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AEROSOL DIVISION

AEROSOL GUIDE

Prepared in Loose Leaf Form to Allow for
Additions and Revisions

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CHEMICAL SPECIALTIES MANUFACTURERS ASSOCIATION, INCORPORATED
EXECUTIVE OFFICES ▫ FIFTY EAST FORTY-FIRST STREET, NEW YORK, NEW YORK 10017 ▫ MURRAY HILL 5-8722

March 1966

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AEROSOL DIVISION

AEROSOL GUIDE

INTRODUCTION

A Guide for Vendors and Fillers of Aerosols and Pressurized Packages

Includes Tests Developed by the Scientific Committee of the Aerosol Division

This Guide is an assembly of recommendations, and developments of the Committees of the Aerosol Division of the Chemical Specialties Manufacturers Association, Inc. In the development of the several sections, extensive discussions and cooperation was received from the United States Department of Agriculture, United States Post Office Department, United States Department of Commerce, the Fire Department of New York City and other large cities, the Bureau of Explosives of the Association of American Railroads, and others.

The Self Pressurized Packaging (Aerosol) Industry continues to develop with great rapidity. Many of those engaged in selling, filling or supplying materials are working diligently to establish sound practices within the industry.

All of the recommendations are the result of long and careful study by the committees of all phases of the industry and by consultations with many authorities in regard to transportation, legal, and other respects. These recommendations will need frequent corrections and additions.

Vendors of Aerosols should study these recommendations and insist that they be carefully followed. Fillers of Pressurized Packages for their own sale or under contract should make certain that the basic recommendations are complied with.

Copies of this Guide are available at nominal cost to anyone interested in the production or sale of pressurized packages. New methods or procedures which are approved and adopted in the future will be periodically made available as costs to be established as additions to the Aerosol Guide.

Government Agencies are being furnished copies to be used in assisting in the proper regulation of these industries.

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PREMARKETING PRODUCT CHECK

Preliminary Studies Before Production. (Prepared by the Product Check List, a Sub-Committee of the Aerosol Division Scientific Committee. Published in the Proceedings of the 38th Midyear Meeting of the C.S.M.A. — Page 53).

Pre-Marketing Check List

The design of the check list assumes that various marketing questions have been answered and that the management of the company has decided to go ahead with the development and marketing of the product. This master check list, of necessity, refers to the broad base of all Aerosol and Aerated products and is meant to be applicable to all those currently produced and new products under development.

I. Formulation Checks

1. Low temperature characteristics (stability, viscosity) of non-volatile concentrate
   a. 100° F
   b. 70° F
   c. 0° F
   d. −20° F
2. Compatibility of non-volatile concentrate with propellent
   a. critical solubility range
   b. effect of ratio of propellent to concentrate on stability
   c. effect of temperature on stability of combination of propellent and non-volatile concentrate
      1. 100° F
      2. 70° F
      3. 0° F
      4. −20° F
3. Specific heat of non-volatile concentrate
4. Vapor pressure characteristics (curve below boiling point through 170° F)
5. Specific heat of propellent
6. Co-efficient of expansion of completed formulation of non-volatile concentrate and propellent (giving head space requirements)
7. Determination of flammability of product
8. pH of product
9. Density of product
10. Determination of toxicity
    a. Review of literature on various ingredients
    b. Confirmation of literature reports by:
       1. Mist chamber tests
       2. Shock or massive-dose type topical application
       3. Chronic topical application
       4. Diet tests
       5. Skin sensitivity tests
11. Effectiveness of product for purpose intended determined by commercially accepted techniques:
    a. Association test methods
    b. Federal specifications
    c. ASTM standards
    d. Bureau of Standards techniques

II. Container and Valve Components vs. Formulation.

Accelerated Aging at Laboratory Level in Actual Container with Valve as Well as Individual Components Under Pressurized Glass

1. Does product affect container?
   a. Effect on internal coating, lacquer or plate
   b. Effect on seam component
2. Does product affect valve mechanism?
   a. Metal components
   b. Elastomeric seals
   c. Plastic components

3. Performance of valve with formulated product
   a. Spray characteristics
   b. Spray rate
   c. Valve action

III. Valve and Container Selection

1. Suitability of container to formula (See II. 2b)
2. Bursting strength of container
3. Effect of formulation on outside finish of container
4. Susceptibility of valve to clogging
5. Delivery rate of valve
6. Spray characteristics of valve
7. Valve attachment to dispenser
8. Syphon tube selection
9. Valve testing prior to use
10. Commercial history of containers and valves
11. Sales appeal of container and valve
12. Adaptability of valve and dispenser to other products
13. Tamper-proof seal
14. Packaging of empty dispensers (receiving cartons)
15. Packaging of filled dispensers (shipping cartons)

IV. Completed Products Use Test

1. Effectiveness of product (See Item I, II)
2. Development of instructions and label (directions and cautions)
3. Determination of odor
4. Ease of operation
5. Possible adverse effects on materials in home
6. Flammability hazards
7. Test pack for obtaining storage data at various temperatures and various positions of container during storage
   a. Effect on component parts of container and valve
   b. Changes in pressure characteristics of product
   c. Changes in odor
   d. Changes in color
   e. Changes in pH
   f. Effect on valve of intermittent spraying
   g. Effect of tendency to leak
   h. Effect on delivery rate
   i. Possible crystallization in expansion chamber
8. Determination of products not exclusive of manufacturing cost

V. Manufacturing

1. Bill of material
2. Filling processing procedure
3. Quality control procedure
   a. Raw materials
   b. Processing
   c. Finished product
4. Manufacturing cost

VI. Regulatory Considerations

1. United States Department of Agriculture — Insecticide, Fungicide and Rodenticide Act. (Also State Acts)
2. Bureau of Explosives. Association of American Railroads and Interstate Commerce Commission
3. Food and Drug Administration—Federal Food, Drug and Cosmetic Act. (Also State Acts)
4. State and Municipal regulations

Reported by the Check List Sub Committee, June, 1952
Adopted by the Scientific Committee, June, 1952

LABELING

The Precautionary Labeling Committee has prepared a suggested warning label to appear on all metal aerosol (pressurized) packages.

This statement applies for metal can aerosols.

WARNING!

Contents under pressure.

Exposure to heat may cause bursting.

Do not puncture or incinerate.

Avoid prolonged exposure to sunlight.

Note: Additional requisite cautions for statements as to hazards to person and property, the safe discharge of contents, etc., will depend upon the particular product.

Appropriate modifications of this labeling is suggested for aerosol containers made of other materials.

For additional information on type size and location of label and labeling consult the following:

1. LAWS, REGULATIONS AND AGENCIES OF INTEREST TO THE AEROSOL INDUSTRY*
2. COMPILATION OF LABELING LAWS AND REGULATIONS FOR HAZARDOUS SUBSTANCES*
3. COMPILATION OF ECONOMIC POISONS (PESTICIDES) LAWS*
4. COMPILATION OF WEIGHTS AND MEASURES LAWS AND REGULATIONS*
5. FEDERAL FOOD, DRUG AND COSMETIC ACT

*all may be ordered from the CSMA office.

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REVISED FLAMMABILITY TEST METHODS FOR AEROSOL PRODUCTS

Recommendations of the Subcommittee on Consumer Hazards and Safety-Task Group No. 2

Approved by Aerosol Scientific Committee September 11, 1963
Adopted by Board of Governors September 12, 1963

1. Abandon the Open Drum Test as a meaningful guide in establishing flammability hazards.

2. The following changes and modifications in the Flame Projection Test, Tagliabue Open-Cup Flash Point Test, and Closed Drum Test:

Flame Projection Test

1. *Equipment required.* The test equipment consists of a base 8 inches wide, 2 feet long, marked in 6 inch intervals. A rule 2 feet long and marked in inches is supported horizontally on the side of the base and about 6 inches above it. A paraffin candle approximately 1 inch in diameter and of such height that the top third of the flame is at the height of the horizontal rule, is placed at the zero point in the base.

2. *Procedure.* The test is conducted in a draft-free area that can be ventilated and cleared after each test. Condition the dispenser to 70° ± 1° F. Shake the dispenser before test. Hold the dispenser upright unless label states otherwise. Place the dispenser at a distance of 6 inches from the flame source. Spray for 4 seconds (one observer noting the extension of the flame and the other operating the dispenser) through the top third of the flame and essentially parallel to the rule. The height of the flame should be approximately 2 inches. The normal bending of the flame is part of the recorded distance. Take 3 readings for each test and average. As a precaution do not spray large quantities in a small, confined space. Free space of previously discharged material.

Tagliabue Open-Cup Flash Point Test

1. *Equipment.* ASTM Designation: D 1310-55 T. Issued 1954; Revised 1955. "Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus". The apparatus proposed for use is the new Tag Fischer open-cup flash point apparatus, with the addition of some means, preferably an open type of vessel to contain dry ice, to chill the aerosol unit.

2. *Procedure.* The aerosol unit, filled as for use, is chilled to a temperature of about 25° F. below zero and also the flash cup and the bath solution (brine or glycol). The chilled formulation is transferred to the test apparatus and the cup filled to the conventional level using the mechanical leveling device furnished with the apparatus. The test liquid is allowed or caused to increase in temperature at a rate of about 2° F. per minute and the test flame taper passed across the cup at intervals of 2° F. until the sample reaches −20° F. or until the test sample has evaporated completely.

This procedure is not applicable to products in which the presence of a solid portion prevents the transfer of a uniform sample to the cup at −25° F.

Closed Drum Test

1. *Equipment required.* The apparatus consists of a 55-gallon open head drum which has been modified as follows:

(1) a closure is fitted over the open head (see note No. 1);

(2) a circular opening 1 inch in diameter is bored through the base, about 2 inches from the edge, in such a position that when the drum is on its side the hole will be at the top.

(3) a metal base 9 inches long, 2 inches wide and at least $\frac{1}{16}$ inch thick is used as a candle support (see drawing No. 1);

(4) a paraffin candle approximately 1 inch in diameter and at least 3 inches in height;

(5) optionally a 6-inch square opening is cut through the center of the base of the drum and securely covered with a piece of safety glass.

2. *Procedure.* Lay the modified drum on its side under conditions where the temperature is between 60° and 80° F., but preferentially as close to 70° F. as possible. Stand a paraffin candle in the drum on a special metal base half-way from each end of the drum. (See drawing No. 1) Condition the dispenser to 70° ± 1° F.

Light the candle on the base and secure closure. (When the film closure is used the candle should be ignited by means of a taper through the 1 inch circular opening). Shake the dispenser and hold upright or, if necessary, in such a position that the liquid contents can be sprayed directly into the drum. As quickly as possible place the dispenser at the one-inch opening and spray into the drum, directing the spray toward the center of the opposite end until an explosion takes place, or for a period up to 60 seconds, whichever occurs first. After each test, open the drum to clear the atmosphere. Clean the drum of any residues which might affect future tests. Repeat the test twice as before using the same dispenser if possible. If size limitations make it necessary to use more than one dispenser, then not more than one dispenser shall be used in the performance of any one test.

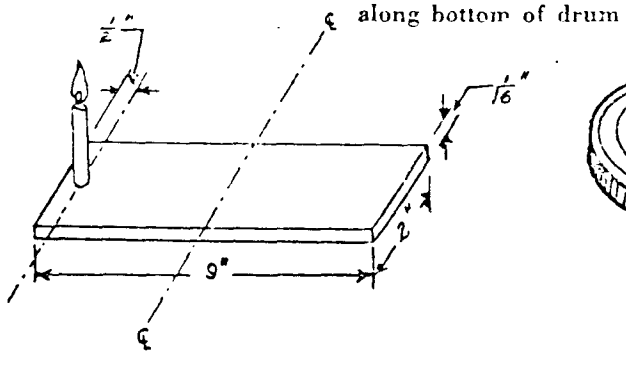
The results of three tests are averaged.

Note 1. A hinged lid or a 1½ mil thickness polyamide film can be used as the closure.

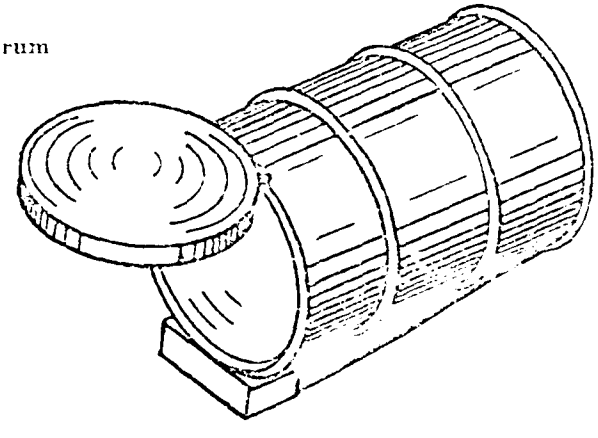
Polyamide Film: The film is stretched over the open end of the drum and held in place by a strong rubber band, that will stretch one inch when a one pound weight is hung from its lowest point, when around the drum. A one-inch slit is cut vertically in the film beginning at a point 2 inches from the top of the drum.

The polyamide film must be drawn taut over the opening.

CANDLE BASE



HINGED LID



Drawing No. 1

CHEMICAL SPECIALTIES MANUFACTURERS ASSOCIATION
INCORPORATED

50 EAST 41st STREET, NEW YORK, N. Y. 10017

RETURN TO

FEDERAL REGULATION — BULLETIN NO. [illegible] July 3, 1969

All Divisions	Aerosol	Automotive	Detergent and Cleaning Compounds	Disinfectants and Sanitizers	Insecticide	Waxes, Polishes and Floor Finishes

HAZARDOUS MATERIALS REGULATIONS BOARD
PART 173 - SHIPPERS
AEROSOL FLASHPOINT RESTRICTION

(From the Federal Register of Thursday, June 26, 1969, Page 9869)

The following is reproduced for the general information of the membership.

A. A. Mulliken
Executive Director

PART 173—SHIPPERS

Aerosol Flash Point Restriction

The purpose of this amendment to the Hazardous Materials Regulations (49 CFR 170–189) is to delete § 173.306(a)(3)(iv) and thereby remove the flash point restriction applicable to aerosol products packaged in compliance with the "exempt shipment" provisions otherwise specified.

On March 12, 1969, the Hazardous Materials Regulations Board published a notice of proposed rule making, Docket No. HM-15; Notice No. 69-6 (34 F.R. 5112) to eliminate § 173.306(a)(3)(iv). The deletion of the subparagraph would have the effect of allowing the "exempt shipment" of aerosol products which are flammable by § 173.300(b) criteria without regard to flash point. However, all the other restrictions specified such as use of metal containers only, 50 cubic inches capacity limitation, pressure limitation of contents in relation to container strength, adequate head space, and a heat test of 130° F. of each complete container filled for shipment, would be retained. Interested persons were afforded an opportunity to participate in this rule making.

A number of comments were received in response to this notice. The large majority of comments concurred with the proposal. One commenter objected to removal of the 20° F. limitation for aerosol products shipped by air or water in the belief that, should leakage occur in a confined cargo space, a flash fire could result from a spark or other source of heat. Another commenter expressed the opinion that the flashpoint limitation should be retained, asserting that there is need to pressurize these containers which explode and splash burning materials over a wide range, thus propagating fire over a wider range. It is true that there is some possibility that these incidents could occur but the same is true for materials having flashpoints higher than 20° F. when they are expelled under pressure.

No adverse experience has been reported to the Department concerning the transportation of several million cans shipped under special permits issued by the Department wherein the flashpoint restriction had been removed. Further, the integrity of each can will be adequately preserved by compliance with the requirements retained in § 173.306 (a)(3). In view of the previous experience under special permits and of the requirements retained, and considering the conditions normally incident to transportation, deletion of § 173.306(a)(3)(iv) is considered reasonable and justified.

In consideration of the foregoing, 49 CFR Part 173 is amended effective September 3, 1969, by deleting § 173.306(a)(3)(iv). However, compliance with the regulations as amended herein is authorized immediately.

This amendment is made under the authority of sections 831–835 of title 18, United States Code, section 9 of the Department of Transportation Act (49 U.S.C. 1657), and title VI and section 902 (h) of the Federal Aviation Act of 1958 (49 U.S.C. 1421–1430 and 1472(h)).

Issued in Washington, D.C., on June 19, 1969.

W. J. Smith,
Admiral, U.S. Coast Guard,
Commandant.

R. N. Whitman,
Administrator,
Federal Railroad Administration.

F. C. Turner,
Administrator,
Federal Highway Administration.

Sam Schneider,
Board Member for the
Federal Aviation Administration.

[F.R. Doc. 69-7523; Filed, June 25, 1969; 8:47 a.m.]

[Docket No. HM-17; Amdt. 173-8]

FLASH POINT DETERMINATION

SPECIAL TEST OF I.C.C. TARIFF 9 [73-302(a)(3)]

Revision applicable June 18, 1957

The details and methods of this test applied to aerosols is for use only in connection with determination of flash points for shipping and markings under Tariff 9-73-302 (a) (3) June, 18 1957.

FLASH POINT DETERMINATION METHOD FOR 73.302 (a) (3)

Bureau of Explosives
August 13, 1956

ASTM Designation: D 1310 --55 T. Issued 1954; Revised, 1955. "Flash Point of Volatile Flammable Materials by Tag Open-Cut Apparatus"

The apparatus proposed for use is the new Tag Fischer open-cup flash point apparatus, with the addition of some means, preferably an open type of vessel to contain dry ice, to chill the aerosol unit.

Detail—The aerosol unit, filled as for use, is chilled to a temperature of about 25°F. below zero and also the flash cup and the bath solution (brine or glycol). The chilled formulation is transferred to the test apparatus and the cup filled to the conventional level using the mechanical leveling device furnished with the apparatus. The test liquid is allowed or caused to increase in temperature at a rate of about 2°F. per minute and the test flame taper passed across the cup at intervals of 2°F. until the sample reaches −20°F. or until the test sample has evaporated completely.

It may be necessary to deviate from the procedure outlined in that the liquid level in the cup will not remain constant during the course of the test.

EXCERPTS FROM REGULATIONS ISSUED FOR THE

FEDERAL HAZARDOUS SUBSTANCES LABELING ACT

§ 191.1 *Definitions.*

(j) *Extremely flammable and flammable substances*—(1) *Extremely flammable substances.* The term "extremely flammable" means any substance that has a flashpoint at or below 20° F., as determined by the method described in § 191.13.

(2) *Flammable substances.* The term "flammable" means any substance that has a flashpoint of above 20° F., to and including 80° F., as determined by the method described in § 191.13.

(k) *Extremely flammable and flammable solids*—(1) *Extremely flammable solids.* A solid substance is "extremely flammable" if it ignites and burns at an ambient temperature of 80° F. or less when subjected to friction, or to percussion or to an electrical spark.

(2) *Flammable solids.* A solid substance is "flammable" if, when tested by the method

described in § 191.14, it ignites and burns with a self-sustained flame at a rate greater than 1 10 of an inch per second along its major axis.

(l) *Extremely flammable and flammable contents of self-pressurized containers* — (1) *Extremely flammable contents.* Contents of self-pressurized containers are "extremely flammable" if when tested by the method prescribed in § 191.15, flashback (a flame extending back to the dispenser) is obtained at any degree of valve opening and the flashpoint, when tested by the method described in § 191.16, is less than 20° F.

(2) *Flammable contents.* Contents of self-pressurized containers are "flammable" if when tested by the method described in § 191.15 a flame projection exceeding 18 inches is obtained at full valve opening or a flashback (a flame extending back to the dispenser) is obtained at any degree of valve opening.

(m) *Substances that generate pressure.* A substance is hazardous because it "generates pressure through decomposition, heat, or other means" if:

(1) It explodes when subjected to an electrical spark, or to percussion, or to the flame of a burning paraffin candle for 5 seconds or less; or

(2) It expels the closure of its container, or bursts its container, when held at or below 130° F. for 2 days or less; or

(3) It erupts from its opened container at a temperature of 130° F. or less, after having been held in the closed container at 130° F. for 2 days.

(4) If it comprises the contents of a self-pressurized container.

§ 191.13 Tentative method of test for flashpoint of volatile flammable materials by Tagliabue open-cup apparatus[1,2]

Scope

1. (a) This method describes a test procedure for the determination of open-cup flashpoints of volatile flammable materials having flashpoints below 175° F.

(b) This method, when applied to paints and resin solutions which tend to skin over or which are very viscous, gives less reproducible results than when applied to solvents.

Outline of Method

2. The sample is placed in the cup of a Tag Open Tester, and heated at a slow but constant rate. A small test flame is passed at a uniform rate across the cup at specified intervals. The flashpoint is taken as the lowest temperature at which application of the test flame causes the vapor at the surface of the liquid to flash, that is, ignite but not continue to burn.

Apparatus

3. The Tag open-cup tester is illustrated in Fig. 1. It consists of the following parts, which must conform to the dimensions shown, and have the additional characteristics as noted:

(a) *Copper bath*, preferably equipped with a constant level overflow so placed as to maintain the bath liquid level 1/8 inch below the rim of the glass cup.

(b) *Thermometer holder. Support firmly* with ringstand and clamp.

(c) *Thermometer.* For flashpoints above 40° F., use the ASTM Tag Closed Tester Thermometer, range of −20 to −230° F., in 1° F. divisions, and conforming to thermometer 9F. of ASTM Standard E 1. For flashpoints from 20° F. to 40° F., use ASTM Tag Closed Tester, Low Range, Thermometer 57F. For flashpoints below 20° F., use ASTM Thermometer 33F. The original Tag Open-Cut (Paper Scale) Thermometer will be a permissible alternate unit January 1, 1962. It is calibrated to −20° F.

(d) *Glass test cup.* Glass test cup (Fig. 2), of molded clear glass, annealed, heat-resistant, and free from surface defects.

(e) *Leveling device.* Leveling device or guide, for proper adjustment of the liquid level in the cup (Fig. 3). This shall be made of No. 18-gage polished aluminum, with a projection for adjusting the liquid level when the sample is added to exactly 1/8-inch below the level of the edge or rim of the cup.

(f) "Micro," or small gas burner of suitable dimensions for heating the bath. A screw clamp may be used to help regulate the gas. A small electric heater may be used.

(g) Ignition taper, which is a small straight, blow-pipe type gas burner. The test flame torch prescribed in the method of test for flash and fire points by Cleveland Open Cup (ASTM designation: D 92) is satisfactory.

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[1] The Food and Drug Administration has obtained permission from the American Society for Testing Materials, Philadelphia, Pa., to reprint this method in these regulations. The text has been slightly modified, for practical reasons.

[2] ASTM Designation: D 1310-59T, issued 1954; revised 1955, 1956, 1959. This tentative method has been approved by the sponsoring committee and accepted by the American Society for Testing Materials in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia, Pa.

(h) Alternative methods for maintaining the ignition taper in a fixed horizontal plane above the liquid may be used, as follows:

(1) Guide wire, 3 32-inch in diameter and $3\frac{1}{2}$ inches in length, with a right-angle bend $\frac{1}{8}$-inch from each end. This wire is placed snugly in holes drilled in the rim of the bath, so that the guide wire is $\frac{5}{8}$-inch from the center of the cup and resting on the rim of the cup.

(2) Swivel-type taper holder, such as is used in ASTM METHOD D 92. The height and position of the taper are fixed by adjusting the holder on a suitable ringstand support adjacent to the flash cup.

(i) Draft shield, consisting of two rectangular sheets of noncombustible material, 24 inches x 28 inches, are fastened together along the 28-inch side, preferably by hinges. A triangular sheet, 24 inches x 24 inches x 34 inches is fastened by hinges to one of the lateral sheets (to form a top when shield is open). The interior of the draft shield shall be painted a flat black.

Procedure

4. (a) Place the tester on a solid table free of vibration, in a location free of perceptible draft, and in a dim light.

(b) Run water, brine, or water-glycol solution into the bath to a predetermined level, which will fill the bath to $\frac{1}{8}$-inch below the top when the cup is in place. An overflow is permissible for water-level control.

(c) Firmly support the thermometer vertically halfway between the center and edge of the cup on a diameter at right angles to the guide wire, or on a diameter passing through the center of the cup and the pivot of the taper. Place so that the bottom of the bulb is $\frac{1}{4}$-inch from the inner bottom surface of the cup. If the old Tagliabue thermometer is used, immerse to well cover the mercury bulb, but not the wide body of the thermometer.

(d) Fill the glass cup with the sample liquid to a depth just $\frac{1}{8}$-inch below the edge, as determined by the leveling device.

(e) Place the guide wire or swivel device in position, and set the draft shield around the tester so that the sides form right angles with each other and the tester is well toward the back of the shield.

(f) If a guide wire is used, the taper, when passed, should rest lightly on the wire, with the end of the jet burner just clear of the edge of the guide wire. If the swivel-type holder is used, the horizontal and vertical positions of the jet are so adjusted that the jet passes on the circumference of a circle, having a radius of at least 6 inches, across the center of the cup at right angles to the diameter passing through the thermometer, and in a plane $\frac{1}{8}$-inch above the upper edge of the cup. The taper should be kept in the "off" position, at one end or the other of the swing, except when the flame is applied.

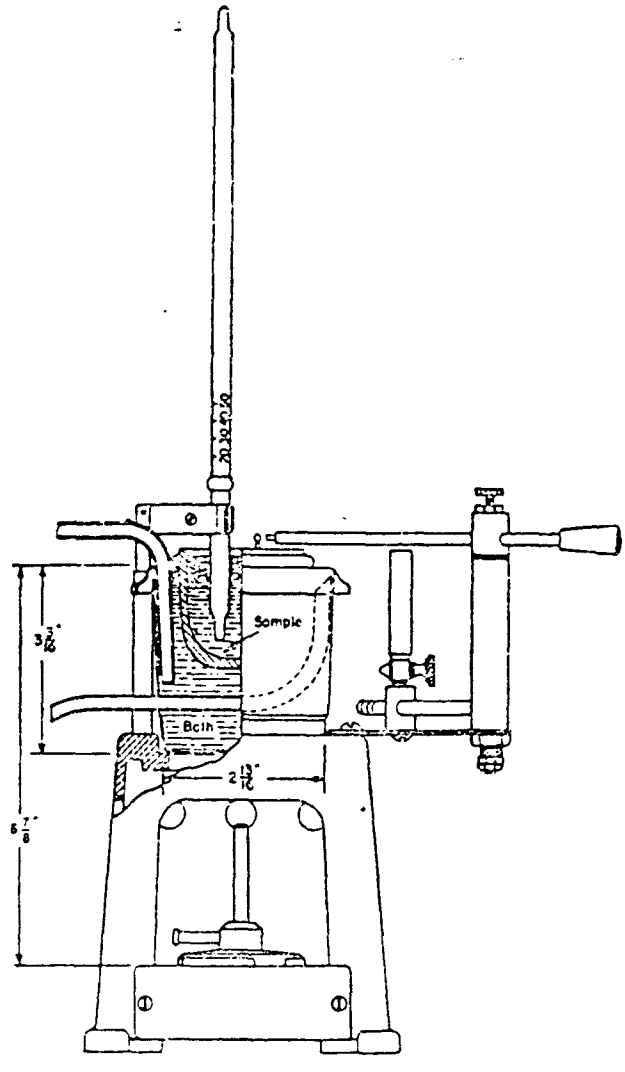

FIGURE 1—*Tag open-cup flash tester.*

(g) Light the ignition flame and adjust it to form a flame of spherical form matching in size the 5 32-inch sphere on the apparatus.

(h) Adjust heater source under bath so that the temperature of the sample increases at a rate of 2 ±0.5° F. per minute. With viscous materials this rate of heating cannot always be obtained.

