provides a simpler means for measuring the nitrogen content of nitrocellulose than the nitrometer described in Test Method D 301. Under controlled conditions, the procedure described is capable of results equivalent to those obtained by the nitrometer.

5. Interferences

5.1 The presence of moisture (or other volatile compo-

nents) in the specimen will affect results. It is recommended that only thoroughly dry specimens be used.

5.2 Temperature rise must be controlled during the titration. The cooling bath provides that control. However, if the rate of titrant addition is too fast, temperature may rise out of control. Results may then be erratic. Adherence to the procedure will avoid temperature excursions. For optimum system efficiency, room temperature should be maintained at $23 \pm 2^\circ\text{C}$.

5.3 The strength of the sulfuric acid used to dissolve the specimen is very important. Too low an acid strength slows the rate of solution which, in turn, causes titrations to be abnormally slow. Results then become erratic.

6. Apparatus

- 6.1 *Acid Bottle Safety Dispenser.*
- 6.2 *Fisher Titralyzer II Titration System*, or equivalent, with 25 mL amber buret:
 - 6.2.1 *Electrode*, platinum.
 - 6.2.2 *Electrode*, glass.
- 6.3 *Desiccator*, with drying agent.
- 6.4 *Weighing bottles*, 12-mL capacity, aluminum (preferred) or glass.
- 6.5 *Analytical Balance*, accurate to ± 0.1 mg.
- 6.6 *Ovens*— 135°C , for drying standards, and 100°C , for drying specimens, having unexposed heating elements and the door latch removed.
- 6.7 *Circulating Unit*, for chilled water, $5 \pm 2^\circ\text{C}$.
- 6.8 *Blender*, with 8-oz blender jar.

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.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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

7.3 *Sulfuric Acid* (H_2SO_4) 95 to 98 %.

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 D01.36 on Cellulose and Cellulose Derivatives.

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 06.03 and 11.01.

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

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."

7.4 *Ferrous Sulfate Solution*—Dissolve 350 g of ferrous sulfate crystals (FeSO_4) in 1000 mL of distilled water. Add 1000 mL of 1+1 H_2SO_4 solution. Makes 2 L.

7.5 *Potassium Nitrate (KNO_3) Standard* or an equivalent nitrate primary standard.

7.6 *Nitrocellulose Standard*—A sample with a known nitrogen value or other known organic nitrate.

8. Hazards

8.1 Since the sample of nitrocellulose must be dried, it is imperative that care be exercised in storage, handling, and disposal. Dry nitrocellulose is extremely flammable. Refer to the Procedure section for Drying Samples of Methods D 301.

8.2 Strong sulfuric acid used as the solvent for the specimen can burn the skin. The ferrous sulfate titrant is also strongly acidic. Take proper precautions to protect the operator and the equipment.

8.3 To prevent burns from acid dripping off the electrodes and dispenser tip, always wipe the electrodes and dispenser tip with a tissue before reaching under them to retrieve a beaker.

9. Preparation of Apparatus

9.1 If the system has been down for at least 8 h, purge the system.

10. Calibration and Standardization

10.1 Weigh a sample bottle containing 0.5000 ± 0.05 g of KNO_3 that has been dried in a 135°C oven for a minimum of 4 h and stored in a desiccator. If KNO_3 has been out of the oven for 4 h or more, redry in a 135°C oven for a minimum of 1 h. (Nitrocellulose specimens are dried in a 100°C oven for a minimum of 1 h. If out of the oven more than 2 h, redry for $\frac{1}{2}$ h.) Turn on the pump of the chilled water circulator to start water flowing through the cooling bath.

10.2 Place the magnetic stirring bar into a dry 250-mL beaker and fill the beaker with 150 mL of H_2SO_4 ($20 \pm 2^\circ\text{C}$).

10.3 Place the beaker in the cooling bath on the stirrer unit. Start the stirrer and adjust the speed for a *slight* vortex. Too vigorous a vortex can cause the nitrocellulose to splash onto the sides of the beaker.

10.4 With the stirrer operating and the electrodes up in the air away from the acid, slowly pour the specimen into the vortex of the swirl. Be careful not to touch the dispensing tip or electrodes with the weighing bottle. Reweigh the weighing bottle to find the weight of the standard by difference.

10.5 Allow most of the KNO_3 to dissolve. Visually inspect the solution until no more chunks or chips of KNO_3 remain. The solution may be cloudy.

10.6 Lower the electrodes and the dispensing tip into the H_2SO_4 .

10.7 Set the controls to the desired settings.

10.8 As the KNO_3 dissolves and HNO_3 is formed by the reaction of H_2SO_4 and the specimen, a millivolt potential change is evident by a rising recorder pen.

10.8.1 When the pen shows a leveling off, it is an indication that the majority of the KNO_3 is in solution. Visually inspect the solution to verify the fact that chips are no longer present.

10.9 Set the buret control to a rate of about 5 mL/min. Allow 10 ± 0.1 mL to dispense at this rate.

10.10 Stop the buret, switch the dispense-rate switch to automatic, and restart. Allow the titration to proceed automatically to the end point.

10.11 At the completion of the automatic titration, immediately record the millilitre reading. Switch the electrode setting to **STANDBY**.

10.12 Raise the electrodes from the acid and allow most of the acid to drip into the titration beaker. Carefully wipe by dabbing the electrodes and dispensing tip with a tissue. (Precaution—See Section 8.) Discard the tissue into a container of water. **DO NOT** allow acid to drip into the circulating-bath water.

10.13 Remove the completed titration beaker from the cooling bath.

10.14 Lower the electrodes and pipet into a beaker of clean H_2SO_4 for soaking. Make sure the acid is not contaminated with FeSO_4 . (It may bleed from the dispenser tip giving a reddish tint to the acid.) Replace with clean H_2SO_4 if this occurs.

NOTE—It is important to soak the electrodes in between each analysis and while the titrators are not in use. Cleanliness of equipment is of the utmost importance in this method of analysis.

10.15 Calculate the nitrogen equivalence factor F for the standard KNO_3 as follows:

$$F = (A \times 13.855)/B$$

where:

A = weight of KNO_3 , g.

13.855 = nitrogen equivalence of nitrogen in KNO_3 or in the standard material used, and

B = amount of FeSO_4 used to titrate KNO_3 , mL.

11. Specimen Preparation

11.1 Cutting:

11.1.1 Use a sample size of approximately 2 heaping tablespoons of wet nitrocellulose.

11.1.2 For high viscosity types, place the sample into a small (8-oz) plastic blender jar. Fill to the fill line with tap water, and screw on the cap with a 4-blade cutter unit inside.

11.1.3 Place on the blender base and blend for 7 min at high speed.

11.1.4 For 1+-% nitrogen and low viscosity types, grind in a tissue disintegrator using a 16-oz glass jar about $\frac{3}{4}$ full of tap water.

11.1.5 Grind each sample at high speed for their respective times as follows:

Hercules Designation	Time, min
SS, all	5
RS $\frac{1}{2}$	4
RS $\frac{1}{4}$	4
RS $\frac{1}{8}$	3
AS, all	3

11.2 At the completion of cutting, remove the sample container and draw off the excess water from the nitrocellulose by filtering on a Büchner funnel through a circular filter paper.

11.3 Dry the material in accordance with the paragraph on small quantities in the Procedure section for Drying Samples of Methods D 301.

11.4 After drying, weigh 3 replicate specimens of 0.4500 ± 0.0075 g each into a weighing can or bottle.

TABLE 1 Suggested Settings for Fisher Titrlyzer II

	EP-1	Select	EP-1 Setting, mV	Proposed Band ^A	Minimum Delivery ^A
Standardizations with KNO ₃	-mV	EP-1	350	80	100
Analysis of nitro-cellulose	-mV	EP-1	200	80	100

^A These are approximations only. Settings can vary between instruments.

11.5 It is desirable to analyze the specimens as soon as they are taken out of the oven. Allow time for cooling in a desiccator before weighing. If specimens are out of the oven for more than 2 h, redry for ½ h at 100°C.

12. Procedure

12.1 Prepare specimens in accordance with Section 11.

12.2 Repeat 10.1 through 10.15, using the settings for the specimen given in Table 1.

12.3 Dispose of any excess H₂SO₄ standard down the sink and rinse with large quantities of water.

13. Calculation

13.1 Calculate the percent of nitrogen *N* as follows:

$$N = (C \times F)/D$$

where:

C = FeSO₄ to titrate specimen, mL,

F = equivalence factor from 10.15, and

D = weight of specimen, g.

14. Precision and Bias⁶

14.1 Tests by two laboratories on two samples gave results equivalent to the nitrometer in precision and bias. No more extensive interlaboratory testing has been undertaken as yet.

⁶ Supporting data available from ASTM Headquarters. Request RR: D01-1056.

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Standard Test Method for Determining the Unreacted Monomer Content of Latexes Using Capillary Column Gas Chromatography¹

This standard is issued under the fixed designation D 4827; the number immediately following the designation indicates the year of original adoption 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 for the determination of the unreacted monomer content of acrylic latexes. Monomers that have been successfully determined by this procedure include *n*-butyl methacrylate, *n*-butyl acrylate, styrene, and methyl methacrylate. The determination of other monomers has not been evaluated, but this test method is believed to be applicable. The established working range of this test method is from 100 to 1000 $\mu\text{g/g}$, but there is no reason to believe it will not work outside of this range, provided that appropriate dilutions and adjustments in specimen size are made.