Initial Test

5. Determine an approximate flashpoint by passing the taper flame across the sample at intervals of 2° F. Each pass must be in an

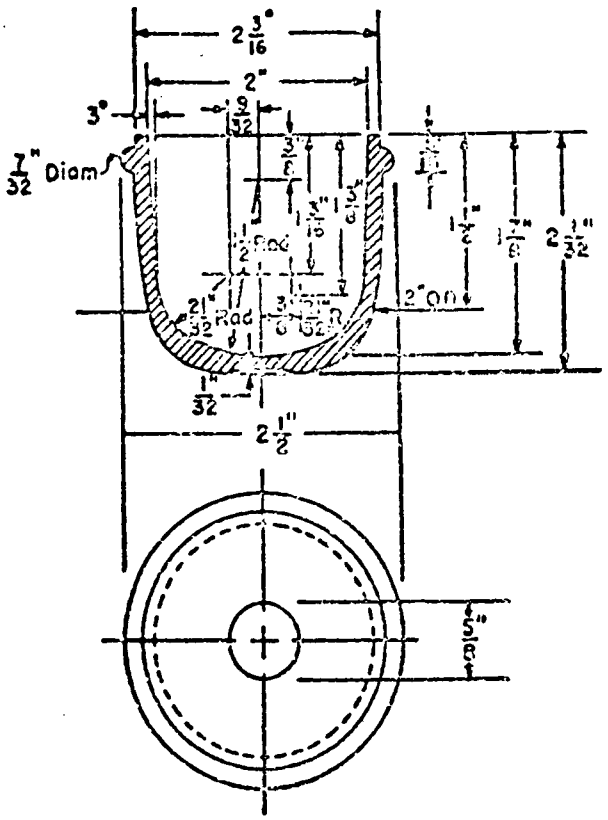

FIGURE 2—*Glass test cup.*

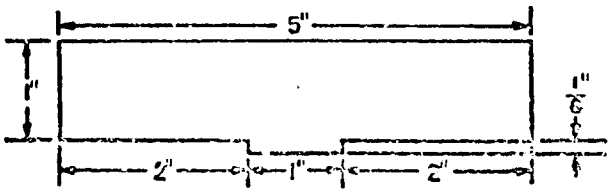

FIGURE 3—*Leveling device for adjusting liquid level in test cup.*

direction only. The time required to pass the ignition flame across the surface of the sample should be 1 second. Remove bubbles from the surface of the sample liquid before starting a determination. Meticulous attention to all details relating to the taper, size of taper flame, and rate of passing the taper is necessary for good results. When determining the flashpoint of viscous liquids and those liquids that tend to form a film of polymer, etc., on the surface, the surface film should be disturbed mechanically each time before the taper flame is passed.

Recorded Tests

6. Repeat the procedure by cooling a fresh portion of the sample, the glass cup, the bath solution, and the thermometer at least 20° F. below the approximate flashpoint. Resume heating, and pass the taper flame across the sample at two intervals of 5° F. and then at intervals of 2° F. until the flashpoint occurs.

Reporting Data

7. The average of not less than three recorded tests, other than the initial test, shall be used in determining the flashpoint and flammability of the substance.

Standardization

8. (a) Make determinations in triplicate on the flashpoint of standard paraxylene and of standard isopropyl alcohol which meet the following specifications:

(i) *Specifications for* p-*xylene, flashpoint check grade.* p-Xylene shall conform to the following requirements:

Specify gravity: 15.56° C. 15.56° C., 0.860 minimum, 0.866 maximum.

Boiling range: 2° C. maximum from start to dry point when tested in accordance with the method of test for distillation of industrial aromatic hydrocarbons (ASTM designation: D 850), or the method of test for distillation range of lacquer solvents and diluents (ASTM designation: D 1078). The range shall include the boiling point of pure *p*-xylene, which is 138.35° C. (281.03° F.).

Purity: 95 percent minimum, calculated in accordance with the method of test for determination of purity from freezing points of high-purity compounds (ASTM designation: D 1015), from the experimentally determined freezing point, measured by the method of test for measurement of freezing points of high-purity compounds for evaluation of purity (ASTM designation: D 1015).

(ii) *Specifications for isopropanol, flashpoint check grade.* Isopropanol shall conform to the following requirements:

Specific gravity: 0.8175 to 0.8185 at 20° C./20° C. as determined by means of a calibrated pycnometer.

Distillation range: Shall entirely distill within a 1.0° C. range which shall include the temperature 80.4° C. as determined by ASTM method D 1078.

Average these values for each compound. If the difference between the values for these two compounds is less than 15° F. (8.5° C.) or more than 27° F. (16° C.), repeat the determination or obtain fresh standards.

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(b) Calculate a correction factor as follows:

$X = 92 - A$

$Y = 71 - B$

$$\text{Correction} = \frac{X - Y}{2},$$

Where:

A = Observed flash of *p*-xylene, and

B = Observed flash of isopropyl alcohol.

Apply this correction of all determinations. Half units in correction shall be discarded.

Precision

9. (a) For hydrocarbon solvents having flashpoints between 60° F. and 110° F., repeatability is ±2° F. and the reproducibility is 5° F.

(b) If results from two tests differ by more than 10° F., they shall be considered uncertain and should be checked. The calibration procedure provided in this method will cancel out the effect of barometric pressure if calibration and tests are run at the same pressure. Data supporting the precision are given in Appendix III of the 1956 Report of Committee D-1 on Paint, Varnish, Lacquers and Related Products, Proceedings, Am. Soc. Testing Mats., Vol. 56 (1956).

§ 191.14 Method for determining extremely flammable and flammable solids.

(a) *Preparation of sample*—(1) *Granules, powders, and pastes.* Pack the sample into a flat, rectangular metal boat with inner dimensions 6 inches long x 1 inch wide x one-fourth inch deep.

(2) *Rigid and pliable solids.* Measure the dimensions of the sample and support it by means of metal ringstands, clamps, rings, or other suitable devices as needed, so that the major axis is oriented horizontally and the maximum surface is freely exposed to the atmosphere.

(b) *Procedure.* Place the prepared sample in a draft-free area that can be ventilated and cleared after each test. The temperature of the sample at the time of testing shall be between 68° F. and 86° F. Hold a burning paraffin candle whose diameter is at least 1 inch, so that the flame is in contact with the surface of the sample at the end of the major axis for 5 seconds or until the sample ignites, whichever is less. Remove the candle. By means of a stopwatch, determine the time of combustion with self-sustained flame. Do not exceed 60 seconds. Extinguish flame with a CO_2 or similar nondestructive type extinguisher. Measure the dimensions of the burnt area and calculate the rate of burning along the major axis of the sample.

§ 191.15 Method for determining extremely flammable and flammable contents of self-pressurized containers

(a) *Equipment required.* The test equipment consists of a base 8 inches wide, 2 feet long, marked in 6-inch intervals. A rule 2 feet long and marked in inches is supported horizontally on the side of the base and about 6 inches above it. A paraffin candle 1 inch or more in diameter, and of such height that the top third of the flame is at the height of the horizontal rule, is placed at the zero point in the base.

(b) *Procedure.* The test is conducted in a draft-free area that can be ventilated and cleared after each test. Place the self-pressurized container at a distance of 6 inches from the flame source. Spray for periods of 15 seconds to 20 seconds (one observer noting the extension of the flame and the other operating the container) through the top third of the flame and at a right angle to the flame. The height of the flame should be approximately 2 inches. Take three readings for each test, and average. As a precaution do not spray large quantities in a small, confined space. Free space of previously discharged material.

§ 191.16 Method for determining flashpoint of extremely flammable contents of self-pressurized containers

The apparatus used is the Tagliabue Open-Cup Flashpoint Apparatus as described in § 191.13. Some means such as dry ice in an open container is used to chill the pressurized container. The container, the flash cup, and the bath solution of the apparatus (brine or glycol may be used) are chilled to a temperature of about 25° F. below zero. The chilled container is punctured to exhaust the propellant. The chilled formulation is transferred to the test apparatus and tested in accordance with the method described in § 191.13.

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50 EAST 41st STREET, NEW YORK, N. Y. 10017

ROUTE TO

RETURN TO

TEST METHOD

BULLETIN NO 247-69 July 7, 1969

All Divisions	Aerosol	Automotive	Detergent and Cleaning Compounds	Disinfectants and Sanitizers	Insecticide	Waxes, Polishes and Floor Finishes

TENTATIVE METHOD FOR DETERMINING THE RELATIVE FLAMMABILITY OF AEROSOL FOAM PRODUCTS

The enclosed test method was developed by the Aerosol Division Consumer Hazards and Safety Subcommittee and was approved at the May 1969 Meeting in Chicago, Illinois by the Aerosol Division Scientific Committee, the Aerosol Division Executive Board and the CSMA Board of Governors.

This method should be inserted in the Aerosol Guide.

S. R. Goff
Chairman, Aerosol Division
Scientific Committee

A. A. Mulliken
Executive Director

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MORE

C S M A

TENTATIVE METHOD FOR DETERMINING THE RELATIVE FLAMMABILITY OF AEROSOL FOAM PRODUCTS

Introduction

Flammability is one of the more important criteria of an aerosol product. The Flame Propagation and Closed Drum Test procedures, which work so well with most aerosols, cannot be made to reliably assess the flammability of those products which are dispensed as stable foams. The present method is designed to fill this void.

Foam products may be sprayed or extruded. They may be of the stable type, semi-stable variety or quick-breaking compositions. Flammability may be brought about through the use of sufficient quantities of flammable or combustible liquids, flammable propellents, or both at once. It is because of these variations that no single method has appeared to be suitable for all foam products. Rather, a dualistic method, based upon two complementary procedures, is required.

The first of these procedures, known as the "Tower Test," is used to assess the rate at which a fixed volume of foam, with a specified surface area, can generate gas; and whether this gas is capable of forming a flammable mixture with air. In the second procedure, the "Trough Test," a flame is touched to the foam surface to determine if the composition is capable of flashing or sustaining a fire.

Apparatus

1. Apparatus for the Tower Method: The dimensions and other specifications are provided in PLATE ONE. The equipment consists of a base section, designed to hold a measured amount of product, over which is fitted a cylindrical chimney pierced with a vertical row of small holes. The suggested material is aluminum, although brass and stainless steel have also been used.

2. Apparatus for the Trough Method: The dimensions and other specifications are provided in PLATE TWO. The equipment consists of a simple flat trough, preferably formed from stainless steel sheet, of about No. 16 Gage (0.062") thickness.

Procedure for the Tower Method

1. The aerosol can is brought to equilibrium temperature by holding for at least 45 minutes in a water bath at 70°F. Tests are run for each of two "use conditions." The first test is conducted on a new can, and the second test on the same can after it has been discharged to 20% of the stated net weight of the contents of the can plus the weight of one test cupful of product. If there is insufficient material in one can for both tests, two different cans for each test may be used. In order to more nearly reproduce "use conditions tests are not conducted on the partially used cans until one hour after the cans have been discharged. Cans should be kept at room temperature (70°F).

OVER

Tentative Method For Determining the Relative
Flammability of Aerosol Foam Products - Continued

2. The sample can is shaken briefly and then discharged in the normal use position into the test cup. Care should be exercised when charging the test cup so that the air pockets are not occluded by the foam, thereby reducing the apparent volume of test material used. An adapter can be used to facilitate charging the test cup with foam.

3. When the foam sample is in the test cup, allow five to ten seconds, counted from the time the cup has been filled, for the foam to expand fully before proceeding to the next step.

4. Level the foam by drawing a spatula across the top of the test cup.

5. The tower unit, with a single length of stripable tape adjusted to seal its apertures, is set in place over the foam filled cup.

6. The apparatus is left undisturbed in a draft-free area for a five-minute holding time before proceeding to the test measurement.

7. After the five-minute holding time, and again without moving the apparatus, the protective tape is stripped from the touchholes at a uniform rate so that each hole is exposed at two-second intervals.

8. As each touchhole is exposed, starting from the top #15 hole, the flame of a small lighted gas jet is brought to the aperture. The flame should just touch the metal at the lower edge of the hole. Do not insert the flame into the hole.

9. A positive result is indicated when a flash occurs in the tube, and the hole number from where the flash occurs is recorded. Reported results should be the average of three tests for each sample.

Procedure for the Trough Method

1. The test samples used for this procedure are the same samples that are used for the Tower Test. The flame propagation tests are run in between the two sets of tower tests; i.e. after the initial tests, but before the cans have been discharged to 20% of the stated net weight of the contents of the can plus the weight of one cupful of product.

2. The aerosol can is brought to equilibrium in a water bath at 70°F.

3. The sample foam is shaken briefly and then discharged into the test trough. Care should be taken when charging the test trough, so that air pockets are not occluded by the foam, thereby reducing the apparent volume of the test material used.

4. When the trough has been filled, allow the foam to expand for five seconds before proceeding to the next step.

5. Quickly draw the edge of a large spatula across the top of the trough to level the foam.

6. Three tests are conducted for each foam sample. The first test, immediately after the foam has been leveled in the trough; the second test, after a two-minute holding time; and the third, after a five-minute holding time. The trough is left undisturbed and in a draft-free area for the designated holding times between tests.

MORE

Tentative Method For Determining the Relative Flammability of Aerosol Foam Products - Continued

7. At the end of the holding time, a small lighted gas flame is brought to the surface of the foam in the area to one-half inch from the end of the trough.

8. A positive test is indicated when the flame is sustained or propagated by the propellent gas or foam material after withdrawal of the flame.

Presentation of test results

A positive result for the Tower Test is reported as the average of three tests for each product. For example:

Test No. 1	Positive; at hole number 7.
Test No. 2	Positive; at hole number 9.
Test No. 3	Positive; at hole number 6.
Average:	Positive. Hole number 7.

A positive result for the Trough Test should be reported in terms of these observations:

P	Indicates propagation of flame.
12	Depicts distance of travel in inches.
SM	Indicates flame is sustained on surface.
RT	Shows flame is not sustained, but travels to end of trough and returns toward the starting point. Numbers may be added to indicate number of inches on return.

Typical positive results might then be recorded as:

P-3	Showing that the flame traveled three inches toward the far end of the trough and then went out.
P-14/RT-7	Indicating that the flame traveled to the far end of the trough and then returned about halfway back before extinguishing itself.
P-14/SM	Flame travels across trough and continues to burn.

These typical results of each test can be compared as follows:

Tower Test:	Touchhole No. 9 indicates a product more flammable than Touchhole No. 6
Trough Test:	P-14/SM is worse than P-14/RT-7 which, in turn, is more flammable than P-3.

OVER

Tentative Method For Determining the Relative Flammability of Aerosol Foam Products - Continued

Discussion

Because of the difficulty in relating the results of these tests to the degree of hazard involved in the transportation, warehousing or end uses of these foam type products, the two procedures are not to be used in order to determine if a product is to be called "Flammable" as distinguished from "Non-flammable." They are presented simply as the means by which the relative flammability of foam products may be assessed.

The procedures have been most thoroughly evaluated on simple shaving cream aerosols, and are certified only for application to this particular product type. In addition, the methods have been applied to a variety of stable foam items, such as upholstery cleaner, charcoal lighter, metal polish and hair shampoo, with good results. The procedures have not been evaluated with thermal foams, foaming gels, quick-breaking foams, anhydrous foams and reticulated gels; so that any conclusions made concerning the relative flammability of these products must be made according to the investigator's own recognizance.

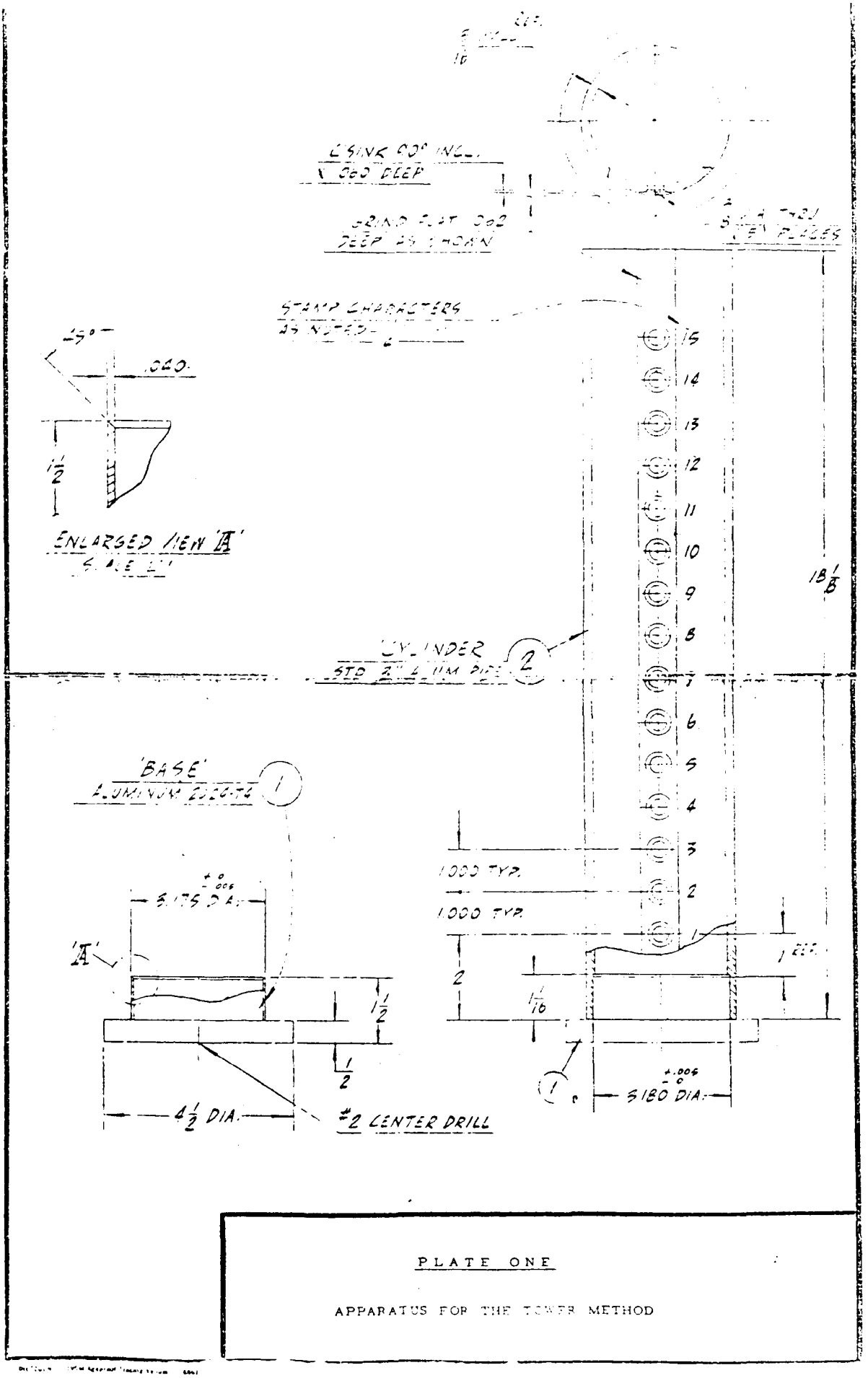

PLATE ONE

APPARATUS FOR THE TOWER METHOD

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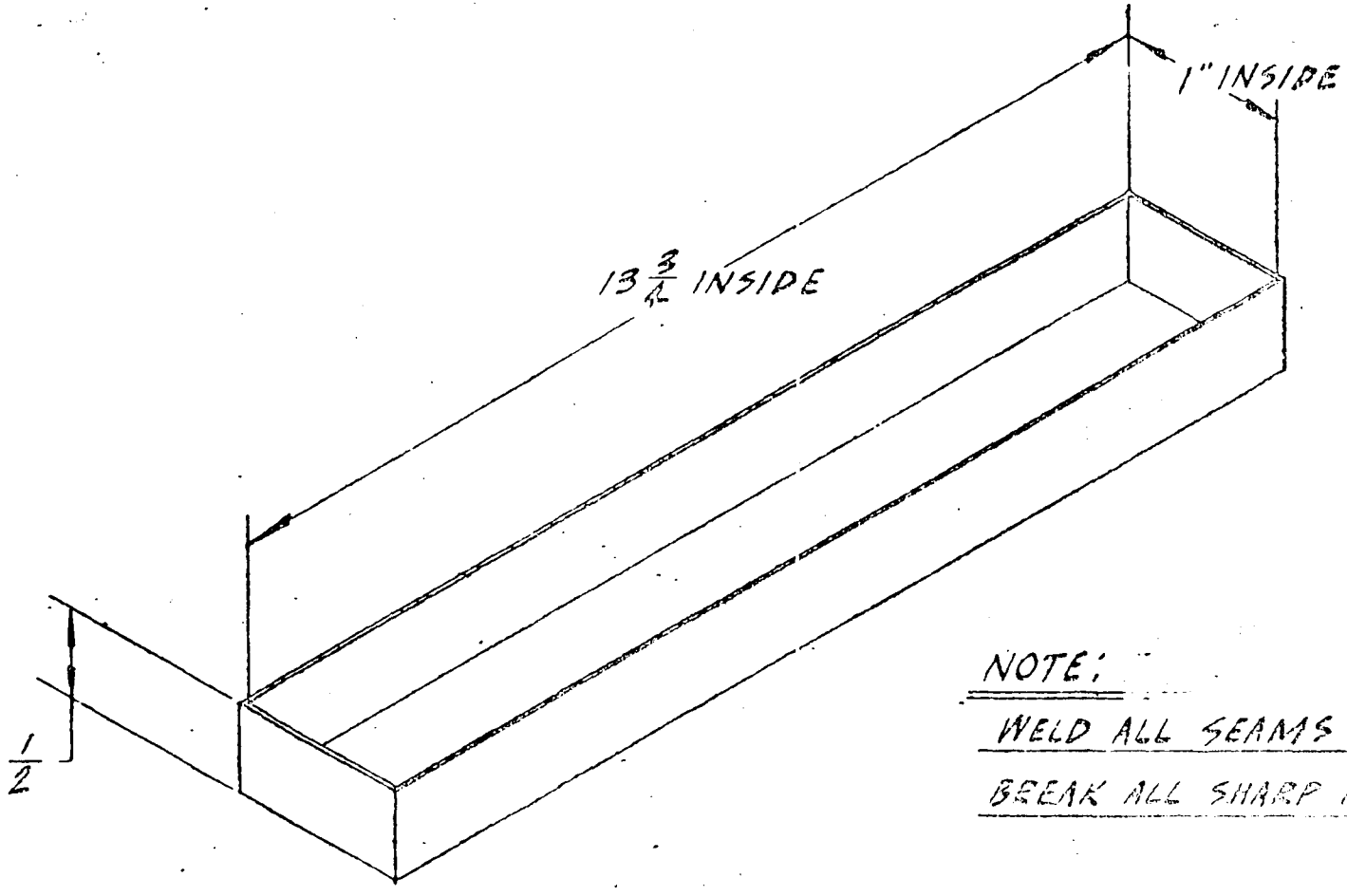

NOTE:

WELD ALL SEAMS

BREAK ALL SHARP CORNERS

PLATE TWO

APPARATUS FOR THE TROUGH METHOD

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MINIMUM FILLS

Minimum Commercial filling of Self Pressurized Packages offers many problems. Special committees of C.S.M.A. are constantly studying these problems. There will be occasional additions to this list. The following were recommended by the Commercial Practices Committee and adopted by the Aerosol Division.

(Originally published in the Proceedings of the 38th Midyear Meeting of the C.S.M.A., Page 53. with revisions passed May 18, 1953, May 21, 1956 and May 22, 1957)

RESOLVED, That the following size packages are recommended for use of aerosols or pressurized products: namely, insecticides, room deodorants, and artificial snow, having no greater than 20% non-volatile materials in the formulation, to contain a label contents not under 12 ounces (avoidupois).

1. American Can size 211 (2-11/16") diameter by 413 (4-13/16") body height.
2. Continental Can Company dome style can, size 211 (2-11/16") diameter by 413 (4-13/16") body height.
3. Crown Can size 211 (2-11/16") diameter by 413 (4-13/16") body height.
4. Continental Can concave style, size 211 (2-11/16") diameter by 413 (4-13/16") body height.
5. Crown Can size 214 (2⅞") diameter by 411 (4-11/16") body height.

RESOLVED, That the following size packages are recommended for use of aerosols or pressurized products: namely, insecticides, room deodorants, and artificial snow having no greater than 20% non-volatile materials in the formulation, to contain a label contents, not under 6 ounces (avoidupois).

1. American Can dome or flat top style, size 202 (2⅛") diameteh by 314 (3⅞") body height.
2. American Can size 202 (2-2 16") diameter by 406 (4-6/16") body height.
3. Crown Can tall style, size 202 (2⅛") diameter by 411 (4-11/16") body height.
4. Continental Can dome style, size 202 (2⅛") diameter by 314 (3⅞") body height.
5. Continental Can concave style, size 202 (2⅛") diameter by 314 (3⅞") body height.
6. Continental Can size 202 (2-2/16") diameter by 406 (4-6/16") body height.

The following cans to contain a label contents, not under specified weight:

1. American Can size 202 (2-2/16") diameter by 214 (2-14/16") body height. Not under 3 ounces (avoidupois).
2. Continental Can size 202 (2-2/16") diameter by 214 (2-11/16") body height. Not under 3 ounces (avoidupois).
3. Continental Can 211 (2-11/16") diameter by 510 (5-10/16") body height. Not under 14 ounces (avoidupois).
4. American Can 211 (2-11/16") diameter by 604 (6-4/16") body height. Not under 16 ounces (avoidupois).
5. Continental Can 211 (2-11/16") diameter by 604 (6-4/16") body height. Not under 16 ounces (avoidupois).

* * * *

The above is as reviewed and approved September 25, 1956 by the Administrative Committee, Aerosol Division, at a meeting held in Osterville, Mass.

Amendments voted May 20, 1957 by the Administrative Committee, Aerosol Division, at a meeting held in Chicago, Ill.

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MAILING AEROSOLS

POSTAL REGULATIONS

(Postal Manual United States Post Office Department, October 18, 1954)

AEROSOLS: MAILING PRIVILEGES

A new Postal Manual has been published. As it states in the forword: "This publication represents the first major overhauling of postal regulations in the history of the Postal Service. *****Many procedural simplifications have been made of benefit to all classes of mailers. *****We plan to publish a revised edition thirty days from now. In the meantime, we would appreciate any comments you might have."

* * * *

Limited excerpts from the Manual are given herewith.

Within certain limits all aerosols meeting I.C.C. specifications can now be mailed. Other products of our industries can now have mailing services. Much of the responsibility is left with the sender, who is held strictly accountable for sending non-mailable matter.

Sections of the Manual dealing with packing, marking, class of product, sizes and weight limits of packages, and rates, ARE IMPORTANT.

The complete Manual should be obtained at once by all those using the mails for their products, or recommending mailing for resale. Send sixty-five cents (65c) cash, check or money order to:

Superintendent of Documents
U. S. Government Printing Office
Washington 25, D. C.

Ask for: "POSTAL MANUAL, UNITED STATES POST OFFICE DEPARTMENT, CHAPTER I, CHAPTER II OCT. 18, 1954" (or later edition)

* * * *

Excerpts from Postal Manual, October 18, 1954

NON-MAILABLE MATTER

124.2—Harmful Matter

.21—General Provisions of Law. Any articles, compositions, or materials, which may kill or injure another, or injure the mails or other property, are non-mailable. This includes but is not limited to:

(a) All kinds of poison or matter containing poison.

(b) All poisonous animals, insects and reptiles.

(c) All disease germs or scabs.

(d) All explosives, inflammable material, infernal machines and mechanical, chemical, or other devices or compositions which may ignite or explode.

.22—General Examples of Harmful matter includes, among others, that which is liable to destroy, deface, or otherwise damage the contents of the mail bags or harm the person of anyone engaged in the postal service, such as caustic poisons (acids and alkalies), oxidizing materials, or highly flammable solids; or which are likely under conditions incident to transportation to cause fires through friction, through absorption of moisture, through spontaneous chemical changes or as a result of retained heat from manufacturing or processing; explosives or containers previously used for shipping high explosives having a liquid ingredient (such as dynamite) ammunition; fireworks; highly flammable liquids or substances; radioactive materials; matches; or articles exhaling a bad odor.

.23—Acceptability if Properly Packed. When authorized by the Postmaster General, various of the articles specified in this part as being nonmailable may be sent through the mails if they conform to special regulations as to preparation and packaging and if they are not out-

wardly dangerous, or of their own force dangerous or injurious to life, health, or property. See part 125.

MATTER MAILABLE UNDER SPECIAL RULES

125.1—Legal Restrictions

.11—Harmful Matter

a. Certain items which are barred from the mails, as set forth in part 124, may be mailed if prepared and packaged in accordance with special regulations of the Postmaster General. These are items which are not outwardly or of their own force dangerous or injurious to life, health, or property. Such items may be mailed in accordance with the standards and rules prescribed in part 125.

b. Notwithstanding any statement contained in this part, which covers generally some of the more common situations, the burden rests with the mailer to assure that he has complied with the law and that anything shipped by him has been properly prepared and packaged. The ordinary test of adequate preparation and packaging is whether the contents of a parcel are safely preserved under ordinary hazards of mail handling and transportation.

c. Products, materials and devices are created or modified with such frequency that the Post Office Department is unable to issue general rulings in advance to govern adequate preparation and packaging. Any mailer may, however, request the Post Office Department, in advance, for a specific ruling as to mailability of his item. The request should be addressed to the local postmaster, who will forward it to the Bureau of Post Office Operations, Mail Classification Division, Washington 25, D. C.

.12—Applicability of Other Laws

a. Although not unmailable, as defined in part 124, certain other items may be mailed only if they comply with applicable Federal laws and regulations.

b. Any special conditions or limitations placed on transportation or movement of certain things shall govern admissibility to the United States mails, when imposed under law by the U. S. Department of the Treasury, U. S. Department of Agriculture, U. S. Department of Commerce, U. S. Department of Health, Education and Welfare, Interstate Commerce Commission or any other Federal department or agency possessing legal jurisdiction.

.13—Penalties

Severe penalties of fine or imprisonment, or both, are provided by law, for anyone who knowingly deposits for mailing or delivery, or causes to be mailed or delivered, anything declared nonmailable under law. Failure to comply with the regulations of the Postmaster General, are prescribed in part 125, as to matter otherwise nonmailable, constitutes a violation of law.

125.2—Adequacy of Preparation and Packaging

.21—General Nature of Precautions Required

a. The restrictions against mailing of harmful matter, from which relief is granted by part 125, are intended to prevent damage or harm to postal and transportation personnel, to prevent damage or destruction of other mail and property, to avoid obnoxious odors, and to prevent the spread of disease and infection. Special preparation and packaging are required to protect against such contingencies.

b. Basic precautions, covered generally in this section, relate to the inner containers holding the harmful matter, internal cushioning and protection, and exterior packaging and marking.

.22—Liquids (Noninflammable) and Powders

a. Precautions to take in the case of liquids generally, pastes, salves, ink powders, pepper, snuff or other pulverized materials are against damage to mails and property from leakage and against caustic, irritant, toxic or soiling effect on mail handling personnel.

b. Containers shall meet any applicable Interstate Commerce Commission or other Federal specifications. Closures must effectively seal the contents against leakage. Friction tops must be fastened so that they will not come off under impact. This may be done by soldering, clips or otherwise.

c. Particularly where the containers of liquids are of glass or other breakable material, they must be packaged to withstand handling enroute. The container shall be cushioned inside the carton to absorb shock and impact. Where feasible, absorbent material shall be used, to take up all the liquid in case of breakage.

d. Poisons for scientific use, which are not outwardly or of their own force dangerous or injurious to life, health, or property, may be shipped between manufacturers, dealers, bona

fide research or experimental scientific laboratories and employees of the Federal, State or local governments who have official use for such poisons. Any such employee must be designated by the head of his agency to receive or send such poisons. The preparation and packaging of such poisonous articles shall be under the same conditions as applied to other articles covered by part 125.

.23—Combustible and Gaseous

a. In addition to precautions specified in section 125.22, containers of inflammable liquids must have sufficient air space to allow for vapor expansion under variations. This is to guard against bursting from internal pressure.

b. Safety matches of a strike-only-on-box or book variety may be mailed provided they are insulated adequately with aluminum foil, asbestos or provided with other fire retardant material. Strike-anywhere matches may not be mailed.

c. Compressed gas containers shall be of metal or nonshattering steel types, as required by the Interstate Commerce Commission or other Federal agencies. In addiiton to being cushioned to absorb shock, containers with release mechanism shall be protected against damage or accidental discharge in transit.

136.2

.21—Classification — Description

Airmail is mail carried by air and by the fastest connecting surface carriers, and is given the most expeditious handling in dispatch and delivery.

Airmail is not given special delivery to the addressee unless a special delivery fee is paid in addition to the airmail postage.

.22—Articles Acceptable

Mail of all classes, except that which may be damaged by low temperatures or high altitudes, is accepted for airmail.

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GLOSSARY OF TERMS USED IN THE AEROSOL INDUSTRY

(Tentative)

Active Ingredient—component of an aerosol formulation that produces the specific effect for which the formulation is designed.

Aerosol—a suspension of fine solid or liquid particles in air or gas, as smoke, fog, or mist. As defined by the Department of Agriculture, 100 percent of the particles in an insecticidal aerosol spray must have a diameter less than 50 microns and 80% of the particles must have a diameter less than 30 microns.

Aerosol Insecticides Storage Test—tentative official method (sponsored by CSMA) for determining storage characteristics of aerosol insecticides.

"Aerosol" Product — self-contained sprayable product in which the propellent force is supplied by a liquefied gas. Includes space, residual, surface coating, foam and various other types of products but does not include gas-pressurized products such as whipping cream. The term aerosol as used here is not confined to the scientific definition.

Aerosol Test Method for Flying Insects—official bio-assay method (sponsored by CSMA) using houseflies and OTA.

Auxiliary Solvent—liquid material used in addition to the primary solvent. Generally used to replace part of the primary solvent to produce some specific effect or as a matter of economics.

Chemical Attack—chemical reaction or solvent effect, causing failure or deterioration of plastic and rubber parts, organic coatings, metals, or lithography involved in the completed package.

Compatibility—broad term meaning that the various components of an aerosol formulation can be used together without undesirable physical or chemical results.

Concentrate—a basic ingredient or mixture of ingredients to which other ingredients, active or inactive, are added.

Container—metal, glass or plastic shell in which an aerosol formulation is packaged.

Corrosion—chemical alteration of the metal parts of container or valve. May lead to package failure and/or product deterioration.

Cosolvent—solvent used to improve the mutual solubility of other ingredients.

Crimp—one operation by which the valve may be permanently seated in some aerosol containers.

Density—weight of a given volume of material at a specified temperature.

Delivery Rate—weight of mixture discharged from dispenser per unit of time at a specified temperature. Usually expresed as grams second at 80°F.

Dispenser—metal, glass, or plastic shell with valve from which an aerosol or pressurized formulation is dispensed.

Eductor Tube—tubing connecting the lower portion of container or dispenser with valve. Sometimes miscalled "syphon tube" or "dip tube".

Foam Product—aerosol formulation containing a solution or emulsion which is dispensed in a highly expanded fluffy form by a liquefied gas propellent.

Head Space—volume in upper portion of dispenser not filled with liquid contents. Usually expressed as percent of total volume of dispenser at a specified temperature.

High Volatile Ingredients — see Volatile Ingredients

Inert (or Inactive) Ingredient—component of an aerosol formulation that does not con-

tribute to the specific effect of the formulation. In some cases, may be quite arbitrarily defined. For example, with insecticides, only the propellents are considered as inert ingredients.

Low Volatile Ingredients—see Nonvolatile Ingredients

Metering Valve—valve that delivers a definite, limited amount of aerosol formulation each time the valve mechanism is operated.

Nonvolatile Ingredients — components of an aerosol formulation with a vapor pressure less than atmospheric pressure (>14.7 lbs./sq. in. absolute) at a temperature of 105°F. Sometimes called *low volatile* components.

Official Test Aerosol, or OTA—a standard insecticide dispenser and formulation prepared by CSMA for use in Official Aerosol Test Method for Flying Insects.

Particle Size — diameter of solid or liquid particles expressed in microns (thousandths of a millimeter).

Pressure—internal force per unit area exerted by any material. Since the pressure is directly dependent on the temperature, the latter must be specified. The pressure may be reported in either of two ways:

(A) Absolute pressure—the total pressure with zero as a reference point. Usually expressed as pounds per square inch absolute (psia).

(B) Gage pressure—the pressure in excess of atmospheric pressure. Under standard conditions at sea level, the numerical value of the absolute pressure is 14.7 higher than that of the gage pressure. The gage pressure is usually expressed as pounds per square inch gage (psig).

Product Deterioration — chemical reaction or physical change within or between components considered compatible in original formulation. May be due to time or temperature of storage or other factors.

Product Formulation—specific formulation of completed product, including propellent (s). Usually expressed as weight/weight (w/w) percent.

Propellent—liquefied gas with a vapor pressure greater than atmospheric pressure (>14.7 lbs. per sq. in. absolute) at a temperature of 105°F.

Solubility—the extent to which one material will dissolve in another. Generally expressed as percent by weight. May also be expressed as percent by volume or parts per 100 parts of solvent by weight or volume. The temperature should be specified.

Solvent—liquid part of an aerosol formulation used to dissolve solid or other liquid parts.

Spray—the dispersed discharge from an aerosol-type dispenser in the form of small droplets or particles. Does not include foam-type discharge.

Spray Coating—aerosol spray product for surface application, which leaves a residual clear or pigmented finish for protective or decorative purposes.

Stability—ability of a product to maintain its original characteristics over extended storage periods, under normal variations in temperature conditions.

Synergist—an auxiliary material that has the property of increasing the effect of the active ingredient even though it may have little specific activity itself.

NOTE: In the case of insecticides, synergists are considered as active ingredients.

Valve—mechanism for discharging products from aerosol-type dispensers.

Viscosity—internal resistance to flow of a solid (powder), liquid or gas at a specified temperature. A definite measurement for the consistency of a material.

Volatile Ingredients—components of an aerosol formulation with a vapor pressure greater than atmospheric pressure (>14.7 lbs. per sq. in. absolute) at a temperature of 105°F. Sometimes called *high volatile* components.

Reported by the Sub-Committee on Definitions and Terms, April 25, 1956
Adopted by the Scientific Committee, May 20, 1956
Adopted by the Administrative Committee of the Aerosol Division, May 20, 1956

FILLER SAFETY MANUAL BY AEROSOL SCIENTIFIC COMMITTEE

An informal safety method for those who are now filling or plan to fill low pressure pressurized products either in the laboratory or in production.

I. RESEARCH AND EXPERIMENTAL WORK

(a) Storage of propellent and chemicals—

(1) *Propellent*: Cylinders should be stored in a cool, dry, accessible place. Care should be exercised in handling cylinders so that they are not dropped or allowed to strike each other violently. When cylinder is in use it should be held securely in place. In tapping cylinders all connectors should be leak free.

(2) *Chemicals*: Suitable storage for flammable chemicals and concentrates should be available. Glass containers should be handled carefully to avoid breakage.

(b) Handling of propellents and concentrates—

(1) *Propellents*: In handling those propellents with low boiling points laboratory personnel should be instructed carefully so as to prevent any propellent burns or freezing. Precautions should also be taken for the possible accumulation of propellents to a point where the normal oxygen content is decreased. Supply adequate vents.

(2) *Concentrate*: Proper ventilation should be provided for the handling of highly volatile and toxic liquids. Manufacturers labels should be observed for toxicity information concerning raw materials used. Instruct personnel not to smoke or have any burners working in the vicinity of the use of flammable liquids.

(c) Handling of sealing machinery—

All machinery in the laboratory such as crimpers and seamers should have adequate protection at those portions where accidents may occur. For example, all moving belts on seamers should have guards, protective shield or device should be installed on crimpers to eliminate the possibility of getting hand caught between crimping head and can.

In sealing valves onto glass containers care should be taken that machinery does not damage bottle thereby causing possible future hazard when bottle and contents are examined at elevated temperatures. All crimps and seams on containers should be carefully inspected before units are brought to higher temperatures thereby minimizing future accidents due to defective containers.

(d) Handling of containers—

All types of aerosol containers can be hazardous in the laboratory regardless of whether the containers are of plain glass, safety coated glass or metal. The following precautions should be observed:

(1) Guard against overfills. When using new type containers determine what is a safe fill before packing.

(2) Guard against defective containers. All glass containers should be inspected as to defects before use. All can seams should be inspected for visible flaws. Take care not to damage containers during the pack, such defects could cause serious accidents later on in the tests.

(3) When examining units in hot water tank adequate protection such as safety shields should be available.

(4) Personnel should always wear protective face shields when working with glass containers which are under pressure.

(5) Glassware under pressure should always be handled carefully regardless of the pressure. All glassware under pressure should be covered with a protective screen or coating.

(6) Guard against excessive pressures in all containers.

(7) Check storage oven mechanisms periodically to prevent possibility of over runs in temperature which may cause explosions with the units under heat storage tests.

II. COMMERCIAL FILLING

(A) Refrigeration Filling:

(1) Handling of Propellent and Concentrate

(a) *Propellent*

1. Safe methods of unloading cylinders.

[a] If cylinders are shipped by truck, building of a platform which will be level with the tailgate of the truck and level with the building floor would be advisable. Fork lift could then go into the truck and would not have to raise its platform more than six inches to unload the cylinders. Provisions should be made so that cylinders could not slip off the fork lift.

[b] When using a hoist to life cylinders off trucks or railroad cars the proper equipment should be used. Hoist, chains, hooks and runner beams should be selected to handle the weight of the cylinders plus a safety factor. Equipment should be inspected periodically for flaws. Personnel should be instructed in the proper use of the equipment. It should also be impressed upon those unloading that all grabbing hooks and chains are to be secure before unloading.

2. Safe methods of storing and tapping cylinders.

Some of the following suggestions taken from Du Pont Kinetic Technical Bulletin B-11 "Emptying One-Ton Shipping Containers for "Freon" Fluorinated Hydrocarbon Compounds".

[a] Cylinders are protected from excessive pressures due to heat by fusible plugs in container and valves which melt at 157° F. Never permit live steam or a direct flame to be applied to any part of the container.

[b] Store containers in a cool, dry, accessible place. Keep containers away from salt or other corrosive chemicals or fumes, as rusting will damage containers and cause valve hoods to stick. Containers must not be dropped nor permitted to strike each other violently. Containers should be securely blocked.

[c] Do not tamper with safety devices in the valve or container.

[d] Replace the brass protection caps on the valves of the cylinders to prevent dirt entering the valves and damage to the threads on the valve connections. Secure the valve hoods after container has been emptied.

If heat must be applied to propellent containers the following methods should be used:

[e] Heat by hot air heat either from steam coils, steam space heaters or electric resistance heaters. Do not immerse the container in a hot water bath or under any circumstances apply a blow torch or open flame.

[f] Another method of applying heat is by use of infra-red lamps. A clamp-on thermocouple on the container surface serves to control the lamps. Additional precaution should be taken against overheating by having a cut out switch on the lamp circuit which is actuated by the pressure of the propellent by means of a direct connection to the container outlet line during the heating period.

[g] Another factor to be considered in the handling of propellents is the excessive accumulation of propellent vapors at various points in the plant. Although propellent vapors themselves are relatively non-toxic the tendency will be for a decrease in the oxygen content of the atmosphere. Excessive vapors would probably accumulate on the floor around the filling line due to the high vapor density of the propellents. If care is not exercised the accumulation of propellent vapors may exceed a tolerable amount. The normal oxygen content of the air is 21%. When the oxygen content is less than 16%, life cannot be supported and a real hazard exists.

A possible solution to this problem would be the installation of an exhaust system along the floor.

(b) *Concentrate*

If concentrate ingredients are blended at the filling plant precautions should be taken as to the handling and mixing of the various ingredients in the concentrate. If ingredients are toxic adequate ventilation should be available e.g. ex

haust system. Regardless of what the ingredients may be, mixing should be done in enclosed tank to prevent splattering of material.

Concentrate tanks should be equipped with safety valves especially if concentrate is highly volatile. Safety valves set to open at a designated pressure relative to the concentrate being used would prevent excessive build up of pressure.

Suitable precautions should be taken if concentrate is flammable or toxic.

(2) Can Unscrambler

Safety devices should be installed if the possibility of injury to the operator setting the cans onto the unscrambler exists. An automatic shutoff in the event of jam ups could be installed.

(3) Point of filling

[a] Precautions should be taken to prevent splattering of concentrate or propellant onto operators, also for the elimination of harmful vapor of concentrates.

[b] Closed in area with clear plastic or glass shield in front of operator could be installed to prevent concentrate or propellant from splattering onto operators.

[c] Exhaust hoods should be installed over filling point of line to evacuate harmful vapors.

(4) From Filling Point to Valve Crimper

[a] Liquid Fill--overfilled cans are dangerous and should be avoided.

The following checks should be installed.

1. Constant check of metering devices on filling units.
2. Some sort of check of liquid contents of cans.

(a) An X-Ray level checker is now in use at a leading beer manufacturing company for checking underfilled cans. It is General Electric's X-Ray monitoring unit called "Hytafill." This unit can check 900 cans a minute and is accurate within plus or minus 1/64 inch of the required level. Hytafill can be modified to detect overfills as well as underfills.

The heart of the device is a crystal of Cadmium Sulfide about the size of a match head. This crystal acts on the signals from an 800,000 volt X-Ray tube. The crystal is placed at the desired height of fill level on one side of the conveyor line with the X-Ray tube on the other. When a can comes along which is not full to the proper level the Cadmium Sulfide crystal gets a charge from the X-Ray tube which changes it from a non-conductor to a conductor of electricity and starts an electric circuit operation. This current is relayed to an automatic air blast unit which blows an underfilled can off the line.

Other similar type devices may be available for overfill detection.

(b) The checking of can weight which is probably the most widely used system for checking contents in the can should certainly be in use if no automatic equipment is available. Such checks should be made as often as possible.

[b] Dangerous Vapors Protection

Here again there is a need for an exhaust system to eliminate dangerous vapors.

[c] Valve Crimper

Operators should be placed at valve crimper to eliminate from the line those cans which have an improperly crimped valve which may cause trouble in hot water bath.

(5) Hot Water Tanks

The following precautions should be taken at this most important point of aerosol filling operation.

[a] Shields over hot water tanks to protect against units which may explode or protective face shields for personnel.

[b] Suitable control of temperature of the bath. A dependable thermostatic control should be installed to keep the temperature of the bath at the desired level. Heating apparatus as well as heat control apparatus should be checked periodically.

[c] For added protection operators should be supplied with safety glasses.

(6) Spray Testing

All spray testing should be done in an exhaust hood. All operators should be instructed as to determining the direction of the spray from the valve depending on the valve being used to prevent spraying on themselves.

(7) Storage of Finished Units

[a] Finished units should be stored in a cool dry place if they are to be retained at the filling plant for any length of time before shipment.

[b] Suitable cartons and methods of stacking should be employed to prevent unnecessary damage to units.

(B) Pressure Filling:

In this section will be listed those safety features recommended for pressure filling equipment. All other safety measures as to handling of propellent and concentrate along with those recommended for the can unscrambler, the point of filling of the concentrate, at the valve crimper and after filling should be the same as for refrigeration filling.

Safety shields should be installed in front of the filler to safeguard against the possibility of container bursting. Safety guards should be installed on all exposed parts of machinery where it is possible for an operator to be injured. A close check should be kept on all metering devices to prevent the possibility of dangerous overfills. Units should be checked for overfills after filling either by an automatic checker or by the weighing of cans.

III. GENERAL PRECAUTIONS

(1) Suitable storage should be supplied both in the plant and in the laboratory for toxic or flammable substances. Manufacturers' labels should be observed for toxicity or flammability information.

(2) Adequate fire extinguishers should be located at vital points in the laboratory as well as the plant. For small laboratory fires, extinguishers such as carbon dioxide or carbon tetrachloride are probably more suitable since they are a little cleaner in operation.

(3) Suitable first aid equipment should be available both in the plant and in the laboratory. Personnel should be instructed as to the proper first aid treatment to be used for different types of injuries which may occur.

(4) Encourage safe practices by lectures or constant reminders such as posters on the value of safety procedures.

(5) Inspect new operations closely for any possible hazards and devise necessary means to guard against such dangers.

Reported by the Filler Safety Committee, May 1954

AEROSOL LIQUID DENSITIES

HYDROMETRIC DETERMINATION OF AEROSOL LIQUID DENSITIES

I. INTRODUCTION

A method is given for the determination of the liquid densities of aerosol systems by the use of a conventional hydrometer. A discussion of the safety factors to be considered is included.

II. METHOD

A sample of liquid aerosol is introduced into a glass pressure tube containing a hydrometer. The hydrometer is read at the desired constant temperature and a small correction deducted to give the density of the sample.

III. APPARATUS

A. Hydrometer

The instrument recommended for this work has a range of 1.000 to 1.600, the scale being readable from both sides of the stem (Figure 1). It is made by the Precision Instrument Co. and can be obtained from the A. H. Thomas Co., Philadelphia, Pennsylvania, under catalog number 6182 by specifying the range and the double scale noted above. Several of this model hydrometer have been hydrostatically pressure tested and on the average they have withstood pressures up to 350 psig, with a few withstanding as much as 500 psig, and a few only 250 psig. Since no safety hazard results from the failure of a hydrometer, it is not deemed necessary to hydrostatically test the instrument before use.

Before using a new hydrometer it should be calibrated with one or two pure liquids of known density at a known temperature. Convenient liquids are distilled water, "Freon-11" trichloromonofluoromethane and carbon tetrachloride. In the case of the latter two liquids, the same average correction (0.009) given in the next paragraph should be deducted from the actual scale reading.

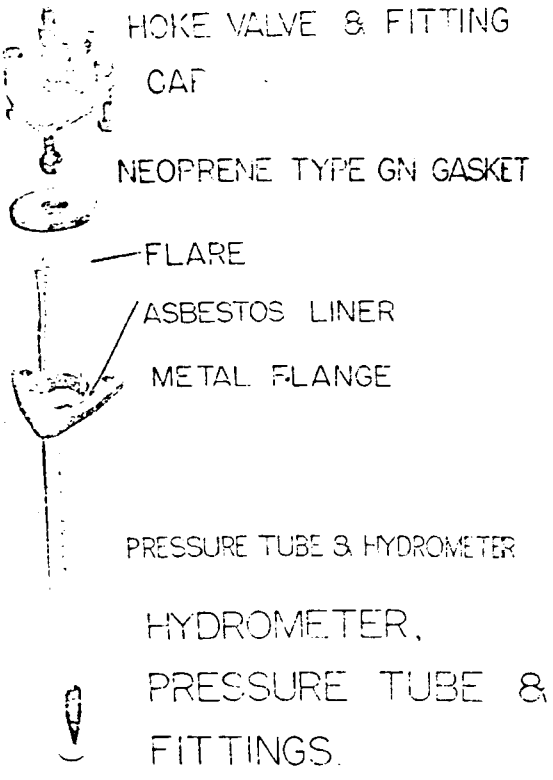

Figure 1

In obtaining the density of an aerosol system it is necessary to deduct a small, average correction, 0.009, from the actual reading of the hydrometer specified above. This correction is determined from calculations based on the physical dimensions of the hydrometer. The calculations for obtaining this correction are given in a paper submitted to the Safe-Fill Subcommittee of the Aerosol Scientific Committee of the CSMA and titled, "Aerosol Liquid Density Measurement". Increased accuracy can be had by using one or more hydrometers of smaller range than the one herein described. In this case new correction terms would have to be calculated for the hydrometers chosen.

B. Pressure Tube and Fittings

The tube used to hold the hydrometer and test liquid under pressure must be made of Pyrex brand glass pipe, one inch internal diameter. One end is sealed off in a round, test tube type end, and the other end terminated in a standard Pyrex brand glass pipe flare for use with the standard Corning metal flange fitting (Figure I). The tube is 15" in length overall; it should be carefully annealed. Such an item can be obtained on order from Lab Glass, Millville, New Jersey or any other such fabricator of laboratory glassware. Some of the fittings, namely the Corning metal flange, asbestos liner (JM-84-S) and the three bolts with nuts (see Figure I), can be obtained from the same source. A ¼" flat steel cap, drilled and tapped to accommodate a "Hoke" valve with standard brass fitting can be made by any machine shop. The gasket is made of ¼" flat Neoprene type GN gasket stock.

The Pyrex brand pressure tube should be carefully inspected for cracks or other flaws before use. A tube in good condition, properly assembled with the fittings described, should be able to withstand pressures well in excess of 500 psig. If the tube has no flaws, it is sealed with the flat metal cap and flange fittings, filled with a suitable liquid and subjected to a hydrostatic pressure test of 500 psig. The hydrometer is left out of the tube during this test since, as noted previously, most hydrometers are capable of withstanding pressures up to only 350 psig.

Each time the flange fittings and cap are put onto the tube, a great deal of care should be exercised to insure even tension on the three bolts holding the cap and gasket onto the flare surface of the tube. Unevenness in this tension can set up strains in the glass and weaken it considerably. It is to be noted that almost all of the failures with this type of glass equipment under pressure have occurred at the junction of the flared shoulder and the cylindrical portion of the glass tube, presumably due to strains set up by varying bolt tensions.

After the hydrostatic test has been made, the cap is removed, both the tube and hydrometer cleaned, the latter carefully inserted in the tube, and the flat metal cap again carefully bolted onto the pressure tube. Since glass can cut glass, care should be taken in putting the hydrometer in the pressure tube so as not to scratch either hydrometer or pressure tube. The hydrometer and tube are now ready for use.

C. Tube Suspension

A number of ways can be devised for the operator to raise or lower the pressure tube from behind the safety shield and the following arrangement is to be taken merely in the nature of a suggestion. A standard brass, flared fitting cap drilled with a 1/16" hole to accommodate 22 gauge copper wire (or its structural equivalent) can be used to provide a convenient means of suspending the hydrometer tube assembly. The wire is passed up, above the temperature bath, over a pulley or smooth steel bar and down outside of the safety shield where it can be operated conveniently (fig. 2). The advantage of such a system for raising and lowering the hydrometer assembly is twofold: 1) it insures the vertical alignment of the pressure tube, preventing the tube's interference with the floating hydrometer and 2) it affords the operator complete protection behind the safety shield without the necessity of temporarily exposing hands and arms during manipulation of the tube.

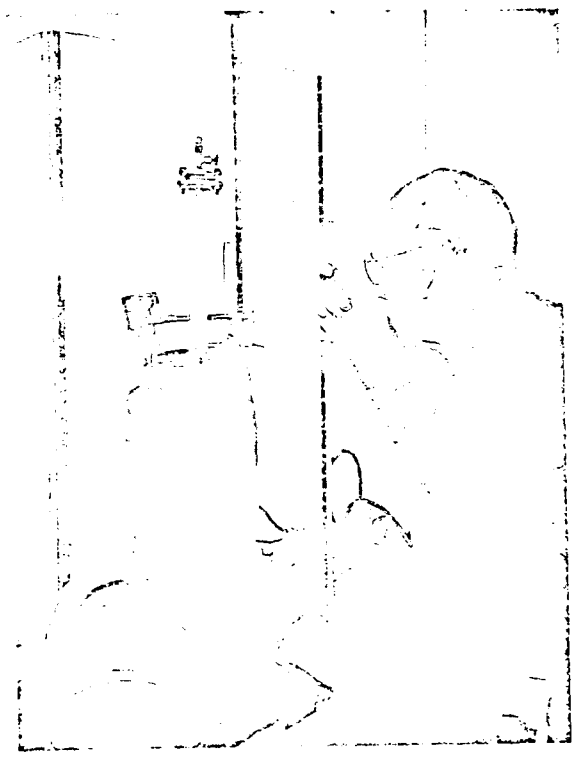

Figure 2

D. Temperature Bath

The bath container should be tall enough to accommodate the entire length of the pressure

tube and cap beneath the surface of the bath liquid (Figure 2). It should be constructed of non-rusting metal. The pressure tube can be partially removed from the bath long enough (20-30 seconds) to take a reading with no effect on the observation, even at 180°F.

For operation at the freezing point of water, clean, crushed ice is used with just enough water to fill the voids; no thermometer is needed here. At 70°F., or other temperatures around that of the room, the only additional equipment required is a thermometer having an accuracy of ± 1°F., a means of agitation and supplies of hot and cold water. Constant agitation is maintained all the time that the hydrometer and its enclosing pressure tube are in the bath. Appropriate additions of hot and cold water are then made so as to keep the temperature to within one degree of the desired value. This type of system can be operated at temperatures above that of the room though it is increasingly more difficult to do so the higher the temperature. If a number of density determinations are to be made at higher temperatures, the added convenience of a completely automatic, electrically operated thermostatic system is well worth the rather nominal investment. The necessary equipment can be obtained from any laboratory supply house at a total cost in the neighborhood of $60.00.

E. Wire Guard

The pressure tube should be enclosed in a stainless steel (18-8 or its structural equivalent) wire screen guard with silver soldered seams during all operations under pressure when not otherwise protected by the "Lucite" acrylic resin safety shield. A three inch diameter cylinder about 16" long is rolled out of a strip of screen and silver soldered to one end of the cylinder. The recommended screen mesh dimensions are 0.032" diameter wire and 0.093" spacing between wires (this corresponds to 1/8" x 1/8" openings measured on centers).