1.2 The unreacted monomer in acrylic latexes is expected to change with time and environmental factors. This time dependence of the determination may be seen as an artificially large deviation of results, making the test method mostly applicable for in-house quality control, where sampling and analysis conditions can be better controlled.

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 7.*

2. Referenced Documents

- 2.1 *ASTM Standard:*
D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 A suitable aliquot of the latex is internally standardized with isobutyl acrylate, diluted with water, and then injected onto a capillary gas chromatographic column containing a stationary phase that separates the internal standard and monomers in question from each other and from other volatile compounds.

4. Significance and Use

4.1 Excessive amounts of unreacted monomer may cause concerns relating to toxicity and odor. This test method is designed to measure the unreacted monomer content of latexes. The results may be used to monitor the extent of polymerization during manufacture, as well as to establish

maximum unreacted monomer content for regulatory purposes.

5. Apparatus

5.1 *Gas Chromatograph*—Any gas-liquid chromatographic instrument having a flame ionization detector and linear temperature programming and a capillary column inlet capable of split operation. The split liner should be constructed of glass and be replaced or cleaned as needed. On-column injection into a wide bore capillary column was not evaluated but is expected to also be satisfactory for this procedure.

5.2 *Column*—30-m by 0.25-mm inside diameter fused silica coated with a 1 μm thick film of a phenyl methyl silicone polymer. A bonded phase is preferred. Other columns having equivalent or superior performance may also be used.

5.3 *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 level of $\pm 0.03\%$ of full scale.

5.4 *Liquid Charging Devices*—A microsyringe, 1.0- μL capacity, or an automatic liquid sampling device using a suitable syringe and appropriate change in split ratio.

5.5 *Dropper Pipettes*, glass, disposable.

5.6 *Vials*, approximately 7 mL capacity, with caps. Open top screw-cap vials fitted with PTFE/silicone septa are preferred.

5.7 *Autosampler Vials*, 2 mL capacity (optional).

5.8 *Analytical Balance*, accurate to 0.1 mg.

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 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 II of Specification D 1193.

6.3 *Carrier Gas*—Helium of 99.995 % or higher purity. High purity nitrogen may also 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 Paints and Paint Materials.

Current edition approved May 27, 1988. Published November 1988.

² *Annual Book of ASTM Standards*, Vols 06.03 and 11.01.

³ "Reagent Chemicals, American Chemical Society Specifications" by Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the Am. Chem. Soc., see "Reagent Chemicals and Standards" by Joseph Rosen, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

6.4 Acetone.

6.5 Isobutyl Acrylate (internal standard), 99+ % pure.

NOTE 1—Isobutyl acrylate was found to be a suitable internal standard, but any other monomer not found in the sample may be substituted. The internal standard chosen should yield a clear chromatographic separation, and should be free of interferences.

6.6 Monomers of Interest, 99+ % pure.

7. Hazards

7.1 Acrylic and methacrylic monomers are considered hazardous. All sample preparations should be done in a well ventilated area, such as a fume hood.

8. Preparation of Apparatus

8.1 *Column Conditioning*—Attach one end of the column to the inlet side of the instrument leaving the exit end of the column disconnected. This prevents the contamination of the detector due to column bleed. Set the helium flow rate at 0.5 mL/min (approximately equivalent to a linear velocity of 20 cm/s) and purge the column at 220°C for 1 h.

8.2 After conditioning, connect the exit end of the column to the detector 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 baseline.

8.3 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 rate to a constant value.

9. Calibration

9.1 Determine the retention time of each component expected to be present by injecting small amounts either separately or in known mixtures. Retention times should be determined each day that the test method is used.

9.2 *Standardization*—Determine in duplicate the relative response of the monomers of interest to the isobutyl acrylate internal standard as follows:

9.2.1 Weigh to within 0.1 mg about 0.05 g of isobutyl acrylate and each monomer of interest into a vial (see 5.6).

Weigh approximately 5 g of acetone into the vial and mix well.

9.2.2 Weigh approximately 0.05 g of the solution prepared in 9.2.1 into another vial, add approximately 5 g of acetone, and mix well.

9.2.3 Inject a 0.5-μL aliquot of the solution from 9.2.2 onto the column and record the chromatogram. The elution order for acetone and each of the monomers using the conditions given in Table 1 is shown in Fig. 1.

9.2.4 Measure the peak areas of the individual components and calculate the relative response factor, *RF*, for the monomers of interest as follows:

$$RF = (W_1 \times A_s) / (W_s \times A_1)$$

where:

RF = relative response factor for each monomer,

*A*₁ = peak area produced by the monomer,

*A*_s = peak area produced by the internal standard,

*W*₁ = weight of monomer used for calibration (see 9.2.1), g, and

*W*_s = weight of internal standard (see 9.2.1), g.

10. Procedure

10.1 If the composition of the latex is not known or if the approximate level of monomers in the latex is not known, a preliminary analysis must be performed by diluting approximately 0.5 g of latex with approximately 5 g of water and injecting a 0.5 μL aliquot into the chromatographic column. Using the same conditions as for standardization, record the peaks of all components at attenuation settings that provide maximum peak heights. Use the relative retention times to identify the monomers present. If the specimen has a component eluting at the same retention time as isobutyl acrylate, choose a different internal standard (see Note 1).

10.2 Prepare a dilute solution of the internal standard by weighing to 0.1 mg about 0.05 g of isobutyl acrylate and 5 g of acetone into a septum vial. Take care to minimize losses due to evaporation. Prepare this solution fresh each day that the test method is used.

TABLE 1 Instrument Conditions	
Detector	flame ionization
Airflow, mL/min	240
Hydrogen flow, mL/min	30
Column: ^a	
Length, m	30
Inside diameter, mm	0.25
Film thickness, μm	1
Carrier gas	helium
Flow rate	0.5 mL/min
Temperatures:	
Injection port, °C	220
Detector block, °C	250
Column:	
Initial, °C	60
Hold time, min	4
Program rate, °C/min	8
Final, °C	200 (or higher as needed)
Final hold, min	10 (or longer)
Injection volume, μL	0.5
Split ratio	20:1

^a Cross-linked 50 % phenyl 50 % methyl silicone. A column of equivalent or better performance may also be used.

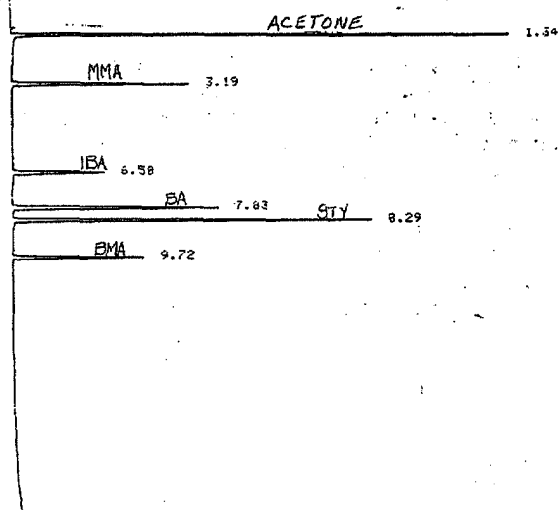


FIG. 1 Typical Chromatogram

10.3 Weigh to within 0.1 mg an appropriate amount of sample into a septum vial using Table 2 as a guide to specimen size. Also weigh to within 0.1 mg 50 mg of the dilute solution prepared in 10.2 into the vial. Add about 3 to 5 g of water or acetone. Shake the vials on a wrist action shaker or other suitable device for 15 min.

NOTE 2—The viscosity of a number of latexes increases upon the addition of an organic solvent. If acetone (or another organic solvent) is found to be compatible with the specimen, it should be used as the diluent instead of water. It should be kept in mind that some organic solvents may interfere with the chromatographic separation.

10.4 Inject 0.5 μ L of the solution prepared in 10.3 and record the chromatogram using the conditions given in 10.1. Measure the peak areas (Note 3) of the internal standard and relevant monomers, multiplying each area by the appropriate attenuation factor to express the peak areas on a common basis.

NOTE 3—Peak areas may be determined by any method that meets the precision requirements given in Section 12. Electronic integration is recommended for best results.

10.5 Repeat 10.3 and 10.4 and calculate the mean values.

11. Calculations

11.1 Calculate the weight of the internal standard present in the diluted specimen (see 10.3) as follows:

$$W_4 = (W_5/W_6)W_7$$

where:

W_4 = weight of internal standard in diluted specimen prepared in 10.3, g,

W_5 = weight of internal standard used to prepare solution in 10.2, g,

W_6 = weight of acetone plus weight of internal standard used to prepare solution in 10.2, g, and

W_7 = weight of the dilute internal standard solution in 10.2 added to the specimen in 10.3, g.

TABLE 2 Suggested Dilutions

NOTE—This table is to be used only as a guide. If the monomer concentrations are outside the range given, appropriate adjustments must be made in terms of specimen size, dilution, and amount of internal standard added.