F. Leather Gloves

Leather Long Cuff No. 3502 gloves from Record Industrial Co., 3301 Arch St., Philadelphia 4, Pa., or other gloves of similar weight and quality are required.

G. Face Shield

Nitrometer Mask No. 1, code name IPCO, from Industrial Products, 2820 N. 4th St., Philadelphia 33, Pa., or a face shield of similar construction and strength must be used.

H. Safety Shield

A very effective safety shield can be constructed from three pieces of 1/4" "Lucite" acrylic resin and two 1 1/2" brass piano hinges 36" long (Figure 2). Two of the pieces of "Lucite" are rectangular, 20" x 36" and are hinged along their 36" edges to the third piece, also rectangular, 20" x 36". A center shield with two side panels such as this is self-supporting and offers protection over a larger sector of arc than would a single flat sheet of "Lucite".

IV. SAFETY

Any piece of glass equipment under pressure is subject to failure even though all precautions have been taken to minimize this possibility. Hence, it is essential that all the safety measures outlined in sections III, V, and VI be followed or that adequate alternates be developed. In actual practice it has been found that the frequency of failures of this type of equipment is extremely low. Nevertheless, any possibility of a failure, however remote, should be guarded against with full safety protection for the operator. It is with these considerations in mind that the safety precautions for this procedure have been written.

V. PROCEDURE

The hydrometer and pressure tube assembly is fastened into the wire screen guard with three evenly spaced loops of number 16 copper wire or its structural equivalent. The operator puts on the face mask and leather gloves and then proceeds to remove the air from the pressure tube. This is done by evacuating to less than five millimeters of mercury or by flushing with either "Freon-12" dichlorodifluoromethane vapor or, preferably, the vapor of the propellent present in the mixture whose density is to be determined.

The pressure tube is then connected to the aerosol container by means of copper tubing and flare nuts. It is filled by opening the valves on the two containers (the sample must be taken from the liquid phase of the sample container). The transfer must be performed only when the sample is at room temperature or below; it may be facilitated, however, by cooling the pressure tube in ice water. The pressure tube is filled to the point where the hydrometer floats half way up the length of the tube and the aerosol container is then removed.

Next, the pressure tube is connected to the pulley device and gently lowered into a bath at room temperature or below (Note 1), situ-

ated behind the safety shield as shown in the Figure 2. The three copper wires holding the pressure tube to the wire screen guard are then disconnected using gloved hands and the pressure tube and contents allowed to equilibrate for a period of ten minutes (Notes 2 & 3). Operating the pulley wire from the other side of the safety shield, the operator draws the pressure tube up high enough for a reading of the hydrometer meniscus (Notes 4 & 5). As soon as the reading is completed the tube is again lowered into the bath, where it is allowed to remain for another five minutes at which time a second reading is taken in the manner just described. This process is repeated till the readings are constant (Note 6).

When the readings are completed, it is important that the hydrometer and pressure tube first be allowed to cool to room temperature or below (Note 7). Without removing the tube from behind the safety shield, the pressure is relieved by opening the "Hoke" valve, using the gloved hands. Finally the equipment is taken apart and cleaned.

VI. NOTES

1) The bath temperature should be at room temperature or below to insure that the pressures within the pressure tube will be below 100 psig. in all cases. This requirement is imposed for safety reasons since in the following step the gloved hands are briefly exposed during disconnecting of the wire screen guard from the pressure tube.

2) The level of the bath liquid should be above the valve or the cap of the pressure tube in order to assure proper thermal equilibrium.

3) When the operator is completely protected by the safety shield, the face mask and gloves may be removed.

4) If the pressure tube is lifted only sufficiently to read the hydrometer, no difficulty will be encountered in lowering the pressure tube into the wire guard at the end of the reading.

5) Care should be taken to avoid parallax when reading the bottom of the meniscus surrounding the hydrometer stem. In addition the pressure tube should be alligned vertically so as to allow the hydrometer to float freely at the time of taking the reading. The type of suspension previously described along with the pulley arrangement will automatically take care of this latter requirement.

6) Ten minutes is usually sufficient for the attainment of thermal equilibrium. However, complete assurance on this point is obtained by this method of comparing successive readings. An idea of the degree of importance temperature has upon the readings can be obtained from the fact that changing the temperature of a 50/50 solution of "Freon-12" dichlorodifluoromethane — "Freon-11", trichloromonofluoromethane from 70° to 80°F. will reduce the density about one per cent. At 180°F. a temperature rise of only 5°F. will lower the density by the same amount.

7) This may be done either by allowing the tube to cool over night in the bath with the heat supply shut off or by lowering the tube into an auxiliary container of ice water. Whichever choice is taken, the operator must be completely shielded by the safety shield at all times until the tube has been cooled.

DETERMINATION OF SPECIFIC GRAVITY OF AEROSOLS PYCNOMETER METHOD

A pycnometer has been developed, which is suitable for measurement of the densities of liquids at temperatures above their normal boiling points.

Description of Apparatus

The apparatus consists of a high pressure aerosol bomb of approximately 500 c.c. capacity, with a bleed valve at one end and a graduated Saran tube and needle valve at the other. There is a steel nipple about four inches long between the Saran and needle valve to provide a small volume as protection against liquid pressure. Precautions should be taken during construction of the pycnometer to prevent dead spaces where air might be trapped, particularly by the nipple entering the top of the bomb, and where liquid might be trapped above the Saran tubing. A photograph of the apparatus is attached.

Calibration

The apparatus is evacuated and weighed. It is then filled with distilled water through the bleed valve until the liquid level is visible in the Saran tubing, and the height of this level in the tubing is read after bringing the apparatus to temperature in a thermostated bath. The apparatus is again weighed, and the volume calculated from the weight of water and its known density. The volume of the Saran tubing per unit length may be calculated from its inner diameter, or may be measured by filling a known length with water, and weighing the water.

Precautions

1. Care must be taken at all times to prevent development of liquid pressure in the apparatus, and particularly for determinations below room temperature where the liquid may expand before or during weighing.
2. The apparatus must be handled gently when charged, particularly at lower temperatures, as the Saran tubing is easily snapped under these conditions.
3. Consideration must be given to the pressure limits of the apparatus, and particularly of the Saran tubing, since its pressure limits decrease as the temperature is raised.

Directions for Operation

a) The apparatus is evacuated (to less than 2 mm. pressure) through the needle valve above the Saran tubing. Any air left in the apparatus may cause a bubble with a consequent error in the measurement.

b) The apparatus is now detached and weighed evacuated.

c) The apparatus is connected to the formulation container and the connecting line pumped to vacuum.

d) The bomb is placed in a water-ice bath and filled through the needle valve liquid phase.

e) The bomb is removed from the ice-bath, *the bleed valve opened, and then the needle valve closed.* The bleed valve is left open until the liquid level appears in the Saran tubing. It is important to make the operations in the above order to prevent development of liquid pressure.

f) The apparatus is raised to the temperature desired, with bleeding as necessary to keep the liquid level in the Saran. For measurements above room temperature, the whole apparatus should be thermostated to prevent condensation in the apparatus above the liquid level.

g) When temperature equilibrium is reached, and any bubbling due to vaporization has ceased, the liquid level is read and the apparatus and sample are dried and weighed. The bomb can be used for runs at 0° C., in which case the bottom of the bomb may be chilled in dry ice before weighing, as protection against development of liquid pressure.

h) The bomb may be raised to successively higher temperatures, with bleeding as necessary, to obtain density data over the desired temperature range.

Calculations

$$\frac{\text{Wt. apparatus full} - \text{wt. evacuated}}{\text{Total volume}} = \text{Density}$$

Errors

Measurements made by this method are subject to the following possible errors:

1) The usual experimental errors, such as weighing, thermostatic control, etc., in the calibration and determination.

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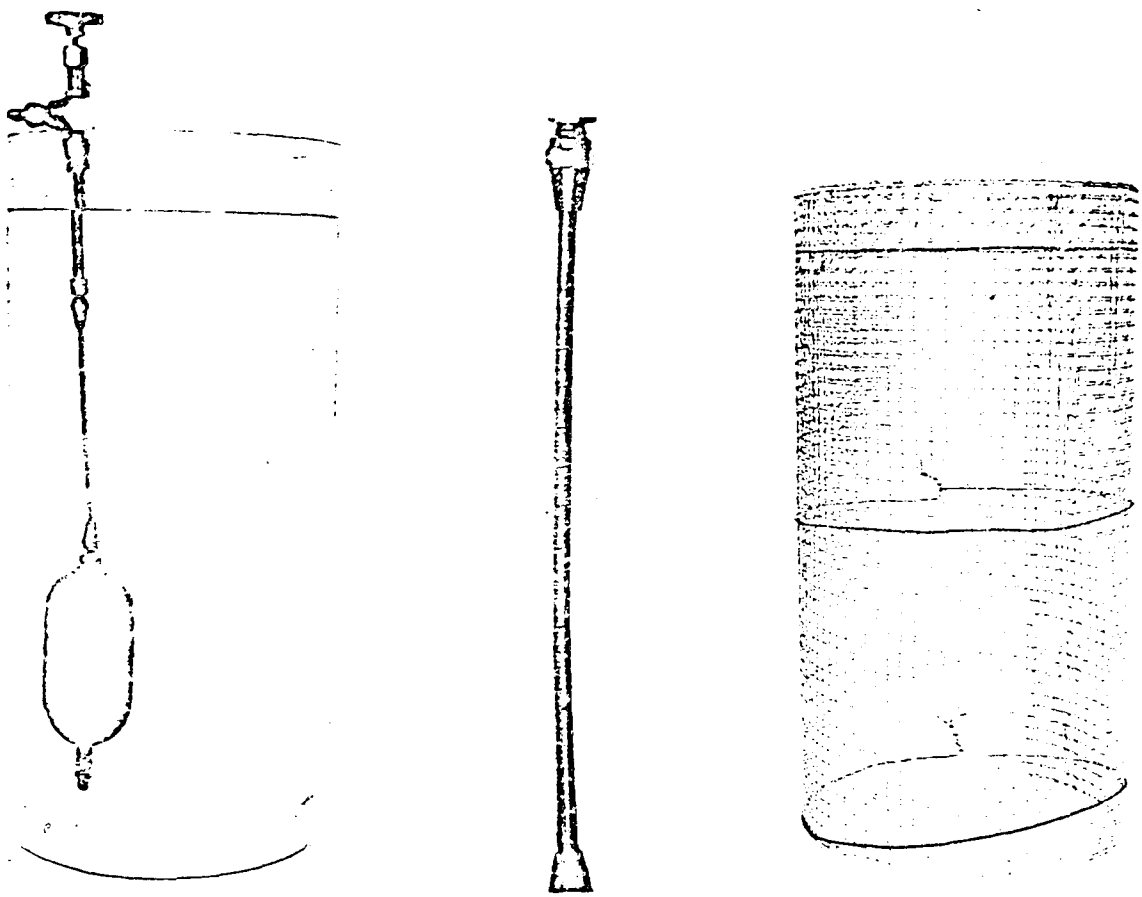
2) Any liquid remaining above the visible liquid level will cause an indeterminate error. This error should be small if the apparatus is properly constructed.

3) The change of volume of the apparatus with temperature and with pressure. This may cause an error in the density value on the order of 0.0004 gm./cc.

4) The vaporization of part of the sample into the free space above the liquid level. The error in density from this source will depend upon the vapor density of the substance being measured, but will be on the order of 0.0005 gm./cc. for aerosols.

5) The air remaining in the apparatus after evacuation may form a bubble below the liquid level. Assuming the apparatus to be evacuated to 2 mm. pressure of air, and all of the air to be trapped below the liquid surface, the error in density would be approximately 0.0001 gm./cc. for a substance with three atmospheres absolute vapor pressure. This may be minimized by evacuation to a lower pressure or by sweeping out the apparatus with a condensable gas before evacuation.



Accepted by the Scientific Committee, November 6, 1953

SAFE FILL FOR AEROSOLS

The committee reported as follows:—

"After consideration of all the factors involved, the Safe-Fill Sub Committee proposes that the following recommendation be submitted to the Aerosol Administrative Committee: *An aerosol container is considered to have a safe-fill if there is present 7½% headspace within the container when the contents are heated to 130° F.*"

The complete report may be seen at the Executive Office of the CSMA. Copies are not available to be mailed.

Reported by the Safe Fill Sub-Committee, December 5, 1955
Adopted by the Scientific Committee, May 20, 1956
Approved by the Administrative Committee of the Aerosol Division, May 20, 1956
Approved by the Board of Governors, May 22, 1956

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METHOD FOR INTERNAL PRESSURE DETERMINATION OF AEROSOL PRODUCTS IN LIGHT WEIGHT METAL CONTAINERS

Introduction

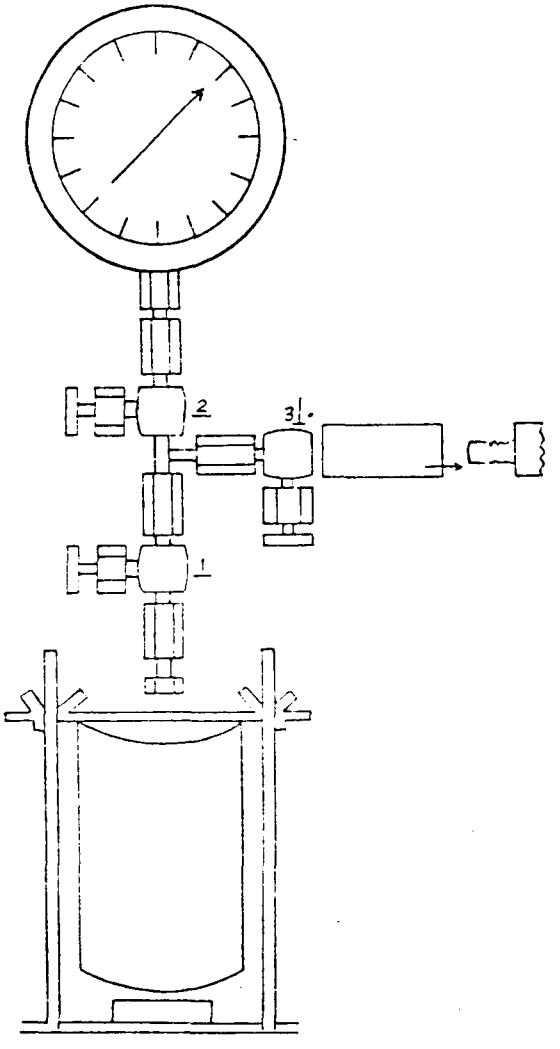
The purpose in developing this method is to make available to industry, the Bureau of Explosives and other Governing Agencies, a proven method of measuring the true and complete pressure of Aerosol Containers without adding or detracting in any way from this pressure.

Apparatus Required

1. Can piercing pressure measuring device manufactured by Builder's Sheet Metal Works, 110 Wooster Street, New York 12, New York.
2. Constant temperature water bath with automatic temperature control having a maximum temperature variation of plus or minus 0.5°F. Must have operating range of from 70°F. to 130°F., and must be at least 10" deep and 8" wide.
3. Two thermometers, gravity A.S.T.M. =12F (−5°F to +215°F.) with 0.5°F. graduations.
4. Gas supply, preferably through pressure regulator. Gas should be inert, but must be unliquifiable under the conditions of the test. Nitrogen,, compressed air, monochlorodifluoromethane and carbon dioxide have been successfully used.
5. Stop watch and/or timer.
6. Access to a dead weight tester for gage calibration. (This may subsequently be eliminated in lieu of a standard pressure package if the latter proves feasible).
7. Barometer, mercury.
8. Silicone stopcock grease.

Procedure

Upon receipt of apparatus, standardize gage over entire pressure range on dead weight tester, correcting for barometric pressure to sea level. This will detect any deficiencies inherent in the gage caused by shipping, assembling and etc.



Loosen wing nuts holding top plate, and raise to allow insertion of a pressure can. Adjust lower wing nuts to about 1 16" below

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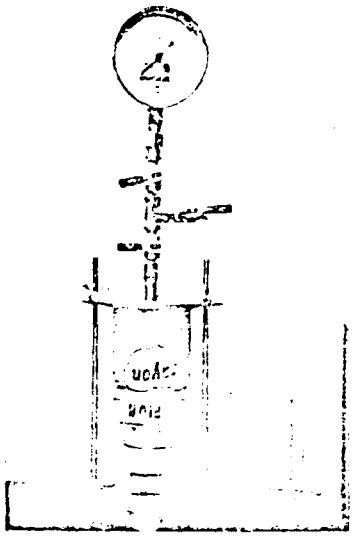

can level. Screw gage assembly upward, in top plate, so that piercing pin is fully retracted to allow plate to firmly contact bottom seam of can. Screw light film of silcone grease to can bottom to lubricate rubber seal. Screw top wing nuts down tightly. All needle valves should be closed at this point. Invert apparatus and quickly screw gage assembly down snug, piercing can. Immerse complete apparatus in 130° water with water level covering all screw joints. Open needle valves (1) and (2) and observe all connections for leaks. The apparatus must be completely leak free. Remove can from apparatus. Place test can in apparatus and pierce in the same manner as before. Immerse can in constant temperature water bath with water level just below valve (1). Container should be surrounded by at least two inches of water on all sides. Place ASTM thermometer alongside can, and second thermometer in center of water body. Readings of both thermometers should agree within 0.1°F. If they do not, inadequate agitation of bath is indicated and must be corrected before proceeding. Containers must be held at constant temperature as follows:

12.5 oz. capacity or less — non aqueous product — 30 min.

12.6 oz. capacity or more — non aqueous product — 60 min.

5 oz. capacity or more — aqueous product — 60 min.

After half of immersion time has elapsed, quickly remove container and agitate with 6 vigorous up and down shakes and replace in water bath. Attach prepressurizing line to quick disconnect, open valve (2) and prepressurize gage to approximately 5 p.s.i. below estimated container pressure by careful control of valve (3) or with pressure regulator. Close valve (3) tightly. After full immersion time has elapsed, carefully open valve (1) very very slightly until gage needle reacts, just perceptively. Close valve (1) and if gage needle moved upward, increase the prepressurizing pressure about 2 p.s.i. If gage needle moved downward, decrease prepressurizing pressure about 2 p.s.i. Repeat careful cracking of valve (1), until gage indicator reacts slightly a second time. Repeat change in prepressurizing pressure until needle movement reverses or does not occur. If no movement occurs, open valve (1), record gage reading, and barometric pressure. If movement reverses, increase or decrease pressure by 1 p.s.i. depending on indicator movement. Open valve (1) and record gage reading. Record barometric pressure, and correct to sea level. This is the true internal pressure of the aerosol. If a pressure is desired at another temperature, the bath may be adjusted and the pressure obtained at the second temperature in the same way. Once the measurements are obtained on a container, the pressure may be gradually relieved by closing valve (2), and opening valves (1) and (3), then disconnecting apparatus from can. Three cans of a product should be checked and the average reading taken as the final pressure.

Approved Special Can Pressure Determination Committee, October 6, 1956
Adopted Scientific Committee, December 3, 1956
Adopted Administrative Committee, Aerosol Division, December 4, 1956

METHOD FOR DETERMINING THE INTERNAL PRESSURE OF GLASS AEROSOL PRODUCTS

Introduction

Pressure is one of the most important criteria of an aerosol product. For the glass aerosol in particular an increase in internal pressure provides greater latitude in formulation, improved operating characteristics at slightly below room temperatures and the use of less expensive propellent combinations. Beyond a certain point, however, increasing pressure creates a hazardous condition from the standpoint of possible breakage and effects thereof.

Since the I.C.C. requires a metal container for compositions having pressures above 25 lbs. psi-gauge at 70°F, this value becomes the limiting pressure for glass aerosols. There is a very definite trend to limit pressures to 15 lbs. psi-gauge at 70°F for uncoated glass aerosols and specify plastic sheathed glass for pressures up to the 25 lbs. psi-gauge maximum at 70°F. These figures are also supported by glass bottle suppliers, major fillers and most merchandisers.

No standard method for determining the internal pressure of glass aerosols has been presented to the industry. One is urgently needed. Ideally, such a method should be simple and rapid; suitable for production control. The present method appears to have these qualifications.

Apparatus

1. *Pressure Gauge Adapter*: The specifications of this adapter are given in PLATE ONE. The suggested material is stainless steel, although brass has been used.*
   The tapered section is designed to make gas-tight contact with the upper stem of glass aerosol valves by insertion within the stem bore. Where the hole through the upper stem is not circular in cross-section, the use of a rubber sealing washer is suggested.**
2. *Pressure Gauge*: This gauge is shown as part of the assembly in PLATE TWO. A two-inch laboratory test gauge is suggested, range 0-60 lbs. psi-gauge graduated in one pound divisions. The thread on the gauge stem must match that on the pressure gauge adapter. The recommended gauge is U. S. Gauge Figure 500S, 2", $1\frac{1}{8}$" lower make connection, 0-60 lbs., specification No. 28208.
3. *Constant Temperature Water Bath*: The bath should have a stirring apparatus and should be of sufficient size to hold several dispensers. It should be deep enough to allow these dispensers to be completely submerged and surrounded with at least one inch of water. This will require a perforated shelf or a screen above the floor of the bath. The temperature should be 70° ± 0.50°F.
4. *Barometer*: Any standard model.

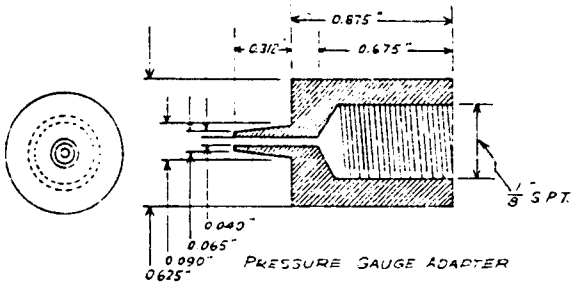

Plate One

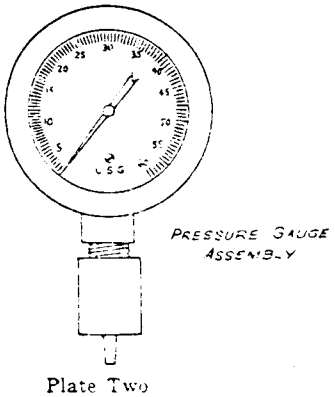

Plate Two

EID12045

Procedure

1. Place uncoated glass aerosols in water bath at least thirty minutes before testing. Plastic sheathed aerosols should remain in the bath for at least one hour prior to testing.
2. Shake dispenser vigorously under water in the bath for one minute. Insert gauge assembly, actuate valve, and shake assembly until pressure reading becomes constant. Record pressure.
3. Remove gauge assembly and shake aerosol under water for one minute. Reinsert gauge system, actuate, and record pressure; shaking as before.
4. Repeat Step 3 to obtain a third pressure reading.
5. Record barometric pressure.

Calculations

The three pressures obtained in this procedure will represent a series which diminishes in fairly linear fashion. In order to arrive at the true pressure, these readings are extrapolated to give an "0"th reading, as in the example:

First Reading:	14.0 lbs. psi-gauge
Second Reading:	13.7 lbs. psi-gauge
Third Reading:	13.4 lbs. psi-gauge

Extrapolated "0"th Reading:
14.3 lbs. psi-gauge.

This is the true pressure, under the conditions of measurement.

The barometer is then read and the gauge reading corrected to give the true gauge pressure if necessary.

Discussion

Although the apparatus is designed to provide as small an internal volume as possible, still, a certain amount of gas or pressurized liquid is withdrawn from the test dispenser at each reading. For airless samples the change in pressure between readings is due only to a slight decrease in propellent concentration in the liquid phase, as required to re-establish thermodynamic balance. For samples containing air, both air and propellent gas are withdrawn from the solution to being the pressure of the expanded head space to new equilibrium condition. Thus the difference of pressure between readings is greater for air-containing samples. In very extreme cases the difference may amount to 2.0 psi. at 70°F. and the pressure differential begins to swing from a linear relationship to a hyperbolic function as a limiting case.

The accuracy of results obtained using the extrapolation method has been checked using a large number of compositions. In such tests the formulations were prepared in metal containers fitted with pressure gauges. When pressures were determined using the procedure described above, variations between the known and extrapolated "0"th reading were 0.2 psi. or less. The greatest error was with products of high air content.

It is felt that results obtained by this test method are accurate to within $\pm$ 1.0 psi. at 70°F.

Modification of Method

This procedure can be adapted to production or routine pressure checking with but slight loss in accuracy.

For a given production run an average figure is determined by experiment for the pressure increment between the "0"th and first readings. This increment is then automatically added to all other consistent first readings throughout the run. Variations of the first reading will in themselves be indicative of production anomalies, such as faulty evacuation of bottles before pressure loading, or the presence of entrapped air resulting from below normal mix temperatures during refrigeration filling operations.

* A suggested source for pressure gauge adapters is

The Modern Machine Shop
123 North Hazel Street
Danville, Illinois

** The adapter has been used successfully with valves for glass aerosols made by

The Risdon Manufacturing Company
Precision Valve Corporation
Valve Corporation of America, Inc.

Approved by Project Committee on Pressure Determination of Glass Aerosols, May 20, 1956
Approved by Glass Aerosol Sub-Committee, May 20, 1956
Adopted by the Scientific Committee, May 20, 1956
Approved by the Glass Aerosol Advisory Committee, May 20, 1956
Adopted by the Administrative Committee, Aerosol Division, May 20, 1956

METHOD FOR DETERMINING THE INTERNAL PRESSURE OF AEROSOL INSECTICIDES AND ROOM DEODORANTS

Introduction

To conform with certain ICC Regulations, low pressure aerosol formulations of insecticides and room deodorants (20% nonvolatiles, or less) must have internal pressures not greater than 40 psig. at 70°F. Pressure measurements also provide a rapid, though rough, method for checking on filling ratios and filling procedures. Abnormally high pressures may indicate faulty evacuation of cans before pressure filling and the presence of trapped air resulting from below normal propellent temperatures during refrigeration filling.

Many methods of varying degrees of complexity have been devised to determine pressures. All are time consuming and do not lend themselves to mass production. In general, pressures taken to the nearest pound are considered sufficient for most purposes.

Because pressure is a function of temperature, it is very important that the contents of the can reach the standard working temperature of 70°F. A constant temperature water bath with suitable agitation is the best method of meeting this requirement. The samples to be measured require a minimum of 15 minutes in the bath before testing.

The measuring system should be constructed in such a way that the pressure is determined through the dispenser valve and does not require destruction of the dispenser. The system is prepressurized with nitrogen or other suitable gas.

The dispenser may be in either an upright or inverted position during measurement of the pressure. Three shut-off valves are located in the measuring system. One is positioned immediately below the gage. Below this valve is a tee with the opening to the outside controlled by the second valve. This opening is for prepressurizing and for cleaning out the line back to the valve block by sweeping with compressed air or gas. The third valve is in the line preceding the valve block and is also used to prepressurize the system.

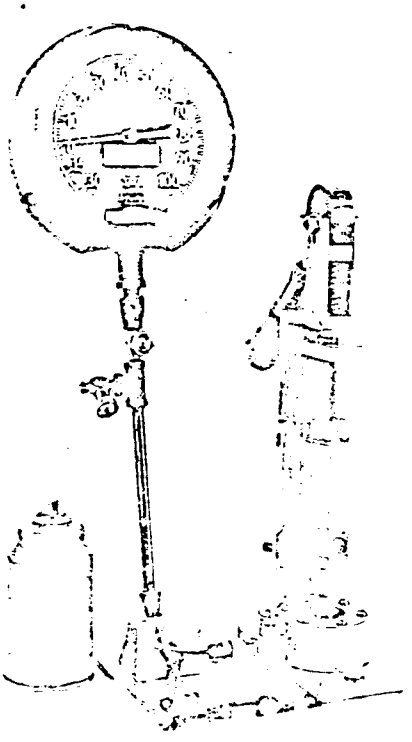

Figure 1

Apparatus

1. A four-inch laboratory test gage, 1-100 psi. range, graduated in one-half pound subdivisions.

2. A jig with valve block for holding the dispenser and which will activate the valve when clamped into position. The valve block has an "O" ring which effects a seal against the outside of the one-inch valve cup of the dispenser and will accommodate most valves. A

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special valve block may be required in certain cases. Exchangeable valve depressing pins of various lengths and shapes complete the apparatus. The hook-up between valve block and gage (see Figure 2) is as follows: ⅛" Hoke valve, ⅛" diameter tubing, gage block, ⅛" pipe of sufficient length to allow the gage to be positioned above the surface of the water bath, ⅛" tee, ⅛" Hoke valve and gage. Reducers and connectors for pipe and tubing are used where necessary. The exhaust end of the tee is controlled by another ⅛" Hoke valve. All plumbing should be brass and copper.

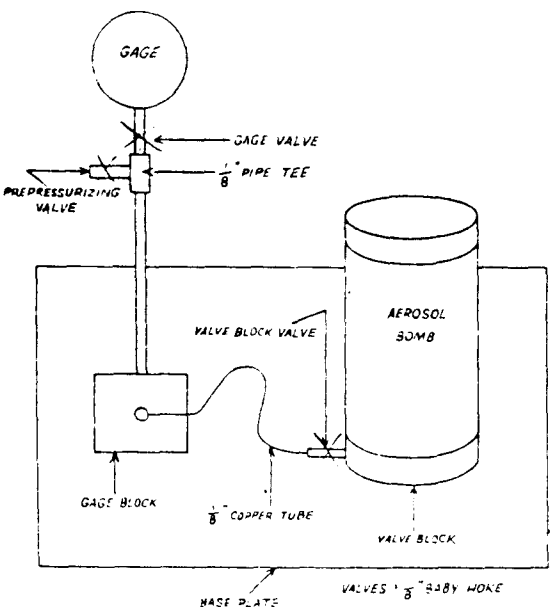

Figure 2
Apparatus for Pressure Method

3. Constant temperature water bath equipped with stirring apparatus and of sufficient size to hold jig and test dispensers. Bath should be deep enough to allow the dispensers to be entirely surrounded with at least one inch of water. This will require a screen or perforated shelf above the floor of the bath. The bath should be 70° + 0.5°F. At 70°F. a 1°F. increase in bath temperature raises the pressure of the Official Test Aerosol (OTA) approximately 0.8 psig.

Procedure

1. Fifteen minutes before testing, place dispensers to be measured in 70°F. water bath along with measuring jig which has been prepressurized to 30 psig. and in which a spare dispenser has been clamped. The valve in the valve block is opened so that pressure in the dispenser is being measured. Use of spare dispenser prevents inaccuracies with the first test can, and keeps water out of the jig. Shake cans occasionally to help bring the contents more quickly to the bath temperature.

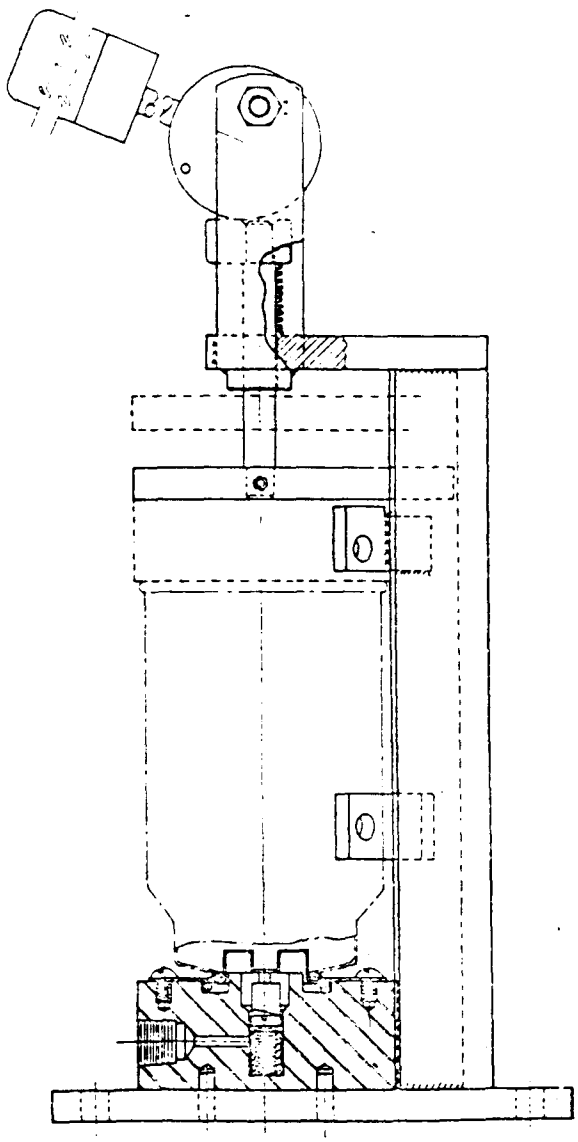

Figure 3
Bomb Holder for Pressure Measurement of Aerosols
Scale: Three Quarter
C.S.M.A. Aerosol Scientific Committee, December, 1955

2. After 15 minutes, remove jig from bath and close valve in the valve block to keep system under pressure. Remove spare container and crack valve block valve just enough to reduce gage reading to the proper starting

pressure (30 psig.). Wipe out any liquid that comes out of the valve block during this operation. Quickly remove first can to be tested from the bath with a pair of tongs and wipe off excess water before placing in jig.

3. After can has been clamped in position, open valve block valve, tap gage lightly, and record gage reading. Close valve block valve and repeat operation for the next can. Replace jig in bath every five minutes.

4. Lines should be blown out periodically. The frequency of cleaning will be governed by the type of formulas encountered, and must be determined by experience. Operation is accomplished by back pressuring through the prepressurizing valve after first isolating the gage by closing the gage valve.

Discussion

It is suggested that a standard consisting of an accurately-filled dispenser having a similar container and valve as the test samples be used to check the proper functioning of the measuring system. The OTA dispenser would serve satisfactorily to check concave style cans. The standard should be used to start off a series of measurements and be used at regular intervals throughout the series.

The process of prepressurizing should be done in such a way that the nitrogen will purge out the air in the system. If the nitrogen cylinder does not have a regulator, great caution must be exercised to prevent damage to the gage. A vacuum pump may also be used to draw air from the system. Here again care must be taken to prevent bending the pointer and throwing the gage out of calibration. The use of a compound gage is the best solution.

Prepressurizing may be accomplished most conveniently and without chance of injuring the gage by using a dispenser filled with the propellent gases alone in the same proportions as the product being tested.

Insecticide Standard Methods Committee
1st Review, March 15, 1955 — 2nd Review, November 10, 1955
Reported November 15, 1955
Adopted by the Scientific Committee, December 5, 1956
Approved by the Administrative Committee, Aerosol Division, May 20, 1957

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METHOD FOR DETERMINING THE VOLATILE–NON-VOLATILE CONTENT OF AEROSOL PRODUCTS
(DENSIMETRIC ANALYSIS)

Introduction

Two methods have been developed for determining the volatile - non-volatile composition of insecticidal and room deodorant formulations. Because of limitations inherent in each, they are regarded as complimentary. The densimetric method is based on the fact that, under isothermal conditions, the density of an aerosol formulation is almost a linear function of the volatile or non-volatile content. The vacuum distillation method utilizes the percentage weight loss of a sample held under moderate vacuum and ambient temperatures as its basis.

Because of the celerity with which densimetric determinations may be run, the method achieves primary significance as a production control operation. The density of test samples must be compared with that of standard samples before the relation of density to volatile or non-volatile content is possible. It is desirable to construct a graph or (preferably) a table of composition vs. density, using several samples of accurately known composition. The test sample densities may then be converted to volatile or non-volatile composition.

Apparatus

1. *Metal or glass cannister:* Diameter 3 to 4 inches; height about 16 inches.
2. *Metal or glass cylinder:* Diameter 1 inch minimum; height about 16 inches. A metal cylinder requires close attention to final filling level but is preferred by experienced operators. A 2½ inch diameter, 3/16" thick copper disc, to which a 16 inch long, 1 inch inside diameter copper pipe has been brazed has been found to be very satisfactory. The weight of the metal cylinder should be such that it will overcome the bouyant effect of the cooling bath in which it will be immersed.
3. *Hydrometer set:* Range to cover anticipated compositions. Length 14½ inches minimum. Scale 0.001 or 0.002 gm./ml. per division.
4. *Thermometer*: Scale—30°F. to 120°F. For three inch immersion. The thermometer should be inserted through a cork large enough to rest upon the rim of the cylinder.
5. *Weighting ring*: (Optional) Bore 1½ inches. Weight about 200 grams. May be slipped over glass cylinders to provide added weight in counteracting bouyancy of cooling bath.

Procedure:

1. Place metal or weighted glass cylinder in canister. Pour dichlorodifluoromethane into canister until liquid level rises to within ½ inch of cylinder top.
2. Chill test sample to approximately −20°F. while still in aerosol container. This is conveniently done by submerging the aerosol in a reserve quantity of dichlorodifluoromethane. Puncture dispenser and pour contents into cylinder, allowing for later introduction of hydrometer in order to prevent overflow.
3. Pre-chill thermometer by momentary immersion in cooling bath. Adjust cork position on stem to give three-inch immersion and then check temperature of aerosol composition. Temperature should read −20°F. before proceeding.
4. Pre-chill hydrometer by momentary immersion in cooling bath. Raise, allow to dry, and immediately dip into test solution. Do not permit hydrometer to bounce. This causes frost to form on the stem. Read and record density.
5. Flush cylinder with propellent and allow to stand inverted in freezer between analyses. Propellent may be maintained in deep freeze for short periods or between samples during continuous operation, although small amounts of additional material will have to be added from time to time. For longer periods it is more convenient to store propellent in pressure containers.

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Calculations

To determine composition from density reference data must be available. To acquire such data a minimum of two known samples; one containing about 1% more volatile material than the required amount; the other containing about 1% less; are tested by the above procedure and densities thus determined. Over a small composition range (up to 5%) density may be said to vary linearly with composition. Accordingly, a graph may be constructed, or the companion equation:

$$K(\text{Density}_{\text{standard}} - \text{Density}_{\text{test sample}}) + \%_{\text{standard}} = \%_{\text{test sample}}$$

derived, where K is the slope. More conveniently a short table can be worked out, giving densities for 0.1% increments on both sides of the optimum composition.

An alternate method is often used, particularly for formulations having high propellent content, such as a room deodorant. The density of a required composition standard sample is determined. Then the density of the pure propellent is either determined or found in the manufacturers literature. These figures are treated as described above.

Discussion

Many minor variations may be applied to this method at the discretion of the individual. A shorter (6¼ inch) hydrometer may be used, but with some loss of accuracy. Propellents other than dichlorodifluoromethane may be used, but generally at greater expense. Because of the time required to reach the temperature of −20°F. (Actual B.P.CCl_2F_2 = −20.4°F. at 1.0 atm.), some operators use −19°F. as the measuring temperature. Normally, a 1°F. temperature error imparts about a 0.1% composition error in the determination. A low temperature toluene or alcohol thermometer may be used if it is considered desirable to read temperatures without lifting the instrument from the cylinder.

Aerosol compositions having pressures of 40 psi gage or less at 70°F. have been found to lose weight at the rate of 0.1% in from 5 to 20 minutes, depending primarily upon the propellent content, when held at −20°F. in a cylinder. The method as described cannot be recommended for compositions boiling at less than 0°F. or having pressures greater than 40 psi-g. at 70°F.

Unusually volatile formulations are determined using coolant liquids boiling at temperatures lower than −20°F. Mixed coolants may be employed but conditions must be identical when determining standard and test samples. Temperatures below about −40°F. should be avoided because of excessive frost formulation and possible separation, gelation or solidification of the aerosol formulation.

For aerosols carrying about 90% propellent the composition changes by about 0.1% for a density change of 0.0011 gm./ml. At 80% propellent the corresponding density change is only 0.00095 gm./ml.

Certain compositions—notably residual insecticide types—are found to deposit toxicants when chilled to −20°F. However, this does not seem to impair the accuracy of analysis; probably because the standard determinations are subject to the same difficulty.

While developed for insecticides and room deodorants, the method is not limited to the analysis of these formulations but has been used with complete success with mothproofers, insect repellents and even aerosol colognes.

The most widespread application of the densimetric determination is in production prechecking of single-staged aerosol batches. Samples are withdrawn from mixing tanks into aerosol containers fitted with screw type shells and attached "Tap-A-Can" valves. These samples must be approved before lots are used.

Reported by the Insecticide Standards Sub-Committee, December 12, 1955
Adopted by the Scientific Committee, May 20, 1956
Adopted by the Administrative Committee, Aerosol Division, May 20, 1956

METHOD FOR DETERMINATION OF VOLATILE – NON-VOLATILE RATIOS OF AEROSOL FORMULATIONS

VACUUM DISTILLATION METHOD

Scope

This method was designed primarily for use with aerosol insecticides and room deodorants in which the formulation does not contain methylene chloride or volatile active ingredients. It is particularly useful for formulations that precipitate at low temperature (−20°F) or contain suspended solids. The Densimetric Method of Analysis is recommended for those formulations not containing solids at low temperature (−20°F) or that do not have volatile active ingredient components which interfere with the vacuum distillation method.

Equipment Required

1. Vacuum source which will produce about a 28" vacuum.
2. Thief equipped with needle valve.
3. Balance accurate to 0.1 gram.
4. Three 125cc vacuum Erlenmeyer type flasks.
5. Capillary tubing (may be made from discarded thermometer).
6. Hot water bath (120°F).
7. Stoppers, vacuum tubing, etc.

Procedure

Three samples should be analysed simultaneously from the unknown dispenser. If possible, a sample should be prepared containing identical ingredients as compared to the unknown being analysed, having a known non-volatile content to check the general procedure.

1. Clean the exterior of the sample(s) to be analysed.
2. Attach thief to the dispenser at a point where the maximum amount of liquid may be withdrawn from the dispenser piercing in the liquid phase. Withdraw a small amount of sample through the needle valve, allow to drain and weigh.
3. Clean and weigh vacuum flasks with stoppers and capillaries in place (record as flask tare). A single hole stopper should be placed in the vacuum flask with the capillary extending all the way to the bottom corner of the flask. Thus, the flask can be tilted so that air being introduced through the capillary can bubble through a maximum amount of liquid.
4. Withdraw sample through thief into vacuum flask placing the end or outlet as far into the flask as possible. Withdraw approximately 50 grams, close needle valve, allow to drain and reweigh. Repeat this sampling procedure into other two previously weighed flasks.
5. Place vacuum flasks in a 120°F. water bath and allow propellant to boil off through side arm. When boiling has subsided, *gently* apply vacuum to flask to avoid excessive bubbling and resultant loss of liquid. Apply continuous vacuum at 120°F for approximately thirty minutes. Release vacuum and swirl flask gently. Reapply vacuum for about thirty seconds. Repeat three or four times and weigh. At this point, the known sample should be subjected to several brief vacuumings with weighings after each one. The point at which the correct percentage of non-volatile is reproduced (end point) should be used to determine the percentage of non-volatile in the unknown samples.

   In the event that a known cannot be prepared, a series of vacuumings and weighings should be carried out on the unknown

until weight loss appears slight and reasonably constant. Once the procedure is established, weighings may be eliminated until the final end point is reached. Record final weight as flask plus non-volatile weight.

Calculations

Total sample weight equals total weight minus can tare (4).

Non-volatile weight equals flask plus non-volatile minus flask tare.

Percent non-volatile equals

$$\frac{\text{non-volatile weight}}{\text{total sample weight}} \times 100.$$

Discussion

The above procedure generally appears to be satisfactory for most products within the scope of the test. Modifications may be made to make it more suitable to fit a specific purpose. For example, it may be undesirable to attach a thief to a dispenser because of destruction of the container making it necessary to sample the container through the aerosol valve. This may be accomplished by weighing the sample and introducing it into the vacuum flasks by spraying into a short length of plastic tubing which can be fitted rather closely over the nozzle. The tubing is projected as far into the flask as possible through a cotton plug, the sample is taken and the tubing is simply dropped into the flask having been included in the tare weight of the flask previously. A small bit of cotton, or some other suitable absorbing material, should be included to wipe off any excess liquid from around the base of the valve which does not go into the tube. This also must be included when taring the flask and should be dropped into the inside of the flask when used.

If at all possible, a vacuum line should be equipped with a gauge to indicate the vacuum being produced. This is particularly necessary since the propellant being pulled off through the vacuum line is quite soluble in the oil and could effectively dilute or thin the oil in the vacuum pump, resulting in a loss of vacuum produced.

The 120°F. temperature to which the samples are subjected was selected to avoid decomposition of insecticidal ingredients so that the non-volatile portion could subsequently be used for entomological tests. If this is unnecessary, higher temperatures may be used but with a shorter vacuum time as determined by trial and error. Care should be taken to avoid excessive distillation as it is possible to distill some relatively high boiling materials and thus come up with a low answer. Also, a low answer will be produced if any liquid material is spilled or lost during the course of the analysis. High results are caused by incomplete distillation generally indicated by significant weight losses when vacuum is applied.

Approved by the Insecticide Standards Sub-Committee, May 8, 1956
Approved by the Scientific Committee, December 3, 1956
Adopted by the Administrative Committee, Aerosol Division, December 4, 1956

METHOD FOR DETERMINATION OF SOLIDS CONTENT OF AEROSOL COATINGS

Summary

Knowledge of the solids content of an aerosol coating can be useful information for predicting the performance of the product. In general, coatings of high solids content are to be preferred because they give greater coverage and protection to the substrate being coated. Various techniques are available for determining the solids content of conventional coatings, but a suitable method for aerosol coatings was not available heretofore. It was desirable, therefore, to develop a simple method for aerosol coatings. A limited amount of experimental work indicates that good results can be obtained with a minimum of technique and equipment. The method is described below.

Apparatus

1. Flexible aluminum foil flat-bottomed dish, 5-in. x 4-in. x 1¾ in. (depth), (can be obtained from Quaker City Products Co., Philadelphia, Pennsylvania).
2. Oven maintained at 120°C.
3. Torsion balance with a sensitivity of 2 mg. and a scale graduated in divisions of 10 mg.
4. Facilities for obtaining a temperature of 0°C. for at least 2 hours.

Procedure

Tumble or agitate dispensers of the aerosol coating product for at least 24 hours prior to testing. Weigh an aluminum dish with the cover and a dispenser of the aerosol coating product under test (at room temperature). Store both the dish and the dispenser at 0°C. for 2 hours then shake the dispenser for 5 minutes by hand. After shaking, spray approximately a 10-gram sample of the aerosol coating product into the aluminum dish from a distance of approximately 6 to 8 inches. Allow the dispenser to rise to room temperature (in 30 minutes) then reweigh. Place the cover of the aluminum dish on loosely, allow the dish to come to room temperature in about 30 minutes (this will allow the retained propellant to evaporate) then place the dish (uncovered) in an oven at 120°C. for 3 hours. Weigh the dish after it reach room temperature (in 30 minutes).

Calculation of Solids Content

The solids content of product under test is then calculated as shown below.

$$\frac{\text{Weight of residue in aluminum dish}}{\text{Loss in weight of aerosol dispenser}} \times 100 = \%\ \text{Total Solids (Solution basis)}$$

Duplicate determinations should agree within 0.2%.

Standard Lacquer

The nitrocellulose metal lacquer shown below (formulated for aerosol application) was used in the development of the above procedure. Good agreement was obtained among several determinations on the same dispenser. The lacquer should be used as control.

Nitrocellulose Metal Lacquer

RS Nitrocellulose, ½ sec.	5
Cellolyn 502 (60% in Xylene)	9
Dibutyl Phthalate	2
RBH Dispersion No. 1(2)	24
Methyl Isobutyl Ketone	49
Butyl Cellosolve	5
Isopropanol	3
Alcohol, 2B	3
	100
Calculated Total Solids, % by Weight	21.0

Aerosol Application of Above Lacquer

(Package in a 12-oz. dispenser with a typical aerosol valve)(1)

Above Lacquer	170 grams
Freon 12	170 grams
Calculated Total Solids, % by Weight	10.5

(1) The Precision valve, Model I (Precision Valve Company) and the Danvern aerosol valve have given good results.

(2) Can be obtained from RBH Dispersions Division of Interchemical Corporation.

Approved by the Protective Coatings Standard Methods Sub Committee, April 26, 1956
Adopted by the Scientific Committee, May 20, 1956
Adopted by the Administrative Committee, Aerosol Division, May 29, 1956

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METHOD OF DETERMINATION OF THE PARTICLE-SIZE DISTRIBUTION OF SPACE INSECTICIDE AEROSOL

Principle

The aerosol is drawn into a wind tunnel so that the individual particles deposit on a rotating microscope slide. The particles on the slide are counted and classified by size. A suitable correction is applied in order to calculate the particle sizes in the original spray. This is an adaptation of methods previously described by Yeomans et al. (Ref. 1) and Bower. (Ref. 2).

Apparatus

Wind tunnel: a tube about 18" in diameter and 4' long. This tube may be attached with a hinged joint to a compartment which contains the exhaust fan and slide rotator. The fan (nominal capacity of about 2400 cubic feet per minute) is operated at reduced voltage and speed so as to give a wind velocity in the tunnel of 500 ± 100 feet per minute. (Note 1).

Slide rotator: a small motor-driven arm fitted with a microscope-slide holder and a counterbalance. The radius of the center of the slide is 4". The slide is held with its 3" side parallel to the axis of the tunnel. The 1" end of the slide is tilted 40° forward in the direction of rotation. The slide rotator is operated at 450± 50 rpm. (slide velocity 10.7 ± 1.2 mph.) (Note 2).

Microscope and accessories: a standard microscope fitted with a 10x ocular lens containing a 50-division micrometer disc, a 10x objective lens and a mechanical stage. A filar micrometer may be used for greater accuracy. The micrometers and the fine focus adjustment must be calibrated in microns per unit, using a stage micrometer slide as standard. Ordinary 1" x 3" microscope slides are used for collecting the droplets and for covering them to prevent evaporation before they are counted. The cover slides are supported by paper shims. These are 1" x 3" pieces cut from heavy paper or light card stock. Three holes 5/8" in diameter are cut in them and one side is marked "UP". (Note 3).

Preparation of Slides

The slides must be treated so that the oily droplets will not spread but will remain separate as small convex lenses on the surface. The slides are thoroughly cleaned, rinsed and dried. They are then dipped in a 10% solution of "Dri-Film" SC-87 (General Electric Company) in toluene, drained carefully and dried at 180-220°F. for 30 minutes. They should be exposed to moist air or rinsed in acetone before use. They may be repeatedly rinsed with acetone and reused. When the droplets no longer form distinct lenses, the slides should be recleaned and recoated. The slides should be stored in a dust-tight box and their surfaces should not be touched before the spray sample is collected.

Operation

The aerosol units to be tested are placed in a water bath at 80 ± 2°F. The ventilating fan is started and its speed is regulated to provide the standard wind velocity of 500 ± 100 ft. per minute. A treated slide is placed in the slide rotator, which is started and regulated to 450 ± 50 rpm.

The aerosol unit is held with the valve at the axis of the wind tunnel, about 6" in from the open end. A piece of cheesecloth is placed over the valve of the aerosol unit, and the valve is opened. After about 1 second's spray (to bring the valve to a steady state and give a typical spray), the cloth is removed from the valve for about one-half second, and the spray is allowed to travel down the tunnel. The valve is then closed and the motors are stopped. The slide is removed from the holder and promptly covered. (Notes 4, 5).

Determination of Particle-Size Distribution

The diameter of a droplet as measured on the slide must be corrected for the spread that has taken place, so that the diameter of the original sphere can be determined. As the spherica

droplet impinges on the slide it becomes a convex lens. The spread correction factor can be found by measuring the diameter and the focal length of this lens by the following method as described by May. (Ref. 3). The filar micrometer is fitted to the microscope, the substage condenser is removed and the flat side of the substage mirror is used. Using the mechanical stage, the field within one of the holes of the cover support is scanned. Every fifteenth droplet is measured for a total of 13 droplets so that about 200 droplets have been scanned. The diameter (2A) of each selected droplet is measured accurately with the filar micrometer.

The focal length (f') of each selected droplet is measured by the following method: The calibrated fine focus adjustment is set on zero, and the plane of the slide is brought into sharp focus by using the coarse adjustment. At the proper setting, the boundary of the droplet appears as a ring which is alternately light or dark as small changes are made in the focus. A window or other wide light source more than two feet away is used. The microscope tube is now raised by using the fine focus adjustment until the light source or the bars of the window are brought into sharp focus as seen through the droplet. The focal lengths so measured are recorded along with the corresponding diameters on the report sheet. The ratio of the focal length to the diameter (f' 2A) is calculated and listed for each droplet. The average value of this ratio is determined and is corrected by multiplying by (V/F), the ratio of the scale factors which corrects the filar micrometer (F) and the fine focus adjustment (V) settings to actual micron values.

The spread correction factor (C) is shown in Figure 1, which has been constructed from

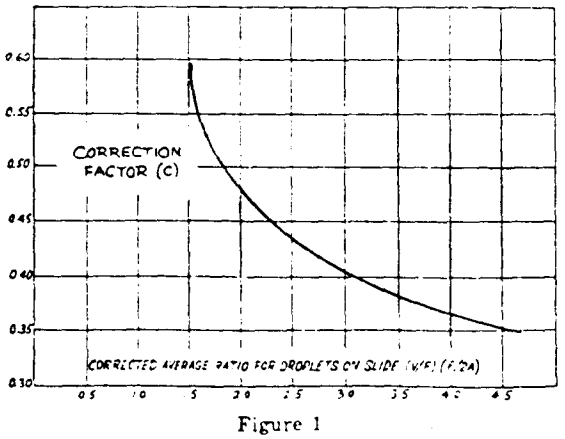

Figure 1

May's data. (Ref. 3). Using the average value of the ratio of the focal length to the diameter of the individual droplets, (V/F) (f'/2A), record the spread correction factor (C) from Figure 1. (Note 6).

The droplets on the slide are now classified by size. The filar micrometer is removed and the 10x ocular lens containing the calibrated ocular micrometer (M microns per unit) is inserted into the microscope tube. The diameters of 200 consecutive droplets are measured by means of the ocular micrometer. Each particle is measured to the nearest half unit on the micrometer scale and classified accordingly. A tally sheet is prepared (on the back of the report sheet) showing the numbers of particles (f) in each of the different size classes. For example, a particle estimated to cover 2.7 micrometer divisions is counted as being in the 2.5 class; a particle estimated as covering 2.8 divisions is classified in the 3.0 class. All the particles counted as being in a certain class are assumed to have the standard diameter of that class (such as 2.5, 3.0, etc.). These numbers are called scale class marks. Each scale class mark is then multipled by the product (CM) of the spread correction factor and the scale correction factor for the ocular micrometer to give the class mark (d), the diameter in microns of the original spherical droplets.

Report

The data should be reported on the basis of the cumulative weight percent of the spray which has a particle-size less than or equal to 5, 10, 15, etc., microns. The mass median diameter, that is, the diameter at 50 cumulative weight percent, should also be reported.

Multiply the number of particles (f) in each size class by the class mark (d). This product (df) is proportional to the weight of all the particles in this size class in the original spray. (Note 7). Add all these products and list the subtotals ($\Sigma\, d_i f_i$) opposite each class mark. opposite each class mark. Divide each subtotal by the total ($\Sigma\, d_i f_i$) to get the cumulative weight percent at each class mark.

Particle-size distributions often follow a logarithmic normal distribution curve. This can be plotted as a straight line by using logarithmic probability graph paper (Codex Book Company, Inc., Norwood, Mass., #3128).