Level of Unreacted Monomer Expected, g/g	Specimen Size, g	Diluent, g
250	2	3
500	1	4
750	0.7	4.3
1000	0.5	4.5

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11.2 Calculate the concentration of each monomer present in the latex specimen from the results obtained in 10.5 as follows:

$$C = [(A_3 \times W_4 \times RF)/(W_8 \times A_4)] \times 10^6$$

where:

A_3 = peak area produced by the monomer,

A_4 = peak area produced by the internal standard,

C = concentration of unreacted monomer, g/g,

RF = relative response factor for each monomer calculated in 9.2.4,

W_4 = weight of internal standard calculated in 11.1, g, and

W_8 = weight of specimen prepared in 10.3, g.

12. Precision and Bias⁴

12.1 In an interlaboratory study of this test method by five laboratories using four specimens, each containing variable concentrations of four different monomers, the following duplicates, repeatability, and reproducibility coefficients of variation were obtained for each monomer:

Monomer	Duplicates	Precision, % Repeatability (Single Laboratory)	Reproducibility (Between Laboratories)
Methyl methacrylate (MMA)	11.7	34.3	86.2
n-Butyl acrylate (BA)	13.3	21.7	49.2
Butyl methacrylate (BMA)	14.5	33.5	96.5
Styrene (STY)	11.3	30.5	71.5

NOTE 4—Variation in results may be due to the changing composition of the specimens used for the study. This precision statement should only be used as a guide since it only represents the magnitude of variation that is possible, which will vary with time depending on the latex and the particular monomers being determined.

12.2 Bias cannot be determined for this test method.

13. Index Terms

13.1 This test method is indexed under the following terms: chromatography—gas; chromatography—gas (capillary column); latex paints; latex vehicles; monomer (unreacted); unreacted monomer content.

⁴ Supporting data is available from ASTM Headquarters. Request RR: D01-1059.



Standard Test Method for Filter-Retained Solids Content of Polymer Latexes¹

This standard is issued under the fixed designation D 5097; the number immediately following the designation indicates the year of original adoption 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 filter-retained solids (grit) content of polymeric latex vehicles, that is, material present in a latex specimen that is retained on a 200-mesh screen.

1.2 The values stated in SI units are to be regarded as 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.*

2. Referenced Documents

2.1 ASTM Standard:

E 11 Specification for Wire-Cloth Sieves for Testing Purposes²

E 145 Specification for Gravity-Convection and Forced-Convection Ovens²

3. Summary of Test Method

3.1 A specified weight of a polymer latex, first filtered through a 20-mesh screen to remove skins and large agglomerates, is water-washed through a preweighed 200-mesh stainless screen. The dried residue on the screen, defined as the filter-retained solids, is expressed as parts per million (ppm) of the polymer latex.

NOTE 1—Other screens or sample sizes, or both, may be used upon agreement of producer and user. Precision may vary with differing mesh size and sample weight.

4. Significance and Use

4.1 The amount of excessive oversized particulate matter present in polymer latexes can detract from the appearance properties of applied formulated coatings and may reduce filtration rates or clog filters in processing or application processes.

4.2 This test method may be useful as a quality control test for latex manufacture and for product characterization.

5. Apparatus

5.1 *Analytical Balance*, capable of weighing to 0.1 mg.

5.2 *Balance*, capacity of 500 g, capable of weighing to 0.1 g.

5.3 *Dessicator*.

5.4 *Forced-Ventilation Oven*, Type II B conforming to Specification E 145.

5.5 *Forceps*.

5.6 *Spectra Mesh Filtration Unit*,³ (90-mm size).

5.7 *Graduated Cyclinder*, 500-mL.

5.8 *Polyethylene Water Wash Bottle*, 500-mL.

5.9 *Two Beakers*, 800-mL.

5.10 *Screen*, stainless steel, 90-mm diameter, 200-mesh (as defined in Specification E 11).

5.11 *Screen*, cloth or stainless steel, 20-mesh (as defined in Specification E 11).

7. Procedure

7.1 Weigh the clean, dry, 200-mesh screen to the nearest 0.1 mg. Record the weight, *A*.

7.2 Place the screen into the filtration apparatus.

NOTE 2—The screen should be held firmly in place while screwing down the filter chamber to ensure that the screen seats smoothly on the plastic ring.

7.3 Weigh 500 g of water into the 800-mL beaker and set aside.

7.4 Hand-stir the polymer latex sample for about 15 s using a wide blade spatula to ensure adequate mixing, taking care not to beat in air. Filter about 150 g of the latex sample through a standard 20-mesh cloth or stainless-steel sieve to remove skins or large pieces of coagulum.

7.5 Stir the filtered sample gently with the spatula. Immediately weigh out by difference about 50 g, weighed to 0.1 g, of the latex into an 800-mL beaker. Record the latex specimen weight, *B*.

7.6 Transfer the 500 g of water from 7.3 to the beaker with the specimen and stir the mixture with the spatula.

NOTE 3—If the latex coagulates on water dilution, the test is not applicable.

7.7 Slowly pour the mixture onto the 200-mesh screen, water-washing the sample from the beaker onto the screen with the polyethylene wash bottle.

7.8 Wash the residue on the screen with 1000 g of water, using the wash bottle to wash down the sides of the filtration apparatus and to move the solid toward the screen center.

7.9 *Specimen Drying*:

7.9.1 Carefully remove the screen from the apparatus using forceps, place the screen in the oven and dry for 1 h at $110 \pm 1^\circ\text{C}$.

7.9.2 Remove the screen from the oven using the forceps and allow the screen to cool for 10 min in a dessicator.

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 July 27, 1990. Published September 1990.

² Annual Book of ASTM Standards, Vol 14.02.

³ Available from Fisher Scientific, catalog number 08-670-173.

7.9.3 Weigh the screen plus dried solids to the nearest 0.1 mg. Record the weight, C .

7.10 Repeat 7.1 to 7.9.3 using a duplicate sample of the latex.

8. Calculation

8.1 Calculate the parts per million of filter retained solid content, S , in the analysis samples as follows:

$$S = \frac{(C - A) 10^6}{B}$$

where:

C = mass of screen and solid residue, g,

A = mass of screen, g, and

B = mass of latex sample, g.

8.2 Calculate the average filter-retained solids content from the duplicate analysis.

9. Report

9.1 Report the following information:

9.1.1 The filter-retained solids content to the nearest 10 ppm as the average of the two determinations and

9.1.2 The mesh size and sample weight used, if other than 200 mesh and 50 g.

10. Precision and Bias

10.1 The precision of this test method will be determined.

10.2 Since there is no accepted reference material for determining the bias of the procedure for measuring filter-retained solids no statement on bias is being made.

11. Keywords

11.1 filter-retained solids; grit; polymer latexes

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Standard Practice for Laboratory Preparation of Gelled Vehicle Samples Using a Microwave Oven¹

This standard is issued under the fixed designation D 5166; the number immediately following the designation indicates the 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 outlines a procedure for preparing gelled vehicle samples using a microwave oven.

1.2 The test samples can be used for characterizing the gelability or reactivity of resins, gelling agents, and vehicles used in the manufacture of oil based printing inks, or both.

1.3 Evaluation of the gelled vehicles may, depending upon preference, be either visual or instrumental.

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:

D 154 Guide for Testing Varnishes²

D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method³

D 1725 Test Method for Viscosity of Resin Solutions⁴

D 4040 Test Method for Viscosity of Printing Inks and Vehicles by Falling Rod Viscometer⁵

E 1 Specification for ASTM Thermometers⁶

3. Terminology

3.1 Description of Terms Specific to This Standard:

3.1.1 *gel*—Any resin solution, or more complex blend of resins and alkyds that has been cooked or reacted with a gelling or cross-linking agent to build molecular weight and that exhibits a pseudoplastic rheology. (Also called “gelled vehicle.”)

3.1.2 *gelling agent*—in the context of ink resins and vehicles, gelling agents or gellants are typically organo-aluminum compounds that react with carboxylic acid and hydroxyl groups present on the backbone of resins and alkyds to form cross-linked networks. (Also called “gellant.”)

3.1.3 *gel length*—References to long, medium, and short gel length refer to the length of a string of gelled vehicle observed when pulling apart a small sample of vehicle with a spatula (that is, a long gel is very fluid and forms a “long”

filament; a short gel has little flow and forms a “short” filament).

3.1.4 *pregel*—The resin solution or vehicle components comprising the vehicle prior to the addition of gelling agent and viscosity adjusting solvent. (Also called “pregel vehicle.”)

4. Summary of Practice

4.1 Prepare a pregel resin solution or vehicle.

4.2 Separate the pregel vehicle into five or more aliquots to which various levels of gellant are added. Alternately, various gellants can be added at a given concentration to compare their effectiveness.

4.3 Place the test samples on a turntable in the microwave oven and heat the samples at full power until the desired test temperature is attained.

4.4 After the prescribed waiting period, test and rate samples for gelability.

5. Significance and Use

5.1 This practice provides a means of preparing a number of gel vehicle samples with minimum use of materials and time. It provides a means of quickly characterizing and comparing the gelability or reactivity of resins, vehicles, and gelling agents.

6. Apparatus

6.1 *Mercury Thermometer*, capable of reading from 0 to 250°C and conforming to Specification E 1.

6.2 *Jars*, (40 mm in diameter by 83 mm in height), 70 cm³ with nonmetallic screw-on lids.

6.3 *Microwave Oven*, common 750-watt.⁷

6.4 *Microwave Oven Carousel*, if not contained in the microwave oven.