Plot the values for the cumulative weight percent at the class marks (particle diameters), and draw the best smooth curve through these

Figure 2

Aerosol Particle-Size Distribution

SPREAD CORRECTION

Drop No.	Filar Micr. Left	Filar Micr. Right	Diam. 2A	Focus f'	Ratio f'/2A
1	178	211	33	62	1.88
2	25	51	26	48	1.85
3	510	550	40	77	1.93
4	655	683	28	58	2.07
5	600	646	46	97	2.11
6	920	1000	80	192	2.40
7	538	602	64	205	3.20
8	246	328	82	200	2.44
9	91	188	97	254	2.62
10	473	504	31	99	3.19
11	600	683	83	192	2.31
12	470	552	82	178	2.17
13	75	152	77	164	2.12

Average f'/2A 2.33 Corr. Av. (V/F) (f'/2A) 2.41

Date 4/25/56

Sample OTA No. 1

Notebook 37-136

Rotor Speed 400 RPM

Fan Speed 825 RPM

Factors (microns per unit)

Filar (F) 0.967

Vertical (V) 1.00

Ratio (V/F) 1.034

Ocular (M) 15.1

Spread Corr. Factor (C) 0.438

Class Mark Factor (CM) 6.61

DISTRIBUTION

Scale Class Mark	Class Mark d	Freq. f	Weight df	Cum. Wt. Σdf	Cum. Wt. %
1	6.6	50	330	330	6.8
1.5	9.9	32	317	647	13.3
2	13.2	51	673	1320	27.2
2.5	16.5	31	512	1832	37.8
3	19.8	34	673	2505	51.7
3.5	23.1	16	370	2875	59.3
4	26.4	40	1056	3931	81.1
4.5	29.7	8	238	4169	86.0
5	33.0	8	264	4433	91.5
5.5	36.4	2	73	4506	93.0
6	39.7	3	119	4625	95.4
6.5	43.0	3	129	4754	98.1
7	46.3	2	93	4847	100

REPORT

Diam., microns	Cum. wt. %
5	4
10	14
15	30
20	50
25	70
30	83
35	92
40	96
45	98.4
50	99.4
20	50

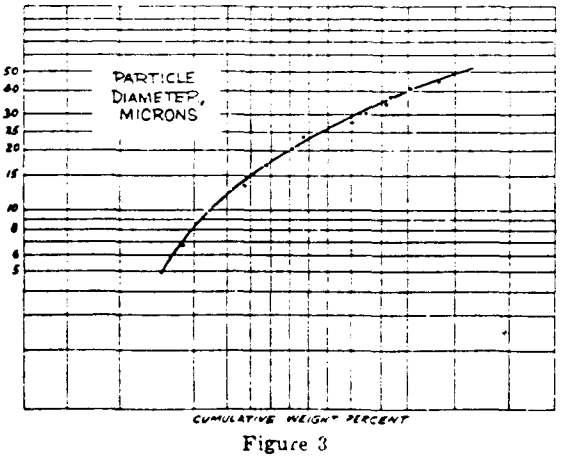

Figure 3

points as shown in Figure 3. The curve thus obtained represents the total particle-size distribution produced by the aerosol unit at 80°F. For the report, record the cumulative weight percent at each diameter 5, 10, 15 . . . 50 microns from the curve. Also list the diameter at 50 cumulative weight percent as the mass median particle-size.

A particle-size analysis of the CSMA official test aerosol insecticide 1955-1960 on the proper type of report form and graph paper is shown as Figures 2 and 3.

Estimation of Error

Since only 200 particles are counted, some size classes contain very few particles. The cu-

mulative weight percent calculated for such a size class may be somewhat inaccurate. An estimate of error at each class mark is given by the equation

$$\text{Error } (\%) = \pm \frac{100 \sqrt{\Sigma^n d_i^2 f_i}}{\Sigma^n d_i f_i}$$

where $\Sigma^n d_i^2 f_i$ is the cumulative sum of the products (d^2f) of the number of particles (f) in each size class, up to and including the one for which the error is being calculated, times the square of the corresponding class mark (d^2); and $\Sigma^n d_i f_i$ is the sum of all the products (df) of the number of particles (f) in a size class times the corresponding class mark (d). This sum will already have been computed in calculating the cumulative weight percent at each true class mark.

Two out of three separate determinations will fall within the range defined by the above function. If greater accuracy is required, more particles must be counted.

Notes

1. The wind velocity should be measured at the axis of the tunnel. The velocity may be correlated with the voltage on the fan motor, or the rate of rotation of the fan, so that the wind velocity need not be determined before each run.

2. The "Visutac", a small hand-held stroboscope, is recommended for standardizing the speed of the ventilating fan and of the slide rotator. This instrument is made by the Boulin Instrument Corporation, 65 Madison Avenue, New York, New York.

3. The slides must be covered to prevent evaporation of the droplets. Microculture slides having a spherical concavity may be used, but this introduces some distortion and requires constant refocusing of the microscope as the droplets are counted. Any cover used must leave 200-300 microns air space above the collecting slide.

4. One of the paper cover supports is placed on a paper towel and several drops of insecticide concentrate are distributed around the border by means of a dropper. This paper is laid carefully over the exposed slide and a cover slide is placed over the paper. This "sandwich" may be held together with scotch tape at the ends. The droplets must be on the upper surface of the lower slide. Additional drops of concentrate may be added to the paper between the slides, if necessary, to keep the paper moist. Insecticide concentrate is easily collected by spraying the aerosol unit directly into the large end of an empty drying tube. The concentrate, containing little or no condensed moisture, is collected in a vial at the small end of the tube.

5. The proper time of exposure is important. One-half second is usually about right for space insecticides. Very fine sprays may require a longer time. Coarser sprays will require a much shorter time. The clear area within one of the holes should contain 600 to 1000 droplets. The proper coverage can be determined by a quick visual inspection.

6. The refractive index of the concentrate is assumed to be 1.5. Kerosene has a value of 1.44. Aromatic or chlorinated solvents or insecticides raise the value towards 1.5. The OTA concentrate obtained as in Note 4 had a value of 1.492.

7. The rotating slide in the wind tunnel does not collect particles of different sizes with equal efficiency. A higher proportion of larger particles is collected than of the smaller particles in the same aerosol spray. The number of particles counted in each size class must be divided by the relative collection efficiency for that size class in order to correct for the sampling bias of the apparatus used.

The weight of each particle is proportional to the cube of its diameter. Therefore, the relative weight of the particles collected in each size class is determined by multiplying the number of particles found in that size class by the cube of the class mark, and dividing by the efficiency of collection on the microscope slide.

For particles having diameters up to about 50 microns, the efficiency of collection in this apparatus, operated as defined, increases directly with the square of their diameters. Therefore, the relative weight of particles in each size class in the original spray can be determined by multiplying the number of particles counted in that size class by the class mark; that is, by the diameter cubed divided by the diameter squared.

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(3) May, K. R.: "The Cascade Impactor: An Instrument for Sampling Coarse Aerosols," J. Sci. Instruments, 22, 187-195 (1945).

Approved Particle Size Sub-Committee, May 8, 1956
Adopted by the Scientific Committee, December 3, 1956
Adopted by the Administrative Committee, Aerosol Division, December 4, 1956

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METHOD FOR DELIVERY RATE OF AEROSOL INSECTICIDES AND ROOM DEODORANTS

Introduction

The delivery rate of an insecticidal aerosol dispenser is determined by measuring the quantity of material atomized by the valve in ten seconds. The duration of discharge and the temperature of the container and contents must be carefully controlled to obtain results that will correlate with subsequent test results.

Delivery rate tests assist in evaluating one aspect of valve performance and are considered a prerequisite to both biological and storage tests. Biological tests are frequently made with one or two dispensers selected from a group similar in all respects except delivery rates. When it is desirable to select dispensers with equal delivery rates, two or three tests should be performed on each dispenser. In the case of storage tests, a single delivery rate test is performed at each examination period to conserve the contents and extend the life of the dispenser.

Equipment

Water bath, maintained at 80± 0.5°F. with a screen or perforated metal shelf 1-inch above the bottom of the bath.

Stirrer, air-driven.

Balance, one-tenth gram scale.

Stop watch or electric timer, (an efficient timer can be assembled by mounting a cardboard disc three or four inches in diameter on the shaft of a 30 or 60 RPM motor. A single line from the center to a point on the circumference of the disc assists in counting the revolutions.)

Procedure

Remove the protective cover and other detachable materials from the dispenser. Activate the valve for 3 to 5 seconds and weigh the dispenser to the nearest 0.1 gram. Place the dispenser on the shelf in the water bath which is at the test temperature of 80± 0.5°F., for 10 minutes. Keep the dispensers in an upright position, spaced 1-inch apart and covered with 1 inch of water while in the bath. Circulate the water in the bath with an air-driven stirrer.

At the end of the 10-minute period remove the dispenser with beaker tongs and place it in an exhaust hood. Immediately open the valve completely for a period of 10 seconds. Dry the dispenser with a cloth or towel and use a blast of compressed air to remove moisture from the valve mounting cup and container seams. Reweigh the dispenser and record the difference in weight.

The results of each test are reported as delivery rate in grams per second and calculated as follows:

$$\text{Delivery rate, gm./sec.} = \frac{A}{10 \text{ sec.}}$$

A = loss in weight in grams.

Repeat the above procedure if duplicate tests are made on dispensers.

This method was experimentally evaluated using the 1949-1950 official test aerosol formulation. It is felt that the test method will apply to all products of a like nature.

Approved Insecticide Standard Methods Sub-Committee
Adopted by the Scientific Committee
Adopted by the Administrative Committee, Aerosol Division

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INSPECTION PROCEDURES FOR GLASS AEROSOLS

In view of the hazard involved in the use of glass containers for aerosol products, the C.S.M.A. Glass Bottle Advisory Committee has directed that standard pretesting procedures be established to recommend optimum filling methods, which will insure marketable pressurized products presenting a minimum of danger to the consumer.

A summary of pretesting procedures is presented herewith to serve as a guide for present and potential packagers of glass aerosol products. These methods have provided excellent results in the field, and are listed in descending order of occurrence after receipt of containers from the glass manufacturer as follows:

1. Specifications

a) *Bottle Capacity*

The internal volumes of randomly selected bottles, with valves inserted, are determined to insure that the headspace will be adequate to prevent a liquid-fill condition from developing under any anticipated combination of filling volume and temperature.

b) *Other Bottle Dimensions*

Overall height, weight, plastic coating thickness and neck diameter specifications are sampled to determine acceptance of bottles on the filling equipment, with particular emphasis on attainment of satisfactory valve sealing. Specifications are to be supplied in blueprint form by the container manufacturer.

2. Visual Inspection

Units are sampled for visual observation of possible defects in the glass or external plastic coating, which may have been imparted during the manufacturing process, or in transit to the filler.

3. Cleaning

Bottles are submitted to a filtered, dry air blast prior to loading, in order to remove dirt or lint particles, which might otherwise present an unsightly appearance in formulations packaged in transparent units, or cause valve clogging upon spraying.

4. Pressure Test

All aerosol bottles must be able to withstand a minimum air pressure of 125 psig without bursting. This test serves as a positive check on glass distribution in the bottle, and eliminates those units exhibiting flaws obtained in handling prior to aerosol packaging. A suitable shield must be provided to protect plant personnel from possible injury occasioned by glass fragments during this test.

5. Evacuation of Entrapped Air

Adequate precautions must be observed to remove air from the bottles during the filling process, in order to maintain product vapor pressures in accordance with theory, and within the maximum limitation of 25 psig at 70°F. Various methods are utilized for this purpose as follows:

a) *Refrigeration Filling*

Product filling temperatures are controlled in order to provide the volatilization of sufficient propellent to displace air prior to valve closure.

b) *Pressure Filling*

Following concentrate loading, a vacuum is drawn on the container headspace to remove air prior to valve closure. An alternative method involving introduction of a small amount of Propellent 12, which flashes off to displace the air, may be utilized in lieu of the vacuum method.

6. Vapor Pressure Determination

The internal pressure of glass aerosol products may be determined in accordance with the standard method proposed by the C.S.M.A. Glass Aerosol Sub-committee.

7. Water Bath Test

Filled units may be subjected to immersion in a water bath generally maintained at a temperature below the deteriorating point of the formulation perfume component. Present practice usually involves a temperature of 100-110°F with an adequate immersion time commensurate with proper attainment of temperature equilibrium conditions. This test provides a check on improper valve closures and possible over-filling on the line.

8. Final Inspection

The units receive a final visual inspection for filling height, and for possible dirt or scratches on the exterior surfaces of the bottles or coatings to insure proper esthetic appearance. The filling height of opaque coated bottles may be checked in many cases by passing the bottles in front of a strong light source. Valves are briefly actuated to check performance, and the units are boxed for shipment.

Approved by the Project Committee on Pretesting Glass Aerosols, May 20, 1957
Adopted by the Scientific Committee, May 20, 1957
Approved by the Glass Policy Advisory Committee, May 20, 1957
Adopted by the Administrative Committee, Aerosol Division, March 21, 1957

METHOD FOR STORAGE TESTS OF AEROSOL INSECTICIDES

Introduction

Aerosol insecticides are subjected to storage tests in order to ascertain the shelf-life of the complete package and to evaluate the degree of suitability of the valve and container components for their intended uses. It is impractical to promulgate a set procedure for conducting storage tests since variations will be necessitated by differences in the ultimate objective. For example, the primary interest of one investigation may be in valve evaluation, while another may be principally concerned with container suitability or the shelf-life of a new product in an existing package. It follows that storage test methods must be flexible enough to accommodate the small procedural changes thus required. An attempt will be made only to outline the principles to be observed in establishing a definite procedure in order to allow the individual investigator the prerogative of adapting these to satisfy his particular requirements.

There are three major points that should be borne in mind when a storage test of aerosol containers is to be made. First, sufficient samples should be available to replace any containers that fail during the course of the test and to make it possible to later extend the storage period, if desired. There is nothing more discouraging than reaching the end of a long storage test with a quantity of samples which, due to unusual conditions arising, are insufficient in number to provide a sound basis for drawing valid conclusions. Second, it is extremely important that the examinations of the test pack be conducted according to standardized procedures and at regularly scheduled intervals. Only if this rule is followed can there be any assurance that important developments will not be missed and that the results will correlate with those of other storage tests. Third, the examinations should be made by personnel familiar with the problem being investigated and well qualified to evaluate the condition of the containers, valve components and product. It is highly desirable to have the same individual conduct all the examinations of a given test pack since most of the data is not obtained by direct measurement and is therefore not entirely objective in nature. This and a standardized examination procedure will do much to minimize the effect of the human element.

Before any samples are committed to storage, certain information should be made available. So that the test pack can be intelligently set up, all pertinent background information concerning the problem should be assembled. Certain tests should be conducted to eliminate, insofar as possible, defective containers or valves from the tests, although the frequency of such defects should certainly be recorded. To make this segregation possible, pressure determinations and hot bath, vial leakage and spray tests should be made on each filled container. Conditions of filling and handling should approximate as closely as possible those that would be encountered commercially.

There are two types of storage tests that may be performed with aerosol insecticides. The first is the so-called "live" storage test, in which the valves are actuated and the determinations made at relatively frequent intervals. The purpose is, of course, to simulate conditions encountered during use of aerosol dispensers. The second type of test, often referred to as "dead" storage, simulates conditions found in warehouse storage and is performed when shelf-life information is sought.

Live Storage Test

Containers for "live" storage tests are generally stored at room temperature. In addition, a higher temperature storage, e.g., 98°F., is frequently employed. Use of the latter storage temperature is particularly desirable when a new valve or product is being evaluated. The use of storage temperatures below 32°F. or the alternate exposure of test containers to subfreezing and elevated temperatures is said to have considerable merit in the screening of new valves or new valve materials.

If the purpose of the investigation is to evaluate a valve, half the samples at each stor-

age temperature should be kept in an inverted position. If the product or any constituent thereof exerts a detrimental effect on the sealing material of the valve often the conditions may be observed more readily in the case of the inverted cans. Six cans inverted and six upright for each temperature is the minimum sample of each variable that should be considered. If the test is to involve only one storage condition, ten to twelve cans per variable, upright and inverted, is a more desirable sample size:

Examinations of "live" storage cans are usually made weekly and possibly oftener if completion of the test in less total elapsed time is necessary. The tests are usually considered completed when 10 grams or less of product remains in the containers. Extension of the test beyond this point may cause erratic and unreliable results. At each examination weight loss and discharge rate (10 seconds) are measured. The determination of internal pressure at each examination is usually not necessary, but it is recommended that pressures be taken initially and two or three other times equi-spaced during the test. Particle size may also be determined, if desired. When any valve becomes inoperative or fails to operate properly, the container and valve should be torn down immediately to ascertain the cause of failure. Each container and valve should be critically examined as soon as possible after the final valve actuation of the test.

Dead Storage Test

A wider range of storage conditions are employed in "dead" storage tests than is the case with "live" storage, 95-100°F., room temperature, 130°F., and below freezing (0-32°F.) being used. Standard procedure usually calls for the use of 98°F. and room temperature storage, while the other temperatures are employed in special cases. Temperatures of from 95° to 100°F., often referred to as incubation temperature, may accelerate container corrosion and leakage if the containers are so predisposed. However, incubated storage should always be used in conjunction with room temperature since it is often difficult, if not impossible, to predict normal shelf-life on the basis of 98°F. tests alone. Storage below freezing is valuable for evaluating the sealing efficiency and suitability of the gasket materials in insecticide valves. Storage at 130°F. is employed when the resistance of the container to structural fatigue is to be determined. Containers at each storage temperature should be held both upright and inverted.

Examinations of containers in dead storage are usually made following 1-, 3-, and 6-months storage and at 6-month intervals thereafter until the test is completed. Most investigations are concluded after 24-months storage, but they may be extended for a much longer period, if the previous results and the objective so require.

Samples should be provided for the "dead" storage test so that a minimum of two dispensers of each variable from each storage temperature can be evaluated and torn down at each scheduled examination. The other samples remain untouched, except for weighing, until they are needed at a subsequent examination. A minimum of twelve extra containers per variable should be stored at each temperature to allow for extension of the test, if such later becomes necessary, and to allow a larger number of samples to be inspected at the final examination. Thus, the minimum suggested number of cans per product, container or valve variable becomes:

$$4np\ (4 + y)$$

where: y designates the duration of the test in years,

n the number of storage temperatures, and

p the number of storage positions to be employed.

Examination

The examination of the pack may be divided into performance determinations, container and valve inspection, and product evaluation. The performance of the complete aerosol insecticide package is ascertained by making weight loss, discharge rate, pressure and possibly particle size determinations. If entomological data is required, such may be obtained by standard procedures now extant. As a check on filling, the volatile/non-volatile ratio may be determined following these tests. After expulsion of the propellant the product should be transferred, and the container and valves carefully torn down and examined. The metal valve parts should be inspected carefully for evidences of corrosion, and the rubber or plastic components for swelling, softening or disintegration. Conditions in the container interiors should then be noted with special emphasis on any staining, detinning, rusting, pitting or other indications of corrosion that may be present. Microscopic examination of valve and container components is recommended for without this assistance im-

portant and indicative developments may be overlooked. The product from the test containers is usually examined for color change, precipitate or sludge formation. If corrosion is found or suspected, it is suggested that the product be analyzed for the moisture, iron and tin content. If any abnormal or undesirable conditions are found in performance of valve or product, sufficient additional samples of the same lot should be examined to confirm the findings.

Safety Precautions

Aerosol storage tests involve a container, valve or product of unknown compatibility and performance. For this reason, and remembering the violence that may accompany the bursting of aerosol containers, it behooves the investigator to observe safety precautions. The necessity of using gloves, safety shield and glasses, and equipment with proper controls does not need further amplification. If, in the course of a test, container perforations or signs of advanced corrosion are found, or if the product, dispensers and/or valves otherwise become unmerchantable, the entire lot of samples should be destroyed. Besides wasting time and space, to continue such dispensers under test is to run the risk of serious accident.

Typical Pack

The above can best be illustrated by presenting in table form a typical aerosol insecticide storage test made to compare in a given container the shelf-lives of a new formulation and a standard formulation. It is assumed that the pretest information has been accumulated, and containers or valves with obvious defects have been eliminated.

	"Live" Storage	"Dead" Storage
Product, container; valve variables	2 (product)	2 (product)
Storage temperatures	R. T., 98° F.	R. T., 98° F., 30° F.
Storage positions	Upright & Inverted	Upright & Inverted
No. of filled cans per formula	24 (half inverted at each temperature)	144 (half inverted at each temperature)
Total No. of filled cans	48 (half inverted)	288
Duration of test	Until completed	2 years
Examination schedule	Weekly	1, 3, 6. 12, 18, 24 months
No. of containers examined each examination	48 cans	24 cans (2 per product per temperature per position), all remaining dispensers examined after 24 months shortage. All dispensers weighed.
Examination procedure	1. Wt. loss 2. Pressure (see above) 3. Discharge rate (10 sec.) 4. (Particle size) 5. Valve and container inspection at final examination or when failure occurs.	1. Wt. loss 2. Pressure 3. Discharge rate (10 sec.) 4. Valve examination 5. Container examination 6. Product examination

Other Test Procedures

There are several other procedures that should be considered in conjunction with an aerosol testing program.

In evaluating a valve for a given formula it may be desirable to subject the containers to a continuous discharge test whereby the containers are emptied in a single burst or a series of long bursts. Such a procedure is very rapid and may give valuable clues on the suitability of the valve gasket material for the product. If indications of weakening, disintegration or undue swelling of the valve gaskets are found by means of this test, particular attention should be directed to the results of the live storage test.

Before commercial packaging of a new product or the use of a new container or valve

is approved, certain information outside the scope of the laboratory test should be obtained. Filled containers should be subjected to normal handling, cartoning and shipping operations to determine the suitability of protective devices and the resistance of containers and valves to shock. Dispensers should be shipped to and stored in warehouses having the extremes in temperatures that could be encountered in distribution and marketing the product. After the predetermined storage period in the various locations, the containers should be returned to the laboratory for complete examination.

Once commercial packaging of a product is initiated, a program may be inaugurated whereby defective or complaint dispensers encountered in the field are returned to the laboratory. By this means a continuous check on quality is maintained and it may enable the packer to be forwarned of difficulty before it becomes serious so that corrective measures may be taken.

Summary

To summarize, the three major prerequisites for an adequate storage test are: (1) the providing of an adequate number of samples, (2) a well planned examination schedule and procedure, and (3) competent personnel to perform the tests. If the basic rules outlined above are adhered to, much valuable information, not obtainable by other means, will result, thus rendering the storage test an indispensible adjunct to the aerosol insecticide testing program.

Approved by the Insecticide Standard Methods Sub-Committee, May 17, 1953
Adopted by the Scientific Committee, May 17, 1953
Adopted by the Administrative Committee, Aerosol Division, October 11, 1953

METHOD FOR DETERMINING THE SEEPAGE RATE OF AEROSOL INSECTICIDES AND ROOM DEODORANTS

Introduction.

This method has been developed to afford a more rapid answer to the ever present problem of weight loss during storage. It is of particular value in determining effectiveness of valve stake and clinch seal elastomers in contact with new formulations. The procedure is also used to evaluate new valves with standard mixtures.

More succinctly, the method permits determination of approximate weight loss due to valve seepage by the collection and measurement of gases seeping through the valve and into a special eudiometer tube, over a relatively short time period.

Apparatus

1. *Constant Temperature Bath*: The bath should be equipped with a thermo-regulator sufficient to maintain water at 80° ± 2°F. The tank should be of sufficient proportions to accommodate the necessary number of dispensers in an upright position so that each is surrounded by approximately one inch of water.

2. *Eudiometer Tube*: Two special tubes are described in Figures 1 and 2. These may be custom ordered or made by hand. The internal volume must be 5.0 ml. net; allowing for any part of the valve that might protrude into the tube. It is convenient to calibrate in 1, 2 and 3 ml. divisions.

   For tests involving many dispensers small test tubes and vials have been substituted for the special tubes.

Procedure

1. Prepare test units in accordance with production methods wherever possible, making certain that clinch diameter and depth of clinch below curl of mounting cup are in agreement with specifications. New units should be pre-tested for leakage by heating contents to 130°F.

Proposed Eudiometer Tubes

This tube is suggested for evaluation of valve and staked seals.

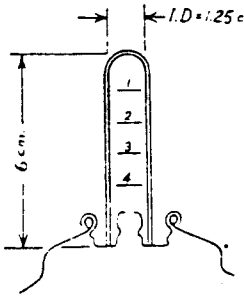


Figure 1

This tube suggested for composite evaluation of valve, staked and clinched seals.

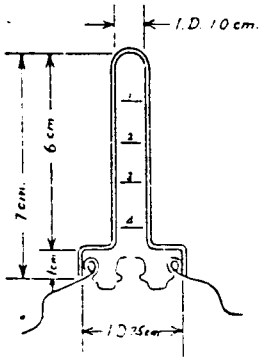


Figure 2

Dimensions approximate only — subject to geometry of valve.

2. Fill bath with water that has been allowed to deaereate for twenty-four hours at room temperature. Bring bath to 80°F. and immerse containers. Scrub bath walls, bottom and dispenser surfaces to remove adhering air. Give cans a hard knock to release air bubbles clinging to valve parts.

3. Submerge eudiometer tubes and fill. Remove air bubbles. Invert tubes over dispenser valves and allow to remain for forty-eight hours.

4. Each dispenser is given a hard knock to free clinging gas into the inverted eudiometer. The amount of gas in each tube is determined and recorded.

Calculations

The volume of gas collected in the eudiometer tube must be corrected to allow for water solubility. Since degree of solubility differs with composition of the gas, the formulas listed below must be used according to the chemical content of freshly diffused gas.

For all mixtures of P-11 & P-12:

$$V_c = V_o + 0.29 + (0.66N_{P\text{-}11}) \quad (1)$$

For difluorodichloromethane only:

$$V_c = V_o + 0.29 \quad (2)$$

For trichlorofluoromethane only:

$$V_c = V_o + 0.95 \quad (3)$$

Formulas (2) and (3) are special cases of (1). Formula (1) is simplified and accurate to 0.1 ml. only. Explanation of terms follows:

V_c = Corrected volume of gases in eudiometer tube.

V_o = Observed volume of gases in eudiometer tube.

$N_{P\text{-}11}$ = Mole fraction or vol.%/100 of trichlorofluoromethane in the gas as it is diffused into the tube. (Before selective solubility changes the gas composition.)

Corrections for aerosols containing several standard propellents are presented in Figure 4. All data is based upon use of standard 5.0 ml. eudiometer tubes.

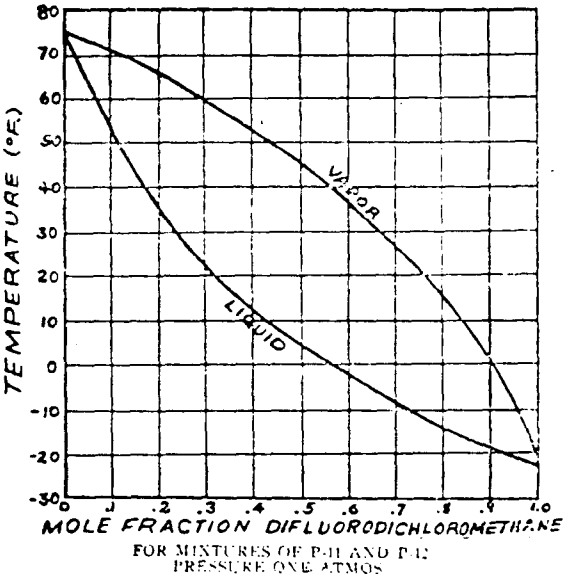


Figure 3
Boiling Point Diagram

For example, an air-free aerosol containing 50% each of P-11 and P-12 as propellents, will diffuse an observed 2.73 ml. of gas under test conditions when the seepage is 0.10 ounce per year through the valve. The gas will be predominantly difluorodichloromethane.

It can be shown that the average refrigeration-filled aerosol seeps to the extent of about 3.0 ml. when the corresponding weight loss is 0.10 ounce per year. This figure is partly based upon air content and is thus subject to variations according to filling conditions. The seepage test is not considered dependable when applied to pressure-filled, unpurged aerosols.

These disclosures will be elucidated later on.

Discussion

It must be noted that the weight loss due to seepage through the valve and "O" ring seal represents only a part of the total weight loss. Leakage will also occur at seams and seam junctures.

There is usually a one to two week adjustment period with new cans, during which some pertubations in seepage rate will occur. After this a reasonably steady day to day rate is assumed.

Many dispensers are found to rust slightly when stored under water for two days. This condition is remedied by employing a bath solution containing 0.5% sodium nitrate and 0.5% triethylene glycol in water. In more concentrated solution triethylene glycol exerts a softening effect upon enamel can finishes.

Theoretical Development of Seepage Concepts

Unless the average molecular weight of collected gas is known with reasonable accuracy it is not possible to work out a true relation between the corrected gas volume and yearly weight loss through seepage. The volume of representative gases which, under test conditions, correspond to a seepage rate of 0.10 ounce per year can readily be derived from gas laws. They are presented in the following table:

TABLE I

GAS	M.W.	V_c
Air (79% N_2 & 21% O_2)	29.0	13.6 ml.
Methylene Chloride	84.9	4.6 ml.
Dichlorodifluoromethane	120.9	3.3 ml.
Trichlorofluoromethane	137.4	2.9 ml.
sym. Dichlorotetrafluoroethane	170.9	2.3 ml.

Where two or more gases are present in the head space of a test aerosol the composition of the gas in the eudiometer tube will differ from that of the liquefied volatile constituents in the

can. Three independent factors contribute to this variation. These are:

1. Distillation Effect
2. Permeation Effect
3. Solubility Effect

and they will be discussed in that order.

A. Distillation Effect: (Raoult's Law) The partial vapor pressure of any volatile constituent of a solution is equal to the vapor pressure of the pure substance multiplied by the mole fraction of that constituent in the solution. Considering an aerosol formulation where volatile ingredients a and b are dissolved with a third, non-volatile medium, nv; where N_a, N_b and N_{nv} are thus the mole fractions of the pure components, the vapor pressure of volatile gases above the liquid phase are, at equilibrium:

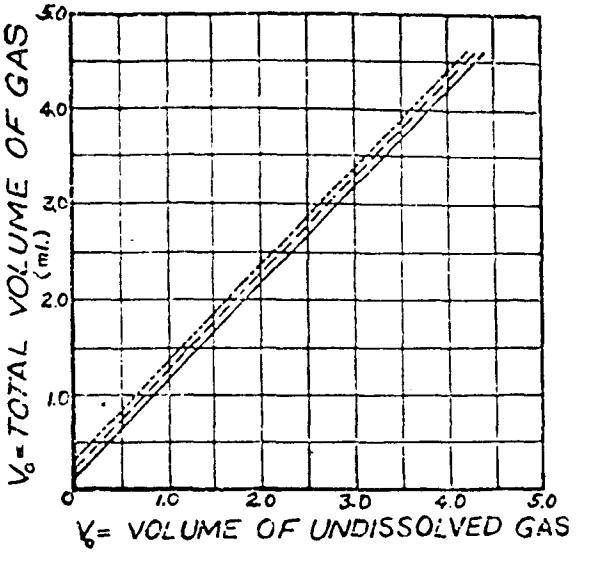
$$P_a = P^\circ_a N_a, \text{ and} \qquad (5)$$

$$P_b = P^\circ_b N_b; \text{ respectively,} \qquad (6)$$

where P°_a represents the vapor pressure of the pure propellent gas a. The total vapor pressure may then be described as

$$P = P^\circ_a N_a + P^\circ_b N_b. \qquad (7)$$

Taking the familiar Official Test Aerosol as an example, we may state the composition as:



Legend
AEROSOL LIQUID PHASE COMPOSITION:
..... 50% P-11 + 50% P-12
– – – – – 67% P-12 + 33% P-11
———— 100% P-12

Figure 4
Fate of Gases Seeping Through Aerosols and into a 5.0 ml. Eudiometer Tube Filled with Water.

volatility will concentrate in the vapor phase in proportion to the difference in volatility between it and the other propellents.

TABLE II

Ingredient	Symbol	M.W.	% (wt./wt.)	Mole Fraction
Non-volatile Components	nv	200*	15.0	0.102
Dichlorodifluoromethane	a	120.9	42.5	0.478
Trichlorofluoromethane	b	137.4	42.5	0.420

* Approximate average value.

Applying equation (7) to this case in point, we have:

$$P = P^\circ_{CCl_2F_2} N_{CCl_2F_2} + P^\circ_{CCl_3F} N_{CCl_3F}$$

$$P = 6.72 + 47.8 = 54.5 \text{ psi-absolute at } 80°F.$$

Dichlorodifluoromethane molecules are found to comprise (47.8/54.5) or 87.8% of the total number in the head space. This is also 87.8 vol.% or 86.4 wt.%.

A boiling point diagram for all mixtures of dichlorodifluoromethane with trichlorofluoromethane is presented as Figure 3. From this the head space composition of many aerosols can be readily ascertained.

It can be shown that in any system containing several propellents the one with the greatest

B. Permeation Effect: Unfortunately, the penetration of gas molecules through elastomeric barriers does not follow the idealized case represented by Graham's Law. The relative diffusion rates for such substances as methylene chloride, trichlorofluoromethane and difluorodichloromethane are quite out of proportion to the square root of their molecular weights but depend rather on their ability to function as solvents. The process of diffusion here is thought to be a consecutive adsorbtion and desorbtion of molecules on polymeric surfaces. The penetrative process is affected only slightly by moderate pressure; primarily in that a directional function is established to greater degree according to a mass action principle.

TABLE III

Ingredient	Liquid Phase Composition	Gas Phase Composition	Diffused Gas Composition
Non-volatile Components	15.0%	.....	.....
Dichlorodifluoromethane	42.5%	86.4 wt.%	68. wt.%
Trichlorofluoromethane	42.5%	13.6 wt.%	32. wt.%

Although composition, pressure and temperature conditions complicate the picture, certain studies indicate that the above three gases diffuse in the ratio of about

1 part by weight dichlorodifluoromethane (P-12)

to 3 parts by weight trichlorofluoromethane (P-11)

and 11 parts by weight methylene chloride $MeCl_2$

under equal pressures at room temperature. The elastomer used was neoprene.

Continuing with the Official Test Aerosol example the results of this penetration can be depicted by the scheme:

Also using the data in Table I, it is possible to determine the V_c value for seepage from the above aerosol which would correspond to a loss of 0.10 ounce per year. This is acplished as follows:

Using the Nearnst modification of equation (7) we have

$$V_c = V^\circ_{cCCl_2F} N_{CCl_2F} + V^\circ_{cCCl_2F_2} N_{CCl_2F_2}$$

where

$V_c = (3.3 \times 0.68) + (2.9 \times 0.32)$, or

$V_c = 3.2$ ml.

This result will apply to all insecticides and room deodorants where the active ingredients are essentially non-volatile and where the propellent is a 50:50 mixture of dichlorodifluormethane and trichlorofluoromethane.

C. Solubility Effect: The solubility of propellent gases in water is rather small at ambient temperature and atmospheric pressure. Still, it is of major importance in the determination of seepage rates.

The corrected solubility coefficient of several gases are presented in Table IV. At equilibrium, mixtures of these gases dissolve independently: (Henry's Law, Dalton's corrollary).

TABLE IV

Gas	Corrected Solubility Coefficients
Air (79% N_2, 21% O_2)	0.0175
Methylene Chloride	5.88
Trichlorofluoromethane	0.190
Dichlorodifluoromethane	0.058
sym. Dichlorotetrafluoroethane	0.035

* Defined as the number of ml. of gas dissolved in one ml. of water to give a saturated solution at 80°F. and under one atmosphere absolute pressure of the dissolved gas.

The concentration of methylene chloride vapor in the head space of aerosol insecticides almost never exceeds one or two percent by volume. Notwithstanding its propensity for penetrating elastomeric barriers, it is evident that the water solubility is so great that essentially no contribution can be made to the volume of collected gas.

Trichlorofluoromethane presents an intermediate situation. Although relatively soluble, it always makes up at least a portion of the collected gas. Referring to Table IV it is seen that 0.95 ml. can dissolve in the standard 5.0 ml. eudiometer tube before the saturation point is reached and gas begins to collect.

Dichlorodifluoromethane is fairly insoluble. It is likewise the preponderant component of gases seeping through dispensers for the compositions being considered. Almost inevitably the first bubbles of gas to form are very rich in dichlorodifluoromethane.

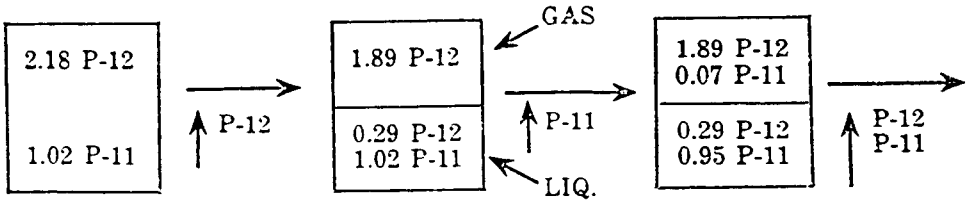
When a gas phase has once been established, the solubility of each gas in the liquid phase beneath becomes a function of its partial pressure in the head space. Thus, the formation of a bubble of difluorodichloromethane over a solution containing trichlorofluoromethane would cause a gradual translation of the latter into gas until the partial pressure of trichlorofluoromethane in the gas space became equal to

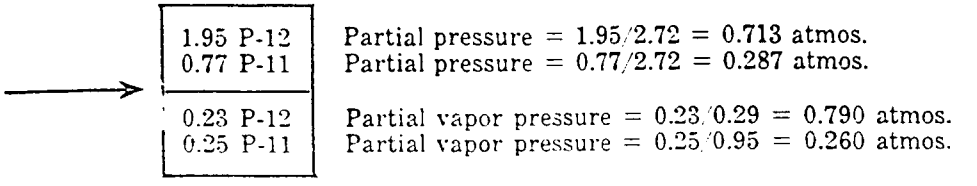
its vapor pressure in the solution. As a secondary effect, the dilution of dichlorodifluoromethane in the gas phase would cause a corresponding decrease in its partial pressure and result in an evolution of dichlorodifluoromethane from the solution until equilibrium was restored. In turn, this would upset the pressure balance of trichlorofluoromethane. Finally, an equilibrium state would be approached where the partial pressures of both gases equal the partial vapor pressures.

In practice, transitions are concurrent rather than stepwise. Also, because of the relatively short time period, low temperature and geometry of the tube, a state of complete equilibrium is never reached.

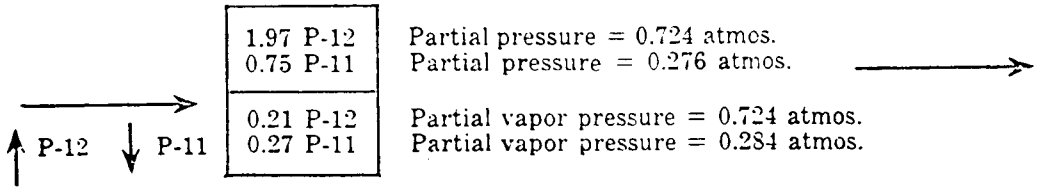
The process of reaching total equilibrium can be illustrated in a step by step manner using the Official Test Aerosol composition. A seepage rate of V_c = 3.2 ml. (0.1 ounce per year) is used.

V_c = 3.20 ml.; 68 vol.% is P-12 (dichlorodifluoromethane) = 2.18 ml.
32 vol.% is P-11 (trichlorofluoromethane) = 1.02 ml.

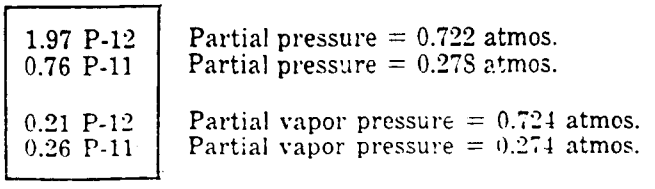




NOTE: The total vapor pressure is greater than one atmosphere, thus gas will pass into the head space from the solution. A small amount of P-12 should suffice. To correct the disparity between pressures of P-11 some should go into solution. Thus:



NOTE: The pressures of P-12 are balanced but a small excess of P-11 exists in the solution. Transferring 0.01 ml. P-11 into the gas phase we have:



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These pressures are sufficiently balanced to give an accurate picture of volume relationships at equilibrium. Over 85% of the original gas remains undissolved, giving a head space volume of 2.73 ml.

One assumption has been made in this development: that the 5.0 ml. eudiometer tube is saturated with both gases before a head space is formed. This permits water to be displaced from the tube by gas without altering the results. Since a relatively small amount of gas is actually dissolved the error introduced by making this assumption is negligible.

Using the optimal mathematical treatment described above the conditions at total equilibrium have been determined over a range of seepage volumes. The results are presented in Table V:

TABLE V

Volume of Gas Entering Tube	Composition of Gas Entering Tube	Composition of Gas in Tube at Equil.	Volume of Gas at Equil	Percentage P-12 in Gas
0.50 ml.	0.34 ml. CCl_2F_2 0.16 ml. CCl_3F	0.10 ml. CCl_2F_2 0.02 ml. CCl_3F	0.12 ml.	85%
1.00 ml.	0.68 ml. CCl_2F_2 0.32 ml. CCl_3F	0.45 ml. CCl_2F_2 0.12 ml. CCl_3F	0.57 ml.	79%
2.00 ml.	1.36 ml. CCl_2F_2 0.64 ml. CCl_3F	1.15 ml. CCl_2F_2 0.40 ml. CCl_3F	1.55 ml.	74%
3.00 ml.	2.04 ml. CCl_2F_2 0.96 ml. CCl_3F	1.83 ml. CCl_2F_2 0.70 ml. CCl_3F	2.53 ml.	72%
4.00 ml.	2.72 ml. CCl_2F_2 1.28 ml. CCl_3F	2.51 ml. CCl_2F_2 1.00 ml. CCl_3F	3.51 ml.	71%

The above results will apply to all insecticides and room deodorants where the active materials are essentially non-volatile and where the propellent consists of 50% dichlorodifluoromethane and 50% trichlorofluoromethane by weight.

In the following table it is shown that the figures for a 50:50 propellent ratio are not much different from those for higher pressure ratios or even pure dichlorodifluoromethane.

TABLE VI

V_c (ml.)	0.50	1.00	2.00	3.00	4.00
V_o 50% Dichlorodifluoromethane 50% Trichlorofluoromethane :	0.12 ml.	0.57 ml.	1.55 ml.	2.53 ml.	3.51 ml.
V_o 67% Dichlorodifluoromethane 33% Trichlorofluoromethane :	0.19 ml.	0.65 ml.	1.63 ml.	2.62 ml.	3.60 ml.
V_o 100% Dichlorodifluoromethane:	0.24 ml.	0.72 ml.	1.71 ml.	2.70 ml.	3.69 ml.

It is evident that the presence of relatively large amounts of trichlorofluoromethane in the aerosol will have little effect indeed upon the end result, V_o. For a given V_c above, the differences in V_o are always less than 0.2 ml.

This fact is of utmost importance here since it is now permissable to consider previous assumptions and approximations as instruments affecting slightly a result that, in itself, has only a minor effect upon the total determination. In other words, a minor error in determining the composition of gases entering the tube will be shown to have negligible significance in analysing for seepage rate.

For easy reference, the following V_o values corespond to seepage rates 0.1 ounce per year:

$V_o = 2.7$ ml. For all 50:50 prepellent mixtures.

$V_o = 2.8$ ml. For all 67:33 propellent mixtures.

$V_o = 2.9$ ml. For all aerosols containing dichlorodifluoromethane only as the propellent.

NOTE: Presence of methylene chloride has no effect.

The presence of air in aerosols has not been considered to this point. Most refrigeration-filled insecticides and room deodorants contain several percent of air in the head space. The amount will vary considerably, depending upon filling conditions. No adequate treatment has been given to the passage of air through aerosol dispensers; thus the diffusion rate is unknown. For test purposes the solubility of air in water can be overlooked.

To account for air in seepage gases the following treatment is suggested: Since it is known that the most important factor contributing to air in aerosols is the use of very cold materials in filling — so that vapors of the propellent are inadequate to cause complete air displacement — it appears that dispensers filled with very volatile propellents would be less likely to contain air. Such propellents would displace air effectively even at very low temperatures. The seepage due to air from these aerosols would then be less than that from units containing less volatile propellent compositions.

On the basis that some allowance is better than none, even though the magnitude remains unknown, the following V_0 values are submitted for the average aerosol, which always contains at least some air. They correspond to 0.1 ounce per year seepage rates:

For all 50% dichlorodifluoromethane, 50% trichlorofluoromethane aerosols $V_{0\,cor.} = V_0 + V_{0\,air} = 2.7 + 0.3$ ml.

$V_{0\,cor.} = 3.0$ ml.

For all 67% dichlorodifluoromethane, 33% trichlorofluoromethane aerosols $V_{0\,cor.} = 2.8 + 0.2$ ml.*

$V_{0\,cor.} = 3.0$ ml.

For all 100% dichlorodifluoromethane aerosols $V_{0\,cor.} = 2.9 + 0.1$ ml.

$V_{0\,cor.} = 3.0$ ml.

* Smaller values for $V_{0\,air}$ are used as the volatility of the propellent increases.

The value 3.0 ml. now becomes standard for all aerosols seeping at 0.1 oz. year. Because the concentration of air is often quite high in the head space of pressure filled aerosols this test is not considered reliable for such units. Operators may, at their discretion actuate pressure filled aerosols while holding in an inverted position, thus "bleeding" the air from the head space. While this does not serve to remove all air a more positive seepage study may then be made.

Summary

A rapid and inexpensive analytical method for determining the seepage rate of aerosol dispensers has been presented. In addition to a discussion of procedure, a critical survey and evaluation of results has been included. It is possible to extend this determination to other fields of aerosol research, such as determination of total seepage for glass packages.

The method is believed to possess sufficient merit to warrant acceptance as a standard method for evaluation of aerosol insecticides and room deodorants.

Approved by the Standard Methods for Insecticides and Room Deodorants Sub-Committee
Adopted by the Scientific Committee, December 3, 1956
Adopted by the Administrative Committee, Aerosol Division, December 4, 1956

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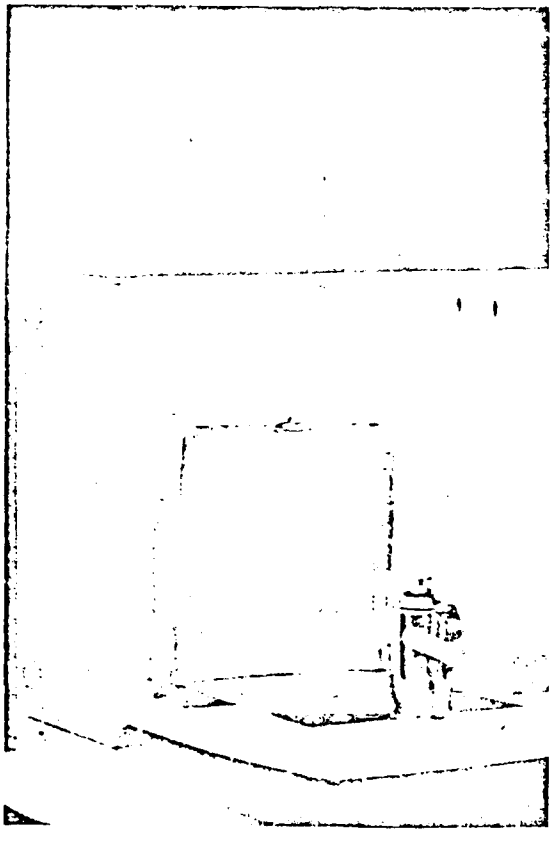
METHOD FOR DETERMINING PICKUP EFFICIENCY OF RESIDUAL AEROSOL INSECTICIDES

Introduction

Because of the wide range of particle sizes found in a residual aerosol, it is not practical to determine an accurate particle-size distribution for these aerosols by present methods. (Ref. 3). Although the efficiency of the residual insecticide depends on particle size, this efficiency can be determined more easily by the percentage of deposit than by the difficult determination of the actual particle size.

The corresponding important values for the residual insecticide aerosol are the percentage of the low-volatile ingredients that strike the target surface, and thus perform their function, and the percentage of the low-volatile ingredients that do not strike the target surface and are wasted, and what is worse, may represent a toxic hazard to the user.



Photograph of Apparatus

In this method, which is an adaptation of previous methods (Refs. 1, 2), a smooth, nonabsorbent target is used to represent an ordinary household application of residual insecticides to baseboards, cabinets, etc. In addition, any very coarse particles that would not reach the target are also collected and weighed with the target deposit. This method permits varying the target distance for fuller study of special types of residual aerosols if desired. A further feature of this method is that it provides a means for eliminating the inaccuracies caused by solvents and propellents depositing on the target and also by the variable amount of moisture condensed on the target by the spray.

Principle

The aerosol is directed from the container onto a specified target. The container and the target are weighed before and after spraying, and the target pickup is calculated as a percentage of the weight of material sprayed. The percentage of low-volatile material in the aerosol is determined, so that the efficiency of deposition of the low-volatiles can be calculated. The percentage of low-volatiles not deposited, multiplied by the concentrations of active insecticidal ingredients, gives a measure of the potential toxic hazard of using the product.

Apparatus

The apparatus required is shown in Figure 1. A box of 24" wide, 20" high and 10" deep is constructed of ¼" plywood on simple framing. The front is hinged to the bottom, and is secured to the top with a cupboard latch. Four sets of three 1" holes are drilled on lines of centers 2" from the tops and bottoms of the two sides of the box. The entire box is painted inside and out with a glossy enamel to make it

easy to clean. This box protects the target from drafts and provides for slow, steady evaporation of the target deposit.

A small triple-beam balance (Fisher 2-015 is suitable) having a capacity of 111 g. and a

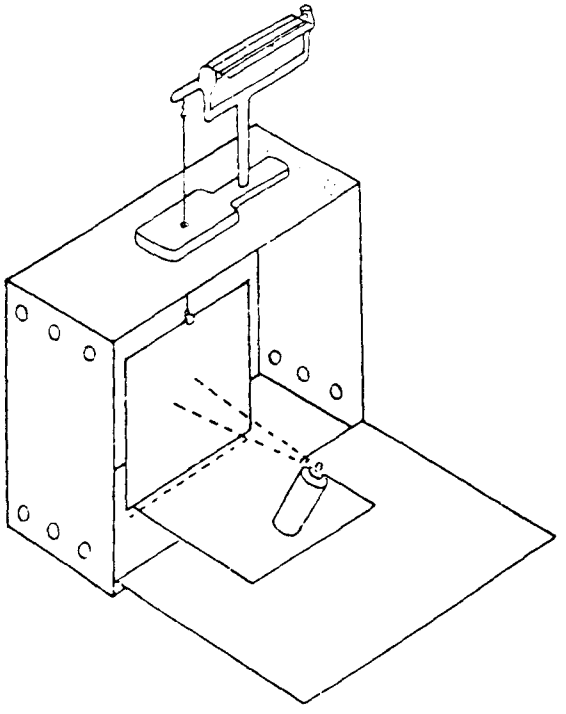


Figure 1 — Apparatus
Pickup Efficiency of Residual Aerosol Insecticide

sensitivity of 0.01 g. is altered by removing the pan and drilling a hole in the base directly under the pan hook. A corresponding hole is drilled in the top center of the box. The baalnce is bolted in position on top of the box. An extra weight, approximately equal to the weight of the pan, may be required. If so, it is attached to the pan hook, and a wire passing through the holes in the base of the balance and the top of the box is attached to this extra weight. A 1" paper clamp is attached to this wire about 14" above the bottom of the box.

The target is made from a 12" x 12½" piece of sheet aluminum about 0.020" thick. A trough is formed by bending the lower ½" of the 12½" side through about 100°. Pieces of ordinary blotting paper about 1/16" thick are cut into 12" x 12" squares. A short piece of scotch tape is placed on the paper clamp. A backstop, such as a square quart bottle (about 3½" on a side), is placed horizontally on the bottom of the box near the back center.

A balance having a capacity of 500 g. and a sensitivity of 10 mg. or less is required for weighing the aerosol unit. Fisher 1-967 is suitable.

A stopwatch and a simple interval timer are convenient but are not required.

Operation

The sheet aluminum target is clamped in the paper clamp with the trough toward the front of the box. A square of blotting paper is placed in the trough, and folded up against the target. The two bottom corners of the blotter should be clipped off to assure good contact with the bottom of the trough. The top of the blotter is held to the paper clamp with scotch tape. The backstop is adjusted so that it restrains the target from swinging but does not touch it when the target is hanging straight. The weight of the target is recorded. (This will not be the actual weight of the target unless the extra weight used weighs the same as the pan which was removed. This method requires only the difference in weight of the target before and after spraying, not its actual weight.)

The aerosol unit to be tested is weighed to the nearest centigram, shaken thoroughly, and brought to the test temperature, 80°F. The blotter is opened downward so that it lies horizontally on the open front of the box, touching the aluminum target, and pushes the target back against the backstop. The aerosol unit is held directly over the front edge of the blotter, so that the valve-to-target distance is 12". The stopwatch is started as the valve is opened, and the spray is directed at the center of the aluminum target for 5-10 seconds, so that about 10 g. of the formulation is sprayed. The blotter is immediately folded up against the target and secured with scotch tape. The box is closed, and the weight of the sprayed target is measured. The time of this weight after the spraying, indicated on the stopwatch, is recorded. The aerosol unit is reweighed, and its weight loss is recorded. The delivery rate is calculated from this weight loss and the duration of the test spraying. Comments about the valve performance, spray pattern, etc., and the room temperature and humidity are also recorded.

In addition to the initial weight of the sprayed target, its weight is also recorded at 3-minute intervals, without disturbing the box, as the spray deposit evaporates. An interval timer (Fisher 6-662-10 is suitable) is convenient for this. The gross weights of the

sprayed target are plotted versus the time after spraying, as shown on the stopwatch, and the straight portion of the plot, representing the slow evaporation of the low-volatiles, is extrapolated back to zero time to give the actual gross weight of the sprayed target. A rapid decrease in weight of the sprayed target during the first few minutes represents the rapid evaporation of volatile solvents or propellents, and of condensed moisture. Usually 2-5 minutes is sufficient to reach a slow, steady evaporation rate.

Determination of Low-volatiles

The percentage of low-volatile material in an aerosol unit may be determined by a modification of the above method. The aerosol unit is weighed and then chilled in a slush of ice and water. The top of a #16 Kraft bag is cut off so that the bag is 12" deep, and several paper towels are placed in the bottom. The bag is then weighed in the box instead of the regular target.

The aerosol unit is placed within the mouth of the bag and the valve is opened to release about the same amount of formulation as is used for measuring pickup. Because of the low temperature of the formulation, it streams with very little spray, so that all of the formulation deposits on the bag. The box is closed, and the bag is weighed at 3-minute intervals as before. The weights are plotted versus the time, and the straight portion, representing the slow evaporation of the low-volatiles, is extrapolated to zero time to give the gross weight of the bag plus the low-volatiles present. This may require 5-15 minutes. The aerosol unit is reweighed to calculate the total weight sprayed.

Pickup Efficiency of Residual Aerosol Insecticide

Notebook 52-67-1 — Date 8-27-56

Sample Exptl.

Target Weight Data

Extrapolated Wt.	81.33 g.
Initial Wt.	78.31 g.
Pickup Wt.	3.02 g.
Target Distance	12 in.
Test Temperature	80°F.

Container Data

Initial Wt.	212.50 g.
Final Wt.	195.04 g.
Wt. Sprayed	17.46 g.

Valve Type Risdon 5210 EZ

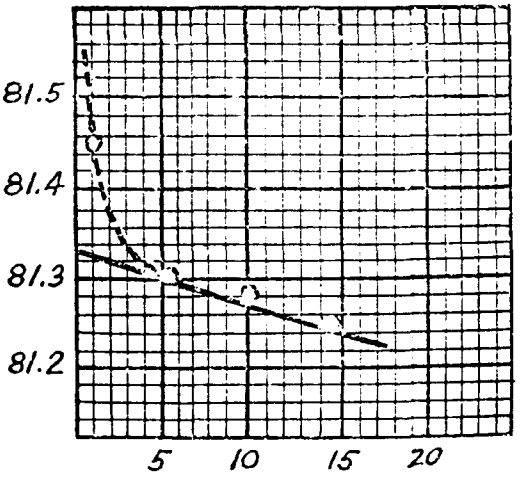
Spray Performance
OK Considerable mist

Pickup Efficiency

Wt. sprayed	17.46 g.
Pickup Wt.	3.02 g.
Pickup	17.30%
Total low volatile	25.0%
Pickup Efficiency	69.2%
Low volatile loss	30.8%

Time After Spray	Weight
1.0	81.45 g.
5.5	81.30 g.
10.0	81.28 g.
14.5	81.25 g.

Spray time	10 sec.
Wt. Sprayed	17.5 g.
Delivery Rate	1.75 g./sec.