6.5 *Glass Dish*, 15.2 cm in diameter.

6.6 *Balance or Scale*, capable of weighing to ± 0.1 g accuracy.

6.7 *Ink Knife or Rigid Spatula*.

6.8 *Rheology Testing Apparatus*, at the discretion of the user.

7. Reagents and Materials

7.1 Nonvolatile materials including resins, alkyds, gellants, etc consistent with those used in the manufacture of lithographic ink vehicles, for example, aromatic hydrocarbon resins, modified rosin ester resins, long oil linseed isophthalic

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 D01.37 on Ink Vehicles.

Current edition approved Sept. 15, 1991. Published November 1991.

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 06.02.

⁵ *Annual Book of ASTM Standards*, Vols 06.01 and 06.02.

⁶ *Annual Book of ASTM Standards*, Vol 14.03.

⁷ Microwave oven, such as the Amana Radarrange Model RR-9TA, Amana Refrigeration, Inc., is available from most appliance stores, and has been found suitable for this purpose.

alkyds, and aluminum compounds.

7.2 Solvents typically used in the manufacture of lithographic ink vehicles, for example, hydrocarbon petroleum distillate with carbon numbers in the range of C12 to C16 and initial boiling point above 150°C.

8. Procedure

8.1 Place 45 g (or the calculated weight) of previously prepared pregel vehicle into each of five or more 30-cm³ bottles.

NOTE 1—Any vehicle, even a very complex formulation, without the gellant added may be used as the pregel vehicle portion for the microwave oven gelability process.

8.1.1 In the absence of a vehicle formula agreed upon between user and seller, the following test vehicle formula is suggested.^{8,9}

	Parts
Pregel Vehicle	
Resin	35
Alkaline refined linseed oil (ARLO)	30
C12-C16 hydrocarbon petroleum distillate	25
	90
Gellant/Solvent Addition	
33.3% oxyaluminum octoate/66.7% C12-C16 hydrocarbon petroleum distillate	10
	100

8.2 Estimate the amount of gellant required to make an acceptable gel vehicle. Add gellant solution (between 30 and 50 % gellant in major vehicle solvent recommended) in 1 % increments plus additional solvent to adjust the vehicle to a 100 % formula. If the amount of gellant required can not be estimated, use a larger range of gellant concentration.

8.2.1 If using the pregel formula as just recommended, the required amount of gellant can be estimated from Table 1.

8.2.2 A sample containing no (0 %) gellant solution is always prepared as the standard for each resin.

8.3 Mix the added liquids into the pregel very well using an ink knife or rigid spatula.

8.4 Place the five sample bottles, without tops, into the 15.2-cm glass dish and place on the carousel in the microwave oven.

8.5 Turn on the carousel and ensure that it is turning before starting the heating cycle. Turn on the light in the oven to observe the process.

8.6 Set the timer on the oven for "5.00" min and start the cooking cycle.

8.7 Upon completion of the cooking cycle, check the temperature of the samples to determine whether a temperature of 105 to 110°C was obtained. If not, continue the heating cycle in 1-min intervals until the samples have reached this temperature.

8.8 Remove the samples from the microwave oven, and allow to cool.

8.9 Seal cooled samples with jar lids.

⁸ This formula works best with higher molecular weight ink resins (for example, phenolic modified rosin esters). It is suggested that the resin solids be increased for low molecular weight resins (for example, simple rosin esters, modified hydrocarbons, etc).

⁹ When a resin is insoluble, reformulate replacing 5 parts of C12-C16 hydrocarbon petroleum distillate with TXIB (2,2,4-trimethyl-1,3-pentanediol, diisobutylate) or TDA (tridecyl alcohol). Caution: TDA may hinder the reactivity of the resin system tested.

TABLE 1 Microwave Gelability Test, Weights and Concentrations for 50-g Samples

Gellant Solution, %	Actual Gellant Concentration, %	Weight Gellant Solution, g ^A (at 33.3 % Gellant in Ink Oil)	Weight Ink Oil, g ^A
0	0.00	0.0	5.0
1	0.33	0.5	4.5
2	0.67	1.0	4.0
3	1.00	1.5	3.5
4	1.33	2.0	3.0
5	1.67	2.5	2.5
6	2.00	3.0	2.0
7	2.33	3.5	1.5
8	2.67	4.0	1.0
9	3.00	4.5	0.5
10	3.33	5.0	0.0

^A Weight added to 45-g pregel sample.

NOTE 2—If more than one set of samples is to be run, allow the microwave oven to cool between runs. Heat is retained in the oven and if a second set of samples is run immediately it will be subjected to more heat than the first set.

9. Evaluation

9.1 Samples should be tested immediately after preparation and after 24 h to determine the effects of post gelation, if any.

9.2 Guide D 154 outlines many methods applicable to the testing of varnishes. It is an excellent guide for those looking for specific areas of evaluation.

9.3 Lightly gelled resin solutions and vehicles can be tested for changes in viscosity by using either Test Method D 1545 or D 1725. These test methods both use Gardner bubble tubes to determine the viscosity of liquids.

9.4 Another test method for testing gelled ink vehicles is Test Method D 4040. This method utilizes a falling-rod viscometer and can be used to determine the non-Newtonian nature of vehicles. Other viscometers used for determining the rheology of gelled vehicles include variable shear rate viscometers, and cone and plate viscometers.

9.5 For the trained analyst the occurrence of gelation is evident and gels can be rated visually (for example, long, medium, medium short, short gel body).

10. Report

10.1 Report the following information:

10.1.1 Sample identification,

10.1.2 Test run,

10.1.3 Brief summary of procedure,

10.1.4 Conditions of test procedure, and

10.1.5 Results of the analyses made. In the case of falling rod viscometer determinations, the following data could be obtained using Test Method D 4040 (see also the Apparatus Section of this test method):

10.1.5.1 Apparent viscosity at 2500 at 25°C, s,

10.1.5.2 Pseudo-Bingham Yield value at 25°C,

10.1.5.3 Shortness ratio (yield value/viscosity), and

10.1.5.4 Slope (non-Newtonian parameter).

NOTE 3—For a measurement of relative resin, vehicle, or gellant reactivity, lots of rheology versus gellant concentration (see Fig. 1) provide an excellent comparison.

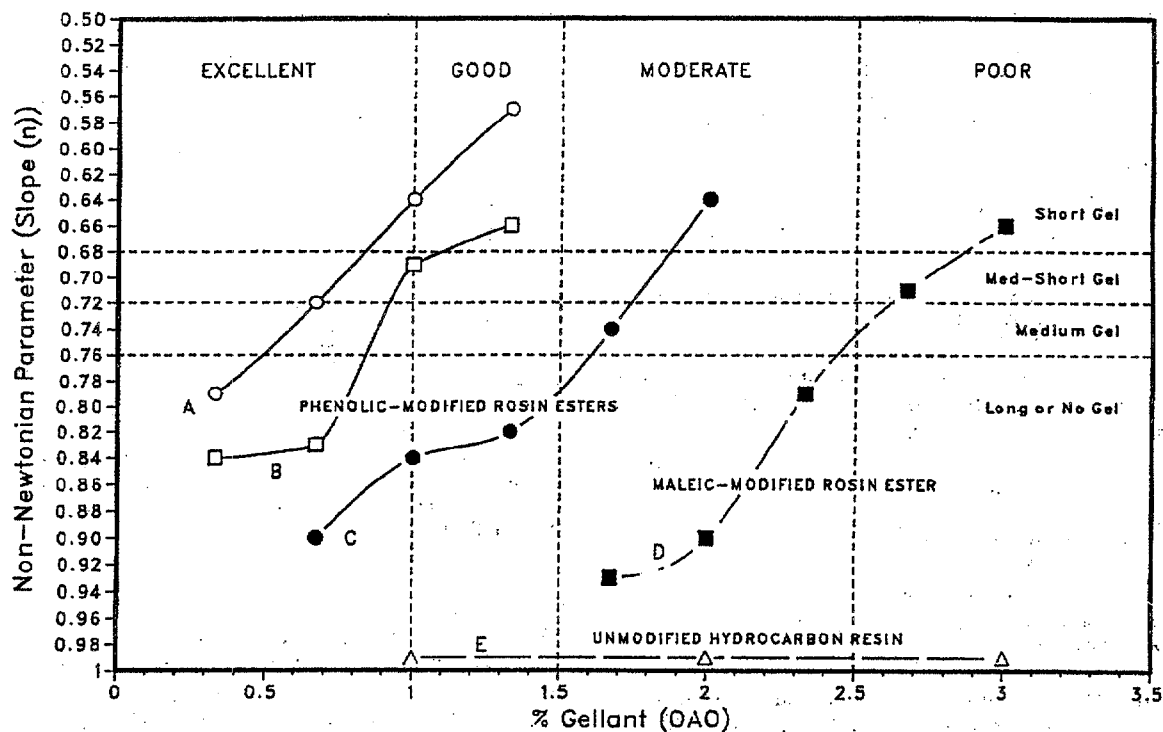


FIG. 1 Determination of Lithographic Ink Resin Gelability

NOTE 4—Falling rod viscosity slope (non-Newtonian parameter), shortness ratio, and visual degree of shortness, if used properly, are all suitable measures of rheology.

11. Keywords

11.1 gel; gelability; gelled varnish; gelled vehicle; microwave oven; non-Newtonian fluid; pregel; pregel varnish; pregel vehicle; shortness ratio; slope, yield value

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RELATED MATERIAL

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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 alphanumeric 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. xxiv and 527
A complete Subject Index begins on p. 561

PAINT AND RELATED COATINGS—AS LIQUIDS

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D 4302 - 90
D 5067 - 90[†]

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Artists' Paints: Oil, Resin-Oil, and Alkyd Paints
Artists' Watercolor Paints

Physical Tests

General Test Methods

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D 4399 - 90
§D 1210 - 79 (1988)[‡]
D 1316 - 87
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§D 185 - 84 (1989)[‡]
D 5062 - 90[‡]
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Fineness of Grind of Printing Inks by the NPRI Grindometer
Freeze-Thaw Resistance of Water-Borne Coatings
Freeze-Thaw Stability of Multicolor Lacquers
Low-Temperature Coalescence of Latex Paint Films
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D 4707 - 87

Brush Drag of Latex Paints, Comparison of
Flow Ratings of Organic Coatings Using the Shell Flow Comparator (Discontinued 1992†)
Leveling Characteristics of Paints by Draw-Down Method (Discontinued 1990†)
Leveling of Paints by Draw-Down Method
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Slump of Face Glazing and Bedding Compounds on Metal Sash
Spatter Resistance to Roller Application, Measuring

Practice for:

D 4941 - 89

Preparing Drawdowns of Artists' Paste Paints

§ 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.

LIST BY SUBJECTS, VOLUME 06.01

Color and Clarity of Liquids

Test Methods for:

§D 1209 - 84 (1988)⁶¹
 §D 1544 - 80 (1989)⁶¹
 D 4838 - 88
 D 2090 - 88

Color of Clear Liquids (Platinum-Cobalt Scale)
 Color of Transparent Liquids (Gardner Color Scale)
 Tinting Strength, Relative, of Chromatic Paints, Determining
 Clarity and Cleanliness of Paint and Ink Liquids (see Vols 06.02, 06.03)

Density, Specific Gravity, and Weight per Gallon

Test Method for:

§D 1475 - 90

Density of Paint, Varnish, Lacquer, and Related Products

Consistency and Viscosity

Test Methods for:

§D 562 - 81 (1990)⁶¹
 D 4359 - 90
 §D 2196 - 86 (1991)⁶¹
 D 4212 - 88
 D 4287 - 88
 §D 1200 - 88
 D 4040 - 91
 §D 1545 - 89

Consistency of Paints Using the Stormer Viscometer
 Determining Whether a Material is a Liquid or a Solid
 Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer
 Viscosity by Dip-Type Cups
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D 960 - 79 (1988)	Castor Oil, Raw
D 1841 - 63 (1988) ^{§1}	Coconut Fatty Acids, Distilled
D 1842 - 63 (1988)	Corn Fatty Acids, Distilled
D 1843 - 63 (1988) ^{§1}	Cottonseed Fatty Acids, Fractionated and Distilled
D 1538 - 60 (1988) ^{§1}	Linseed Fatty Acids, Distilled
D 260 - 86 (1990)	Linseed Oil, Boiled
D 234 - 82 (1991) ^{§1}	Linseed Oil, Raw
D 601 - 87 (1991) ^{§1}	Oiticica Oil (Permanently Liquid)
D 1392 - 87	Safflower Oil
D 1537 - 60 (1988) ^{§1}	Soybean Fatty Acids, Distilled
D 124 - 88	Soybean Oil, Degummed
D 1462 - 87	Soybean Oil, Refined
D 3169 - 89	Sunflower Oil
D 1984 - 69 (1988)	Tall Oil Fatty Acids
D 12 - 88	Tung Oil, Raw

Test Methods for:

D 1950 - 86	Acetone Tolerance of Heat-Bodied Drying Oils
D 1980 - 87 (1991)	Acid Value of Fatty Acids
§D 1951 - 86	Ash in Drying Oils and Fatty Acids
§D 1952 - 86	Break in Drying Oils, Quantitative Determination of
D 2090 - 88	Clarity and Cleanness of Paint and Ink Liquids
§D 1967 - 86	Color After Heating of Drying Oils (Heat Bleach), Measuring
D 1981 - 86 (1990)	Color After Heating of Fatty Acids, Measuring
§D 1544 - 80 (1989) ^{§1}	Color of Transparent Liquids (Gardner Color Scale)
D 1358 - 86	Diene Value, Spectrophotometric, of Dehydrated Castor Oil and Its Derivatives
§D 1983 - 90	Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters
D 3457 - 87 (1991) ^{§1}	Fatty Acid Composition by Gas-Liquid Chromatography, Preparation of Methyl Esters from Fatty Acids for Determination of
§D 2800 - 87	Fatty Acid Composition by Gas-Liquid Chromatography, Preparation of Methyl Esters from Oils for Determination of
§D 3725 - 78 (1988) ^{§1}	Fish Oil in Drying Oils and Drying Oil Fatty Acids by Gas-Liquid Chromatography, Semiquantitative Determination of
§D 93 - 90	Flash Point by Pensky-Martens Closed Tester
§D 3278 - 89	Flash Point of Liquids by Setaflash Closed-Cup Apparatus
D 1966 - 69 (1991) ^{§1}	Foots in Raw Linseed Oil (Gravimetric Method)
§D 1954 - 86	Foots in Raw Linseed Oil (Volumetric Method)
§D 1955 - 85 (1989) ^{§1}	Gel Time of Drying Oils
D 1957 - 86	Hydroxyl Value of Fatty Oils and Acids
§D 2245 - 90	Identification of Oils and Oil Acids in Solvent-Reducible Paints
§D 1959 - 85 (1989) ^{§1}	Iodine Value of Drying Oils and Fatty Acids
D 1541 - 86	Iodine Value, Total, of Drying Oils and Their Derivatives

§ 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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§D 1960 - 86 (1990)
D 1958 - 86 (1990)
D 2575 - 70 (1987)
D 1240 - 82
D 1466 - 86
§D 1962 - 85 (1989)^{ei}
D 1963 - 85 (1989)^{ei}
D 803 - 82 (1987)
D 1585 - 82
D 1982 - 85 (1989)^{ei}
§D 1964 - 85 (1989)^{ei}
§D 1965 - 87 (1991)^{ei}
§D 1545 - 89

Loss on Heating of Drying Oils
Oiticica Oil, Chloroform Insoluble Matter in
Polymerized Fatty Acids
Rosin Acids in Fatty Acids
Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes, and Related Materials
Saponification Value of Drying Oils and Fatty Acids
Specific Gravity of Drying Oils, Varnishes, Resins, and Related Materials
Tall Oil
Tall Oil Rosin, Fatty Acids Content of
Titer of Fatty Acids
Tung Oil Quality
Unsaponifiable Matter in Drying Oils and Fatty Acids
Viscosity of Transparent Liquids by Bubble Time Method

Guides for:

§D 555 - 84 (1988)^{ei}
D 1467 - 89
D 4140 - 82 (1991)^{ei}

Drying Oils, Testing
Fatty Acids Used in Protective Coatings, Testing,
Volatile and Nonvolatile Content of Driers, Drying Oils, Naval Stores, and Solvents, Determining

FATTY NITROGEN PRODUCTS

Test Methods for:

D 2076 - 64 (1987)^{ei}
D 2074 - 66 (1987)^{ei}
D 2073 - 66 (1987)^{ei}

D 2077 - 64 (1987)
D 2071 - 87 (1991)
D 2075 - 89
D 2078 - 86 (1990)
D 2080 - 64 (1987)
D 2082 - 82 (1987)
D 2079 - 82 (1987)
D 2081 - 64 (1987)
D 2083 - 66 (1987)
D 2072 - 66 (1987)^{ei}

Acid Value and Amine Value of Fatty Quaternary Ammonium Chlorides
Amine Values, Total, Primary, Secondary, and Tertiary, of Fatty Amines by Alternative Indicator Method
Amine Values, Total, Primary, Secondary, and Tertiary, of Fatty Amines, Amidoamines, and Diamines by Referee Potentiometric Method
Ash in Fatty Quaternary Ammonium Chlorides
Fatty Nitrogen Products
Iodine Value of Fatty Amines, Amidoamines, and Diamines
Iodine Value of Fatty Quaternary Ammonium Chlorides
Molecular Weight, Average, of Fatty Quaternary Ammonium Chlorides
Non-amines, Percent in Fatty Nitrogen Compounds
Nonvolatile Matter (Solids) in Fatty Quaternary Ammonium Chlorides
pH of Fatty Quaternary Ammonium Chlorides
Primary, Secondary, and Tertiary Amines, Percent in Fatty Amines
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SOLVENT SPECIFICATIONS

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D 3735 - 87

Petroleum Spirits (Mineral Spirits)
VM&P Naphthas

Terpene Solvents

§D 13 - 82 (1987)

Turpentine, Spirits of

Aromatic Hydrocarbons and Related Materials

D 836 - 84
D 835 - 85
D 2359 - 85a
D 3055 - 86
D 3734 - 91
D 4077 - 81 (1986)^{ei}
D 2827 - 88^{ei}
§D 362 - 84
D 841 - 85
D 4076 - 86
D 843 - 80 (1985)^{ei}
D 846 - 84

Benzene, Industrial Grade (Discontinued 1991)
Benzene-485, Refined (Nitration Grade)
Benzene-535, Refined
Cyclohexane 995
High-Flash Aromatic Naphthas
Isopropylbenzene (Cumene)
Styrene Monomer 996
Toluene, Industrial Grade (Discontinued 1991)
Toluene, Nitration Grade
o-Xylene 950
Xylene, Nitration Grade
Xylene, Ten-Degree (Discontinued 1991)

Alcohols and Ether Alcohols

D 319 - 90
D 330 - 89
§D 304 - 90
D 1007 - 85
D 2694 - 87
D 2696 - 87
D 4836 - 90
D 331 - 90

Amyl Alcohol (Synthetic)
2-Butoxyethanol
n-Butyl Alcohol (1-Butanol)
sec-Butyl Alcohol (2-Butanol)
Diethylene Glycol
Dipropylene Glycol (Discontinued 1992†—Replaced by Specification D 5164)
Dipropylene Glycol Monomethyl Ether
2-Ethoxyethanol

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D 2693 - 87	Ethylene Glycol
D 2636 - 91	Hexylene Glycol
§D 1719 - 90	Isobutyl Alcohol (Isobutanol)
D 770 - 90	Isopropyl Alcohol (Isopropanol)
D 3160 - 91	Isopropylbenzene (Cumene), Phenol Content
D 3128 - 89	2-Methoxyethanol
D 1152 - 89	Methanol (Methyl Alcohol)
D 2635 - 91	Methyl Isobutyl Carbinol
D 3622 - 90	<i>n</i> -Propyl Alcohol (1-Propanol)
D 2695 - 87	Propylene Glycol (Discontinued 1992†—Replaced by Specification D 5164)
D 4837 - 89	Propylene Glycol Monomethyl Ether

Ketones

§D 329 - 90	Acetone
D 2627 - 91	Diacetone Alcohol
D 2916 - 88	Isophorone
D 4360 - 90	Methyl <i>n</i> -Amyl Ketone
D 740 - 89	Methyl Ethyl Ketone
D 3729 - 84	Methyl Ethyl Ketone (99.5 % Grade) (Discontinued 1989†—Replaced by Specification D 740)
§D 2917 - 91	Methyl Isoamyl Ketone
§D 1153 - 90	Methyl Isobutyl Ketone

Esters

D 3540 - 86	Amyl Acetate, Primary, Synthetic (98 % Grade)
D 4615 - 86 [†]	<i>n</i> -Butyl Acetate (All Grades)
D 3728 - 88	2-Ethoxyethyl Acetate (99 % Grade)
D 4614 - 86	Ethyl Acetate (All Grades)
D 5137 - 90	Hexyl Acetate
§D 1718 - 86	Isobutyl Acetate (95 % Grade)
D 3131 - 88	Isopropyl Acetate (98 % Grade)
D 2634 - 86	Methyl Amyl Acetate
D 3130 - 86	<i>n</i> -Propyl Acetate (96 % Grade)
D 4835 - 89	Propylene Glycol Monomethyl Ether Acetate

SOLVENT TEST METHODS

General Methods

Test Methods for:

D 801 - 57 (1987)	Dipentene, Sampling and Testing
§D 233 - 65 (1981) [†]	Turpentine, Sampling and Testing
D 268 - 90	Volatile Solvents for Use in Paint and Related Coatings and Materials, Sampling and Testing

Chemical Tests

Test Methods for:

D 1612 - 90	Acetone in Methanol (Methyl Alcohol)
§D 1613 - 91	Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products
§D 847 - 87	Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons
§D 1614 - 91	Alkalinity in Acetone
D 1492 - 87	Bromine Index of Aromatic Hydrocarbons by Coulometric Titration
D 2324 - 81 (1989) [†]	Carbon Disulfide in Benzene
D 848 - 81 (1989) [†]	Color, Acid Wash, of Industrial Aromatic Hydrocarbons
D 849 - 88	Copper Corrosion by Industrial Aromatic Hydrocarbons
§D 130 - 88	Copper Corrosion from Petroleum Products
§D 1617 - 90	Ester Value of Lacquer Solvents and Thinners
§D 853 - 82 (1987) [†]	Hydrogen Sulfide and Sulfur Dioxide Content (Qualitative) of Industrial Aromatic Hydrocarbons
§D 1363 - 88	Permanganate Time of Acetone and Methanol
D 3961 - 89	Sulfur, Trace Quantities in Liquid Aromatic Hydrocarbons by Oxidative Microcoulometry
D 1685 - 86	Thiophene in Benzene, Traces of, Using Isatin and Spectrophotometry
D 890 - 58 (1987)	Water in Liquid Naval Stores
§D 1364 - 90	Water in Volatile Solvents (Fischer Reagent Titration Method)
E 203 - 75 (1986) [†]	Water Using Karl Fischer Reagent (see Vol 15.05)

Physical Tests

Test Methods for:

§D 3545 - 90	Acetate Esters, Alcohol Content and Purity by Gas Chromatography
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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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Test Methods for:

§D 3257 - 88	Aromatics in Mineral Spirits by Gas Chromatography
D 2935 - 81 (1985)	Apparent Density of Industrial Aromatic Hydrocarbons
D 4367 - 89	Benzene in Hydrocarbon Solvents by Gas Chromatography
§D 1209 - 84 (1988) ^{e1}	Color of Clear Liquids (Platinum-Cobalt Scale)
D 3054 - 81 (1985)	Cyclohexane, Purity and Benzene Content by Gas Chromatography
§D 850 - 86	Distillation of Aromatic Hydrocarbons
§D 86 - 90	Distillation of Petroleum Products
§D 1078 - 86	Distillation Range of Volatile Organic Liquids
D 5008 - 89	2-Ethylhexonal, Ethyl Methyl Pentanol Content and Purity Value by Gas Chromatography
§D 3539 - 87	Evaporation Rates of Volatile Liquids (see Vol 06.01)
D 3934 - 90	Flash/No-Flash—Equilibrium Method by a Closed-Cup Apparatus
§D 1310 - 86 (1990) ^{e1}	Flash Point and Fire Points of Liquids by Tag Open-Cup Apparatus
D 93 - 90	Flash Point by Pensky-Martens Closed Tester
§D 56 - 87	Flash Point by Tag Closed Tester
D 3941 - 90	Flash Point of Liquids by Equilibrium Method With Closed-Cup Apparatus
§D 3278 - 82 ^{e1}	Flash Point of Liquids by Setaflash Closed-Cup Apparatus
D 3893 - 90	Methyl Amyl Ketone and Methyl Isoamyl Ketone Purity by Gas Chromatography
§D 2804 - 88	Methyl Ethyl Ketone Purity Using Gas Chromatography
§D 3329 - 89	Methyl Isobutyl Ketone Purity by Gas Chromatography
D 2360 - 82 (1987) ^{e1}	Monocyclic Aromatic Hydrocarbons, Trace Impurities in, by Gas Chromatography
§D 1353 - 90	Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products
§D 1296 - 84 (1988) ^{e1}	Odor, Residual, of Volatile Solvents and Diluents
D 4773 - 89	Purity of Propylene Glycol Monomethyl Ether, Dipropylene Glycol Monomethyl Ether, and Propylene Glycol Monomethyl Ether Acetate
D 852 - 87	Solidification Point of Benzene
D 1493 - 84 (1988)	Solidification Point of Industrial Organic Chemicals
D 4206 - 89	Sustained Burning of Liquid Mixtures by Setaflash Apparatus (Open Cup)
D 4207 - 91	Sustained Burning of Low-Viscosity Liquid Mixtures by Wick Test
D 3962 - 80 (1989) ^{e1}	Styrene Analysis by Gas Chromatography
D 3009 - 72 (1981) ^{e1}	Turpentine Composition by Gas Chromatography
D 1553 - 83	Volume and Weight of Industrial Aromatic Hydrocarbons; Calculation of
§D 95 - 83 (1990)	Water in Petroleum Products and Bituminous Materials by Distillation
D 2306 - 81 (1985)	Xylene Isomer Analysis by Gas Chromatography
D 3797 - 88	<i>o</i> -Xylene Analysis by Gas Chromatography
D 3798 - 89	<i>p</i> -Xylene Analysis by Gas Chromatography
D 1217 - 86	Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer (see Vol 05.01)
D 1015 - 84	Freezing Points of High-Purity Hydrocarbons
D 1016 - 84	Purity of Hydrocarbons from Freezing Points
D 1218 - 87	Refractive Index and Refractive Dispersion of Hydrocarbon Liquids (see Vol 05.01)

Solubility and Miscibility Tests

Test Methods for:

D 611 - 82 (1987) ^{e1}	Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents
§D 1720 - 88	Dilution Ratio of Active Solvents in Cellulose Nitrate Solutions
§D 1476 - 88	Heptane Miscibility of Lacquer Solvents
§D 1133 - 90	Kauri-Butanol Value of Hydrocarbon Solvents
§D 1722 - 90	Water Miscibility of Water-Soluble Solvents

Liquid Naval Stores

Test Methods for:

D 801 - 57 (1987)	Dipentene, Sampling and Testing
§D 86 - 82 ^{e2}	Distillation of Petroleum Products
§D 56 - 87	Flash Point by Tag Closed Tester
D 802 - 82 (1987)	Pine Oil, Sampling and Testing
D 856 - 49 (1987)	Pine Tars and Pine-Tar Oils
D 1131 - 53 (1981) ^{e1}	Rosin Oils
D 803 - 82 (1987)	Tall Oil
§D 233 - 65 (1981) ^{e1}	Turpentine, Sampling and Testing
D 3009 - 72 (1981) ^{e1}	Turpentine Composition by Gas Chromatography
D 890 - 58 (1987)	Water in Liquid Naval Stores

General

Definitions of Terms Relating to:

D 804 - 79 (1987)	Naval Stores and Related Products
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MISCELLANEOUS RAW MATERIALS

Specifications for:

D 4710 - 87	Acetaldehyde
D 3620 - 90	Acetic Acid, Glacial
D 4416 - 89	Acrylic Acid
D 3547 - 91	<i>n</i> -Butyl Acrylate
D 608 - 90	Dibutyl Phthalate
D 3548 - 86	Ethyl Acrylate
D 3193 - 91	Ethylbenzene
D 3541 - 91	2-Ethylhexyl Acrylate
D 1969 - 91	2-Ethylhexyl (Synthetic)
D 2378 - 84 (1987)	Formaldehyde
D 1257 - 90	Glycerin, High-Gravity
D 3504 - 91	Maleic Anhydride
D 3845 - 89	Methacrylic Acid, Glacial
D 4709 - 87	Methyl Acrylate
D 600 - 90	Paint Driers, Liquid
D 2403 - 68 (1982) ^{ei}	Phthalic Anhydride-1308, Refined
D 363 - 90	Tricresyl Phosphate
D 2190 - 89	Vinyl Acetate

Test Methods for:

D 3362 - 84 (1987)	Acrylate Esters, Purity by Gas Chromatography
D 4415 - 91	Acrylic Acid, Determination of Dimer in
D 2192 - 89	Aldehydes and Ketones, Purity of
D 2119 - 87	Aldehydes in Styrene Monomer
D 2613 - 85 (1990) ^{ei}	Calcium and Zinc in Paint Driers by EDTA Method
D 3970 - 80 (1990) ^{ei}	Cerium in Paint Driers by Oxidimetric Determination
D 2373 - 85 (1990) ^{ei}	Cobalt in Paint Driers by EDTA Method
D 2379 - 84 (1987)	Formaldehyde Solutions, Acidity of
D 2194 - 89	Formaldehyde Solutions, Concentration of
D 2087 - 89	Formaldehyde Solutions, Iron in
D 2380 - 84 (1987)	Formaldehyde Solutions, Methanol Content of
D 3546 - 90	Formic Acid in Glacial Acetic Acid
D 1258 - 90	Glycerin, High-Gravity, Testing
D 1728 - 83	Glycerin, Phthalate Ester Color of High Gravity (Discontinued 1991†)
D 3804 - 86 (1991) ^{ei}	Iron in Paint Driers by EDTA Method
D 2374 - 85 (1990) ^{ei}	Lead in Paint Driers by EDTA Method
D 2375 - 85 (1990) ^{ei}	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) ^{ei}	Paint Driers, Liquid
D 2195 - 84	Pentaerythritol
D 3989 - 81a (1990) ^{ei}	Rare Earth Metals, Total, in Paint Driers by EDTA Method
D 3962 - 80 (1984) ^{ei}	Styrene Analysis by Gas Chromatography
D 2119 - 87	Styrene Monomer, Aldehydes in
D 2120 - 87	Styrene Monomer, <i>p</i> - <i>tert</i> -Butylcatechol Inhibitor in
D 2340 - 82 (1987) ^{ei}	Styrene Monomer, Peroxides in
D 2121 - 84	Styrene Monomer, Polymer Content of
D 1721 - 84 (1988) ^{ei}	Tricresyl Phosphate, Permanganate Time of
D 1399 - 90	Tricresyl Phosphate, Unsaponifiable Content of
§D 1468 - 84 (1988) ^{ei}	Tricresyl Phosphate, Volatile Matter in
§D 1638 - 74 ^{ei}	Urethane Foam Isocyanate Raw Materials (Discontinued 1991†)
D 3988 - 85 (1990) ^{ei}	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) ^{ei}	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) ^{ei}	Acid Number of Rosin
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LIST BY SUBJECTS, VOLUME 06.03

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</i> - <i>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</i> - <i>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 06.03

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) ^{e1}	Pyridine Bases in Cresylic Acid by Direct Titration
D 2031 - 84 (1989) ^{e1}	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) ^{e1}	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) ^{e1}	Tar Acid Composition by Gas Liquid Chromatography
D 1685 - 86 (1990)	Thiophene in Benzene, Traces of, Using Isatin and Spectrophotometry
D 4735 - 87 (1991) ^{e1}	Thiophene in Refined Benzene, Trace, by Gas Chromatograph
§D 1555 - 91	Volume and Weight of Industrial Aromatic Hydrocarbons, Calculation of
D 1631 - 85 (1989) ^{e1}	Water in Phenol and Related Materials by the Iodine Reagent Method
D 2030 - 84 (1989) ^{e1}	Water Solubility of Refined Pyridine
D 2306 - 81 (1985)	Xylene Isomer Analysis by Gas Chromatography
D 3797 - 88	<i>o</i> -Xylene by Gas Chromatography
D 3798 - 89	<i>p</i> -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	<i>p</i> -Xylene, Analysis of (Discontinued 1992†—Replaced by Specification D 5136)
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Terminology of:

D 4790 - 89a	Aromatic Hydrocarbons and Related Chemicals
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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)
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METRIC PRACTICE

Practice for:

§E 380 - 91	Use of the International System of Units (SI) (the Modernized Metric System) (Excerpts) (see Related Material section)
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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.

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² From the French name, *Le Système International d'Unités*.

TABLE 1 Base SI Units

Quantity ^a	Unit	Symbol
length	metre	m
mass	kilogram	kg
time	second	s
electric current	ampere	A
thermodynamic temperature ^a	kelvin	K
amount of substance	mole	mol
luminous intensity	candela	cd

^a For a discussion of Celsius temperature see 3.4.2.

TABLE 2 Supplementary SI Units

Quantity ^a	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-

TABLE 3 Derived SI Units with Special Names

Quantity ^a	Unit	Symbol	Formula
frequency (of a periodic phenomenon)	hertz	Hz	$1/\text{s}$
force	newton	N	$\text{kg}\cdot\text{m}/\text{s}^2$
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 ^a	$^{\circ}\text{C}$	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	$1/\text{s}$
absorbed dose ^b	gray	Gy	J/kg
dose equivalent	sievert	Sv	J/kg

^a Inclusion in the table of derived SI units with special names approved by the CIPM in 1976.

^b 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 appropriately 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

^a "Quantity" as used in the headings of the tables of this standard means measurable attribute of phenomena or matter.

TABLE 4 Some Common Derived Units of SI

Quantity ³	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

⁴ See 3.4.4.

measurements and clothing sizes.

3.2.3 Prefixes in Compound Units⁴—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*⁵ (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 X1.11.1.

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 ^a	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	° = ($\pi/180$) rad
plane angle	minute ^A	'	1' = ($1/60$)°
			= ($\pi/10\ 800$) rad
	second ^A	"	1" = ($1/60$)'
			= ($\pi/648\ 000$) rad
volume	litre ^a	L	1 L = 1 dm ³ = 10^{-3} m ³
mass	metric ton	t	1 t = 10 ³ kg
area	hectare	ha	1 ha = 1 hm ² = 10 ⁴ m ²

^A Use discouraged except for special fields such as cartography.

^a 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 ^a	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 radionuclide)	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

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 (N/m² = Pa), energy (N·m = J), and power (N·m/s = 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

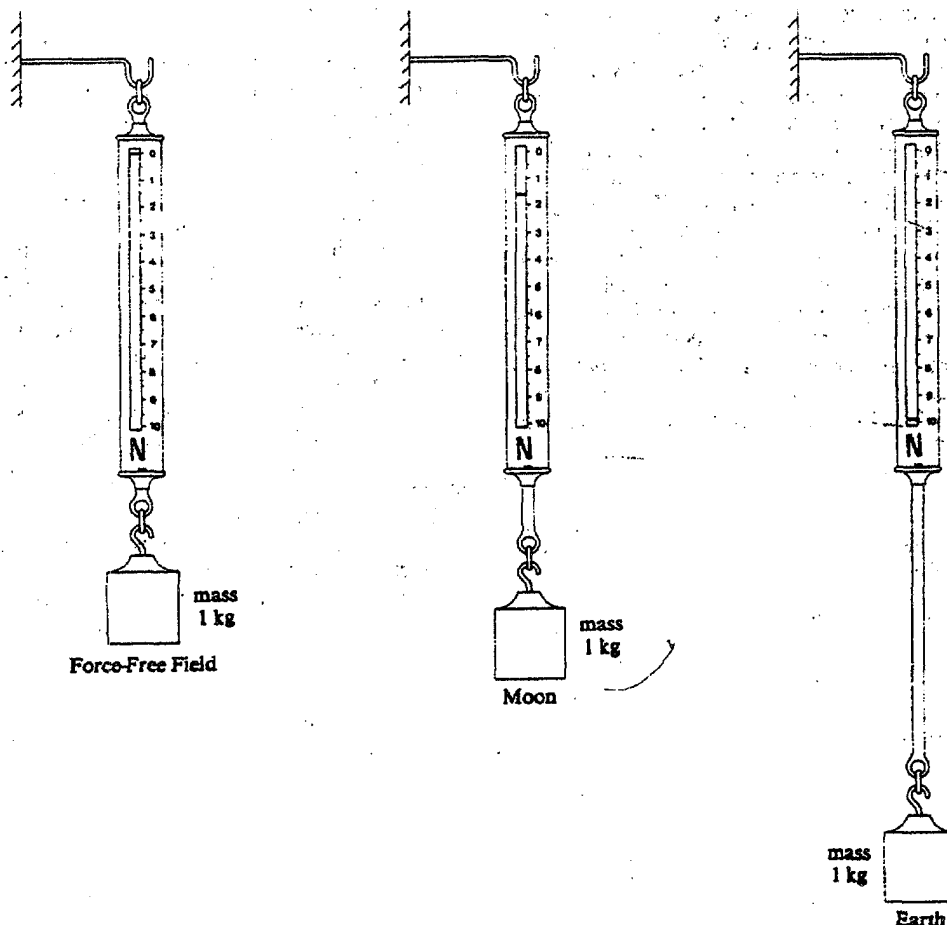


FIG. 1 Illustration of Difference Between Mass (Unit = kilogram = kg) and Force (Unit = newton = N) (see 3.4.1)

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 (°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 $T_0 = 273.15$ 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 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/s² respectively. In accordance with Sec. 2.3, since the radian is here taken to be dimensionless, the units 1, 1/s, and 1/s² 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 N·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 kg·m².

3.4.4.4 *Angular Momentum* (moment of momentum) is linear momentum (kg·m/s) times moment arm (m). Its SI unit is kg·m²/s. For a rotating body the total angular momentum is equal to the moment of inertia I (kg·m²) 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 (N·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 (N·m) divided by angle of twist (rad). Its SI unit is N·m/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/s².

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:

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 print-outs, 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:

$J/(\text{mol} \cdot \text{K})$ or $J \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$ 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:

joules per kilogram or J/kg or $J \cdot \text{kg}^{-1}$

but not

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: $(3 \times 0.3048) + (2.5625 \times 0.0254) = 0.979\,487\,5\text{ m}$, 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 (j as in jig, a as in about)
mega	as in megaphone
kilo	kill' oh
hecto	heck' toe
deka	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.

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 $\frac{1}{16}$ 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 $1\frac{3}{16}$ 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 $\frac{1}{2}$ in ($\pm \frac{1}{4}$ 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 $\pm 0.25\%$ 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:

$1\frac{7}{8}$ in = 47.625 mm exact

47.6 mm normal rounding

47.5 mm (approx) rounded to preferred number

48 mm (approx) rounded to whole number

4.2.2.2 A quantity stated as a limit, such as "not more than" or "maximum," must be handled so that the stated limit is not violated. For example, a specimen "at least 4 in 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, consideration must be given to the implied or required precision of the integral value to be converted. For example, the value "4 in" may be intended to represent 4, 4.0, 4.00, 4.000, or 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 specific value or quantity is said to be significant. When measured to the nearest 1 m, a distance may be recorded as 157 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 figure rounded to thousands was 203 185 000. The six left-hand digits of this number are significant; each *measures* a value. 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 only possible through knowledge of the circumstances. For example, the number 1000 may be rounded from 965, 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 must be drawn from a variety of sources where they have been recorded with varying degrees of refinement. Specific rules 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, the first 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, and the sum taken as follows:

163 000 000
217 900 000
96 400 000
477 300 000

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 the 8 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.

A dimension is expressed in inches as	1.950 ± 0.016
The limits are	1.934 and 1.966
Conversion of the two limits into millimetres gives	49.1236 and 49.9364
<i>Method A</i> —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
<i>Method B</i> —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 ± 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 ± 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 × 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 (±1)	1 (±0.5)
4 (±2)	2 (±1)
10 (±5)	6 (±3)
20 (±10)	11 (±5.5)
30 (±15)	17 (±8.5)
40 (±20)	22 (±11)
50 (±25)	28 (±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 ± 5°F; implied accuracy estimated to be 2°F.

37.777 ± 2.777°C rounds to 38 ± 3°C.

1000 ± 50°F; implied accuracy estimated to be 20°F.

537.777 ± 27.777°C rounds to 540 ± 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

depends 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 irrefragable recognized standard or specified value. This concept involves a certain systematic error of an operation, which is seldom materially 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.

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E 380 SELECTED CONVERSION FACTORS

To convert from	to	multiply by
atmosphere (760 mm Hg)	pascal (Pa)	$1.013\ 25 \times 10^5$
board foot	cubic metre (m ³)	$2.359\ 737 \times 10^{-3}$
Btu (International Table)	joule (J)	$1.055\ 056 \times 10^3$
Btu (International Table)/h	watt (W)	$2.930\ 711 \times 10^{-1}$
Btu (International Table)·in./s·ft ² ·°F (<i>k</i> , thermal conductivity)	watt per metre kelvin [W/(m·K)]	$5.192\ 204 \times 10^2$
calorie (International Table)	joule (J)	4.186 800*
centipoise	pascal second (Pa·s)	$1.000\ 000^* \times 10^{-3}$
centistokes	square metre per second (m ² /s)	$1.000\ 000^* \times 10^{-6}$
circular mil	square metre (m ²)	$5.067\ 075 \times 10^{-10}$
degree Fahrenheit	degree Celsius	$t^{\circ}\text{C} = (t^{\circ}\text{F} - 32)/1.8$
foot	metre (m)	$3.048\ 000^* \times 10^{-1}$
ft ²	square metre (m ²)	$9.290\ 304^* \times 10^{-2}$
ft ³	cubic metre (m ³)	$2.831\ 685 \times 10^{-2}$
ft·lbf	joule (J)	1.355 818
ft·lbf/min	watt (W)	$2.259\ 697 \times 10^{-2}$
ft/s ²	metre per second squared (m/s ²)	$3.048\ 000^* \times 10^{-1}$
gallon (U.S. liquid)	cubic metre (m ³)	$3.785\ 412 \times 10^{-3}$
horsepower (electric)	watt (W)	$7.460\ 000^* \times 10^{+2}$
inch	metre (m)	$2.540\ 000^* \times 10^{-2}$
in. ²	square metre (m ²)	$6.451\ 600^* \times 10^{-4}$
in. ³	cubic metre (m ³)	$1.638\ 706 \times 10^{-5}$
inch of mercury (60°F)	pascal (Pa)	$3.376\ 85 \times 10^3$
inch of water (60°F)	pascal (Pa)	$2.488\ 4 \times 10^2$
kgf/cm ²	pascal (Pa)	$9.806\ 650^* \times 10^4$
kip (1000 lbf)	newton (N)	$4.448\ 222 \times 10^3$
kip/in. ² (ksi)	pascal (Pa)	$6.894\ 757 \times 10^6$
ounce (U.S. fluid)	cubic metre (m ³)	$2.957\ 353 \times 10^{-5}$
ounce-force	newton (N)	$2.780\ 139 \times 10^{-1}$
ounce (avoirdupois)	kilogram (kg)	$2.834\ 952 \times 10^{-2}$
oz (avoirdupois)/ft ²	kilogram per square metre (kg/m ²)	$3.051\ 517 \times 10^{-1}$
oz (avoirdupois)/yd ²	kilogram per square metre (kg/m ²)	$3.390\ 575 \times 10^{-2}$
oz (avoirdupois)/gal (U.S. liquid)	kilogram per cubic metre (kg/m ³)	7.489 152
pint (U.S. liquid)	cubic metre (m ³)	$4.731\ 765 \times 10^{-4}$
pound-force (lbf)	newton (N)	4.448 222
pound (lb avoirdupois)	kilogram (kg)	$4.535\ 924 \times 10^{-1}$
lbf/in. ² (psi)	pascal (Pa)	$6.894\ 757 \times 10^3$
lb/in. ³	kilogram per cubic metre (kg/m ³)	$2.767\ 990 \times 10^4$
lb/ft ³	kilogram per cubic metre (kg/m ³)	$1.601\ 846 \times 10$
quart (U.S. liquid)	cubic metre (m ³)	$9.463\ 529 \times 10^{-4}$
ton (short, 2000 lb)	kilogram (kg)	$9.071\ 847 \times 10^2$
torr (mm Hg, 0°C)	pascal (Pa)	$1.333\ 22 \times 10^2$
W·h	joule (J)	$3.600\ 000^* \times 10^3$
yard	metre (m)	$9.144\ 000^* \times 10^{-1}$
yd ²	square metre (m ²)	$8.361\ 274 \times 10^{-1}$
yd ³	cubic metre (m ³)	$7.645\ 549 \times 10^{-1}$

* Exact

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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.

¹ This index was developed by ASTM Committee D-1 and is not an official ASTM document. It is published for information purposes only.

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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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- For members serving on technical committees, each organization may ask that the membership be transferred without payment of additional fees, if the fees were paid by the company. However, no additional free volume of the Book of ASTM Standards will be allowed. Committee participation is not transferable. An application will have to be submitted by the new member of the appropriate committee.

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- All publications must be shipped to the member.
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