EID12076

Figure 2

Report

A suitable report form is shown as Figue 2. Enter the initial weights of the target and of the container, the target distance (12"), the test temperature (80°F.) and the type of valve. Enter the weight of the target after spraying in the upper right opposite the corresponding stopwatch times. Enter the final weight of the container and subtract it from the initial weight to get the weight sprayed. Divide this weight by the spray time to get the delivery rate. Plot the target weight versus time readings and extrapolate the linear part to zero time. Enter this extrapolated weight. Subtract the initial weight from it to get the pickup weight. Divide the pickup weight by the weight sprayed to get the pickup as a percentage.

Enter the total low-volatile percentage either by adding up the percentages of low-volatile ingredients, including petroleum distillates, methylated naphthalenes, etc., as given on the label, or by determining the low-volatiles as described in the previous section. Divide the pickup percentage by the total low-volatile percentage to get the pickup efficiency. Subtract this pickup efficiency from 100 to get the low-volatile loss. This low-volatile loss is the percentage of each of the low-volatile components which is not deposited on the target. It is, therefore, a measure of the wasted insecticidal potency of the product, and also of the relative toxic hazard to the user.

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Results

Data obtained using this method with a variety of experimental formulations are shown in Table 1. Reproducibility on duplicate runs was within 5%. Determination of the low-volatile content was done in three cases and was within 1% of the nominal value.

TABLE I

Pickup Efficiency of Experimental Aerosol Units

No. / Composition, Wt. %	1	2	3	4	5	6	7
"Deobase" White Oil	15.0	20.0	25.0	30.0	—	25.0	—
"Sovacide" 544-C	—	—	—	—	16.	12.0	—
Methoxychlor, tech.	—	—	—	—	4.0	3.0	3.0
Pyrethrum Extract, 20%	—	—	—	—	—	—	1.0
Methyl Ethyl Ketone	—	—	—	—	—	—	56.0
"Freon-11"	42.5	40.0	37.5	35.0	35.0	20.0	—
"Freon-12"	42.5	40.0	37.5	35.0	45.0	40.0	40.0
Test Results							
Delivery Rate, g./sec.	1.5	1.5	1.8	1.5	1.1	1.6	1.0
Valve	—Risdon 5210 E-2—				VCA	K-38 .025	Prec.
Pressure at 80°F., psig.	40	39	37	36	47	44	38
Mist or Bounce-off	Much	Much	Some	Some	Much	Some	V. Sl.
Pickup, %	7.13	12.7	17.5	22.4	11.5	33.9	4.06
Total Low Volatiles, %	15.0	20.0	25.0	30.0	20.0	40.0	4.0
Pickup Efficiency, %	47.5	63.4	70.0	74.6	57.5	84.7	102*
Low-Volatile Loss, %	52.5	36.6	30.0	25.4	42.5	15.3	—

* read as: "100"

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COCKROACH AEROSOL METHOD

I. Introduction

Starting in 1942 extensive studies were made by various CSMA and Federal laboratories to develop a standard test method for evaluating aerosol insecticides against flying insects. This work led to the adoption of the CSMA Aerosol Test Method for Flying Insects on October 12, 1952 (1), following the use of essentially the same method on a tentative basis starting in 1949 (2). During the same period a number of laboratories developed methods for evaluating aerosol insecticides against cockroaches. Descriptions of several of the methods appear in the literature (3, 4, 5), while other techniques were reported at meetings of the Insecticide Scientific Committee.

Early efforts of the Committee to develop a standard cockroach aerosol method centered around the use of space treatments in Peet-Grady or larger aerosol chambers with the insects exposed in suitable open cages. Following a cooperative test of a space spray method in 1952 and 1953, the Committee decided to use a direct spray treatment rather than a space treatment. It was also decided to use large nymphs of the German cockroach as the test insect and to try to adapt the procedure to the use of the same spray chamber specified for the Official Cockroach Spray Method (6). During 1954 a direct spray method was developed and studied by several CSMA and Federal laboratories. In a cooperative series of tests (early 1955), using coded dispensers with three different formulas (2.0 per cent DDT plus 0.2 per cent, 0.4 per cent, and 0.8 per cent pyrethrins), all five laboratories were able to differentiate between the three formulas. A decision was then made by the Committee in May, 1955, to prepare the method in official form so that it might be considered for adoption on a tentative basis.

Reports of the Committee's studies appear in the minutes of the Insecticide Scientific Committee and the Committee reports in the CSMA PROCEEDINGS OF THE 42nd ANNUAL MEETING. (7, 8, 9, 10)

In this method it should be understood that the term "aerosol" applies to pressurized formulations containing 20 per cent by weight or less low volatile ingredients (insecticides, base oils, solvents, etc.) and 80 per cent or more propellent liquefied gases (trichloromonofluoromethane, dichlorodifluoromethane, methylene chloride, etc.). Whether the method can be satisfactorily applied to formulations containing greater than 20 per cent low volatile ingredients will depend on additional study and cooperative tests.

The method here described is thought to offer a satisfactory means of determining the relative efficiency of aerosol formulations when applied as direct sprays to cockroaches. It is not designed to measure residual action. As a biological test it is subject to the variations that accompany the reaction of living organisms and should be employed under the supervision of a person familiar with the biological testing of insecticides. In order to measure with reasonable tolerance the relative effectiveness of different insecticides, the test is run in conjunction with the Official Test Aerosol, which is designated as the basis of comparison. The format of the method follows that of the Official Cockroach Spray Method.

II. Apparatus

A. Reference Insecticide. The reference insecticide shall be the Official Test Aerosol (OTA) prepared by the CSMA. The OTA must be dispensed from the container in which it is supplied with particular care being taken that the OTA dispenser employed meets the specifications designated on its label.

B. Dispenser for Experimental Aerosol. No restriction is made on the dispenser employed in connection with the experimental aerosol formulation. However, it should be noted that the test results apply only to the experimental formulation as dispensed from the particular unit employed. In reporting results, the dispenser used with the experimental aerosol shall be specified.

C. Test Insect. The test insects shall be healthy, normal, undeformed last nymphal instars of the German cockroach, *Blattella germanica* (Linn.). Recently emerged last nymphal instars, e.g., those whose pigmenta-

tion is not dark shall not be used for testing purposes. It is recommended that the last nymphal instar stage shall have been attained at least three days prior to testing.

D. Rearing Room. This room may be of any convenient size, constructed so as to be free from strong drafts and maintained at a temperature of 75 to 85°F. and a relative humidity of 30 to 50 per cent. It should be separate from the testing room in order to eliminate the possibility of traces of insecticide coming in contact with the test insects. Ventilation should be provided to reduce odor.

E. Testing Room. This room may be of any convenient size permitting adequate space for the operator to handle the test efficiently. While tests are being conducted this room shall be maintained at a temperature of 78 to 82°F. It is suggested that relative humidity be held between 30 and 50 per cent.

F. Spray Chamber. The spray chamber shall be a box-like structure of solid material measuring 18 inches wide, 18 inches long, and 25 to 30 inches high. The open floor of the chamber shall be covered with ½-inch mesh wire hardware cloth. Suitable guides shall be fastened to the chamber floor to permit the centering of the treatment container in some definite position in respect to the nozzle of the OTA and test aerosol dispensers. The top of the chamber shall be open. The front wall of the chamber may be in the form of a sliding door permitting convenient access to the interior of the chamber. The chamber shall rest on a stand, placing it at the proper height for convenient operation of the test.

An adjustable hinged shelf shall be affixed to the outside of the center of the back upper edge of the spray chamber. Any suitable shelf (see Figure 1) which will permit the OTA and test aerosol dispensers to be held in a standard position can be used. One satisfactory shelf is made of aluminum sheet fitted with adjustable guides (in slots with wing nuts) which permit adjustment for dispensers of different sizes. An adjustable metal support rod (casement window adjuster) can be used to regulate the angle of the shelf. Markings can be made on the rod and plate to permit rapid adjustment for different dispensers.

FIGURE 1—*Apparatus*

The above-described spray chamber is identical with that specified for the C.S.M.A. OFFICIAL COCKROACH SPRAY METHOD, except that the braces and mounting for the spray atomizer have been removed and the hinged shelf for holding the aerosol dispenser has been added.

G. Treatment Container. The treatment container shall be a screen-bottomed container 3½ inches in diameter with 3-inch side walls. Sixteen mesh wire screen shall be soldered in place to form the bottom of the container in such a manner that the entire bottom is completely open. Ordinary tin cups of the proper dimensions with handles removed and the solid bottom replaced by wire screening have been found useful as test containers.

H. Insecticide Paper. Unsized, nonglazed, absorbent paper such as brown kraft or gray bogus paper shall be used beneath the treatment container during the application of the aerosol mist. No special weight is specified, although 60 to 80-pound gray bogus paper has been found excellent.

I. Recovery Dishes. Glass crystallizing dishes measuring 125 millimeters in diameter and 65 millimeters high shall be employed as recovery cages. The bottoms of the recovery dishes shall not be covered with filter paper or other material. Sixteen mesh wire screen covers may be employed as recovery dish covers during the 48-hour holding period following spray application in order to prevent the entry of wild roaches.

III. Procedure

A. Rearing of Test Insects. Any suitable method permitting the production of large numbers of the test insect under controlled conditions of temperature and humidity as previously described may be employed. The rearing technique described by Woodbury and Barnhart (11), which makes good use of a brood chamber containing adult females from

which large numbers of first instar nymphs may be collected at frequent intervals, has been successfully used in a number of laboratories. All molded food, dead females, and empty egg cases should be removed weekly. Wild cockroaches shall not be used, and all test insects shall have been reared under uniform conditions.

B. Food. Up until the time of testing, cockroaches shall be provided at all times with food and water. The standard food shall be Purina Laboratory Chow manufactured by the Ralston Purina Company, St. Louis, Missouri, or equivalent. (12)

C. Test Procedure. Last instar nymphs shall be isolated in the recovery dishes or other suitable containers from the cultures in groups of 20 by means of a suction device, by anesthetizing them with carbon dioxide gas or any other suitable method which does not injure them. In selecting the test insects, every effort shall be made to obtain uniform test groups.

Prior to use, the OTA and test aerosol dispensers shall be calibrated at 82 ± 2°F. to determine their spray rate in grams per second. The dispensers shall be aligned on the adjustable shelf and settings determined for the various dispensers to permit rapid handling. The dispensers shall be so aligned that the aerosol mist is directed into the open top of the treatment container which rests on the floor of the spray chamber. The treatment container shall be placed against the center of the front wall of the spray chamber. Suitable guides on the chamber floor or front wall of the chamber will aid in placing the treatment container in a standard position. A line may be drawn on the chamber wall and on the dispenser to aid in aligning the nozzle with the center of the top of the treatment container. The treatment container shall rest on a 5 x 5 inch square of insecticide paper. Except for the 5 x 5 inch square of paper, the chamber floor ($\frac{1}{2}$ inch mesh wire hardware cloth) shall be open. The paper shall be changed after each spray application.

Immediately before spray application the cockroaches shall be transferred to the screen-bottomed treatment containers. These containers shall be free from all traces of insecticides and shall have the entire inner wall surface suitably oiled or greased to prevent the escape of the cockroaches and to confine them to the container floor. Prior to spray application the treatment container shall be agitated sufficiently to distribute the test insects uniformly over the container floor. The treatment container shall be removed from the spray chamber 30 seconds after the start of spray application. The test insects shall be immediately transferred from the treatment container to the recovery dish. The treated cockroaches shall be held under rearing room conditions through the 48-hour observation period and shall receive neither food nor water.

In evaluating a test sample, a minimum of 10 individual test groups shall be run for the test dispenser in conjunction with 10 test groups receiving the OTA. An equal number of replicates shall be made for members of any given test series on a given test day. The average dosage employed shall be approximately the same throughout a given series of tests and of such magnitude as to result in an average of 50 per cent to 75 per cent of the insects dead and moribund at 48 hours with the OTA. Average dosages shall be considered the same if they agree within 0.2 grams. Cooperative tests among CSMA laboratories have shown the required dosage to be 2.0 to 4.0 grams. Test dispensers shall be weighed before and after the spraying of each test group of insects, and the weight of material used shall be recorded.

D. Assembly and Evaluation of Data. Evaluation of test dispensers shall be made on the basis of observations taken 48 hours after spray application, at which time the percentage of test insects normal, moribund, and dead shall be determined. Any insect showing signs of life but incapable of locomotion shall be considered moribund. Similar records taken at 24 hours or at intervals longer than 48 hours may be of interest in critical studies. It is recommended that if the test insects are to be held under observation longer than 48 hours, they be furnished food and water at the end of the initial 48-hour observation period. Insects that withstand insecticide treatments shall be destroyed and in no case returned to the stock cultures or employed in further tests.

The basis of comparison shall be the average percentage dead and moribund of the test sample as compared with that of the OTA. In reporting the test results the test sample shall be reported as "meeting the standard" if its average percentage dead and moribund determination at 48 hours is equal to, greater than, or within 10 percentage points less than that of the OTA employed in conjunction with it. In no case shall actual numerical values be reported officially or any letter grade designation be assigned to the test samples as a measurement of their effectiveness against cockroaches. The following table records the results of a typical series of tests.

Example of Test Data

Test	Date	OTA Dosage (grs.)	OTA % D&M 48 hrs.	Sample A Dosage (grs.)	Sample A % D&M 48 hrs.	Sample B Dosage (grs.)	Sample B % D&M 48 hrs.
1	2/15/55	3.1	90	3.3	75	3.2	35
2	2/15/55	3.2	80	3.8	80	3.1	40
3	2/15/55	3.2	35	2.0	65	2.9	20
4	2/22/55	3.3	50	3.2	75	3.2	35
5	2/22/55	2.7	55	3.3	75	3.1	50
6	2/22/55	3.0	80	2.9	60	2.8	35
7	2/22/55	3.0	60	3.1	65	3.3	35
8	2/22/55	2.9	50	3.0	80	2.9	25
9	2/22/55	2.9	60	3.1	75	2.9	35
10	2/22/55	3.3	35	3.0	70	3.6	50
Average		3.06	59.5	3.07	72.0	3.10	36.0

Reported as follows:
Sample A—Meets Standard
Sample B—Does not meet Standard

IV. Conditions for Official Evaluation

A. The test shall be conducted in accordance with the procedure previously described.

B. Twenty test groups of insects, numbering 20 cockroaches each (10 test sample, 10 OTA), shall be employed in making an official evaluation.

C. The average dosage shall be approximately constant throughout a given series of tests and of such magnitude as to give an average of from 50 to 75 per cent of the OTA treated cockroaches dead or moribund 48 hours after spray application.

D. The toxicity of the test sample shall be reported as meeting the standard if its average percentage dead and moribund determination at 48 hours is equal to, greater than, or within 10 percentage points less than that of the OTA run in conjunction with it. In no case shall numerical values be reported or any letter grade designations be assigned to the test samples as a measurement of their toxicity to cockroaches.

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At the meeting of the Board of Governors of the Chemical Specialties Manufacturers Association on December 7th, 1955, the following action was taken:

"A motion was made, seconded and adopted that the Official Aerosol Cockroach Test be adopted as a Tentative Specification and published in the Proceedings."

Adopted by the Insecticide Division Scientific Committee, December 5, 1955
Adopted by the Board of Governors, December 7, 1955
(Revised May 1959)

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TEST METHOD FOR EVALUATING AEROSOL AND PRESSURIZED SPACE SPRAY INSECTICIDES AGAINST FLYING INSECTS

I. Introduction

Early in the developmental period of liquefied gas aerosols starting in 1942, and especially following their appearance on the civilian market on a large scale in 1946, the need for a common method of biologically assaying aerosols became apparent. The literature records several testing techniques (among them 1, 2, 3, 4, and 7) employed by various investigators at that time, but the necessary cooperative tests leading to the development of an official method were not initiated until 1947. The first series of cooperative aerosol tests among industrial and federal laboratories was organized and conducted in 1947 under the direction of the NAIDM* Aerosol Committee (8). These tests employed a standard formulation in a standard dispenser at three dosage levels by the method in current usage in the cooperator's laboratory. Employing the results of the first cooperative tests as a basis, a second series of cooperative tests was designed and conducted under the direction of the NAIDM's Insecticide Scientific Committee. In this second series of tests made during the period May to October, 1948, four conventional low pressure aerosol formulations each packaged in a standard dispenser, were tested by nine cooperating laboratories. In these tests (9), the use of free flying flies, a standard dosage and a standard testing technique were employed. A third series of cooperative tests were conducted by ten participating laboratories. These tests (10) made use of three coded formulations containing 0.2%, 0.4%, and 0.8% pyrethrins. The method here presented is based on the outcome of the first, second, and third series of cooperative tests and, in so far as practical, follows the Official Peet-Grady (11) Test Procedure (both large and small group). This technique for testing aerosols should be regarded as a practical test method designed for the comparison of formulations in the dispensers in which they will be employed by the consumer. It is restricted at present for use against house flies, although it is felt that with modifications in dosage the general procedure would be satisfactory for other flying insects.

With the growth of pressurized insecticides between 1949 and 1958, it was felt by the CSMA that a grading system and a distinction between aerosols and the newly developed pressurized space sprays should be made. In view of the changing trade practices, therefore, definitions (12) of aerosols and pressurized sprays, and a grading system are now incorporated in this method. Grading of aerosols and pressurized sprays is set forth under Section IV Conditions for Official Evaluations, and definitions for aerosol and pressurized sprays are as follows:

1. Aerosols: The spray from aerosol dispensers should be in finely divided form in which 80 percent or more of the individual spray particles have a mean diameter of 30 microns or less, and none of the spray particles has a diameter of more than 50 microns. Aerosols must be no less effective in biological performance (5-minute, 10-minute, and 15-minute knockdowns and 24-hour mortality) than the Official Test Aerosol (OTA) when tested against house flies at the same dosage or less.

2. Pressurized Sprays: These products deliver mist sprays intermediate between aerosol-type sprays and those which are intended to deposit an insecticidal residue of a chemical. They must be no less effective in biological performance (15-minute knockdown and 24-hour mortality) than the Official Test Aerosol (OTA) when tested at no more than twice the dosage specified for the OTA.

II. Apparatus

A. Reference Insecticide.

The reference insecticide shall be the current Official Test Aerosol (OTA) prepared by the CSMA. The OTA must be dispensed from the container in which it is supplied with particular care being taken that the OTA dispenser

employed meets the specifications designated on its label. References to the OTA (other than in this presentation) should identify the OTA by date.

B. Dispenser for Experimental Insecticide.

No restriction is made on the dispenser employed in connection with the experimental aerosol or other pressurized formulations. However, it should be noted that the test results apply only to the experimental formulation as dispensed from the particular unit employed. In reporting results, the dispenser used with the experimental aerosol shall be specified.

C. Test Insect.

The test insect shall be the house fly *(Musca domestica,* L.) reared from strains mixed under the supervision of the CSMA. Healthy test groups having an average age of four days shall be used and individual flies in the test groups shall be not less than three nor more than six days old at the time of testing. The strain shall be of such susceptibility that the Official Test Insecticide (OTI) will cause a 24-hour mortality of 30 to 55 per cent and with approximately 95 per cent of the flies paralyzed at ten minutes following spray application by the Peet-Grady method (11).

D. Fly Cages.

Cages of any convenient type may be used if they provide at least 1 cubic inch of space per fly and have at least two sides and the top screened. It is suggested that the base be square in shape to provide maximum floor space. The floor of the cage is preferably detachable to facilitate cleaning and inserting a paper floor covering. The cages are constructed of wood or other suitable material and 16 mesh wire screening; they are fitted with a sleeve opening, rubber membrane, or a door.

E. Rearing Room.

This room may be of any convenient size constructed so as to be free from strong drafts, and maintained at a temperature of 82 ± 2 degrees Fahrenheit and relative humidity of 50 ± 5 per cent. It should be separate from the testing room in order to eliminate the possibility of traces of insecticide coming in contact with the test insects. Ventilation should be provided to reduce odors and gases from fermenting media.

F. Testing Room.

This room shall be of any convenient size, capable of holding the aerosol test chamber (Peet-Grady Chamber or larger chamber) and permitting adequate additional space for the operator to handle the test efficiently. While conducting tests, this room shall be maintained at a temperature of 75 to 85 degrees F. It is suggested that the relative humidity be held between 40 and 70 per cent. Since the exhaust fan of the chamber will move relatively large quantities of air, the temperature of the air entering this room should be approximately that specified above.

G. Aerosol Test Chamber.

The test chamber shall be a Peet-Grady Chamber as specified in the Peet-Grady Method, or a larger chamber meeting the general specifications of the Peet-Grady Chamber. In the cases of larger chambers, it is recommended that the dimensions be such as to approximate a normal room.

H. Exhaust Fan.

An exhaust fan moving not less than 1,000 cubic feet of air per minute through the Peet-Grady Chamber, or a fan of proportionately larger capacity for testing chambers larger than the Peet-Grady Chamber shall be used to ventilate the chamber after each test. It shall be arranged with adequate piping to exhaust the chamber vapors outside of the building.

I. Insecticide Paper.

Unsized, nonglazed, absorbent paper, such as brown kraft or gray bogus, shall be used to cover the chamber floor. No special weight is specified although 60 to 80-lb. gray bogus paper has been found excellent. In certain laboratories testing chamber ceilings and walls have been covered with cardboard, kraft paper or other material suitably arranged for easy renewal to reduce chamber cleaning difficulties.

J. Apparatus for Picking Up Flies.

Any convenient means of picking up the paralyzed flies without injuring or appreciably disturbing them may be used. If a vacuum device is used, it must produce gentle suction, have a sufficiently large receptacle to prevent crowding of the flies, and shall be cleaned after each test with the same materials used in cleaning the chamber.

III. Procedure

A. Rearing and Handling Flies.

In this procedure, eggs are transferred to medium suitable for the development of larvae. The pupae are collected from the medium and placed inside of cages, and the adult flies emerge and remain in these cages until the day of testing. A culture is defined as all adults resulting from the seeding of eggs collected at one time on a given date.

(*a*) *Larval medium.* The preferred containers are cylindrical glass battery jars approximately 6 in. in diameter and 9 in. high. For one jar mix 340 gm. (12 oz.) dry CSMA Standard Fly Larval Media[1] with approximately 750 cc. of an aqueous suspension containing 15 gm. moist cake yeast[2] or 5 gm. dry yeast[2] and 10 cc. non-diastatic Diamalt[2]. Mix thoroughly until a loose, fluffy medium is obtained, transfer it to the battery jar without packing, cover with cloth and set in the insectary. The amount of suspension required for best rearing results will need to be determined in each laboratory and it may be varied in order to prevent mold growth. It is suggested that the medium be prepared in the late afternoon of the day before egg collection.

(*b*) *Eggs*: Eggs are collected for a period not longer than 16 hours from food dishes or other oviposition media in cages containing mature flies not more than 8 days old. It is suggested that fresh oviposition medium be placed in fly cages in the late afternoon and eggs be collected early on the following morning. After collecting the eggs they must be measured and seeded without delay. Wash the eggs in tap water at room temperature and measure 2,000 eggs as accurately as possible. This may be done by allowing the eggs to settle in a calibrated pipette or graduate (0.1 cc. settled eggs contains about 700) or the eggs can be filtered and measured in calibrated pits or cells. Use 10 cc. of tap water to measure and to scatter the eggs in a pit or trench 0.5 inches deep and located in the center of the surface of the larval medium. Cover the eggs with loose medium, replace the cloth covers on the jars, and set jars in the insectary so that at least 1.5 inch separates each jar to permit free air circulation. The maximum temperature in the jar (about 3 days later) must not exceed 130°F. Under normal conditions, more than 85 per cent of the eggs should hatch within 36 hours of the time they are laid.

1. Order from Ralston Purina Co., St. Louis, Mo.
2. Diamalt and yeasts are products of Standard Brands, Inc., and are usually available from local distributors.

(*c*) *Pupae*: Mature larvae migrate to the top portion of the medium and normally all larvae will have pupated by the seventh day after seeding eggs. When this occurs, the portion of the medium containing pupae is loosened, poured into a shallow tray, and air dried at room temperature. An electric fan may be used to hasten drying. Pupae are separated from the dry medium by sprinkling the pupae-medium mixture on an inclined tray or chute set in front of an air blast such as that from an electric fan. The pupae must be handled gently and as little as possible in order to avoid injury. Any method that permits at least 95 percent of flies to emerge is considered satisfactory.

An air separation apparatus (5) for separating pupae is used by several laboratories and found to be more rapid than the inclined tray method. The apparatus employs a blower, a cyclone collector, and a suction pipe. The device separates the heavier pupae from a layer of vermiculite placed on the surface of the fly medium before the larvae pupate. According to (6), a 2-inch layer of vermiculite should be placed on the larval medium three or four days after seeding. Six days after seeding, the mixture of vermiculite and pupae is loosened and poured on a tray and separated. Terra Lite Brand Vermiculite Soil Conditioner, available in 20-pound bags from some garden supply stores, has been found satisfactory for use in this procedure.

All of the pupae maturing on a given day are combined into one lot, mixed, and measured into test units. Each group is placed in a shallow dish which is, in turn, placed in a cage which provides at least 1 cu. inch of space per pupa. If the large group procedure is used, the test unit consists of approximately 500 pupae. If the small group procedure is used, more than 500 pupae are placed in stock cages and adult flies are sampled prior to testing. Under normal rearing conditions, at least 80 adult flies should be obtained for each 100 eggs seeded.

(*d*) *Adult Fly Food*: The food for adult flies shall consist of 5 per cent spray dried, nonfat milk solids and 2 per cent granulated sugar thoroughly dispersed in water. Roller dried or caked milk solids settle out of suspension within a few hours and are unsuitable as food. A 40 per cent formalin solution may be added to the food at the rate of 1/1500 to delay souring. Each cage is supplied daily with a dish containing at least 15 cc. food for

each 100 flies, and so prepared as to prevent the flies from drowning. Satisfactory food must be available to the flies at all times. The adult flies are held until the second day of oviposition (usually 12 to 14 days after eggs are seeded) when they are ready for testing.

B. Testing Flies.

Before a test is started, the test chamber must be clean and have clean paper on the floor, all ports and openings must be closed, and the temperature must be 82 ± 2°F., and all windows must be equally shaded. In chambers where walls and ceilings are covered with paper or other material, contamination, if present, must be at sufficiently low levels so as not to influence test results. Chambers are considered to be contaminated and unsatisfactory for test use when test flies, held in them for a 12 to 16 hour period with food but without insecticide treatment, show mortalities in excess of 10%, or when over 10% of the flies are paralyzed within 30 minutes after liberation. It is recommended that laboratories make a standard practice of taking contamination observations, employing a normal fly test group, following each day's testing. In both the large and small group procedures, only flies which are capable of flying shall be liberated into the aerosol test chamber. In the small group method, a sample of 100 ± 5 flies is used in each test; but in the large group method, all flies in one cage are used in a single test. Samples may be taken by liberating the flies directly into the chamber and continuing until about 10 per cent of flies remain in the stock cage. These are discarded. Samples may be taken also by discarding the first 10 flies and then counting 50 flies into each of a series of small cages. One hundred flies are counted into the last cage and, working backward, 50 flies are added to each. Flies remaining in the stock cage are discarded. The order of spray treatments must be randomized.

After liberating the flies in the chamber, and with the container at 82 ± 2°F., a total of 3.0 ± 0.5 grams of aerosol mixture per 1000 cubic feet for a Grade A aerosol or pressurized spray evaluation shall be applied in a continuous flow. For a grade B pressurized spray evaluation, a total of 6.0 ± 0.5 grams of mixture per 1000 cubic feet shall be applied in a continuous flow. In Peet-Grady Chambers, this is 0.648 ± 0.108 grams for a Grade A spray and 1.296 ± 0.108 grams for Grade B. The dispenser nozzle may be oscillated slowly to effect uniform distribution of the mist within the test chamber.

The mist shall not be directed onto chamber wall and testing surfaces. The test dispenser shall be weighed before and after the liberation of the aerosol or pressurized space spray mixture and the actual weight of material introduced shall be recorded. The chamber is closed at a constant temperature in the range of 82 ± 2°F. for 15 minutes from the time the mist is introduced.

Where a Peet-Grady Chamber is used, the nozzle or exit orifice of the valve should be directed so that the spray goes through a port, meeting specifications of the Peet-Grady Method. Several CSMA laboratories use adjustable fixtures to hold the dispenser and permit the spray to be distributed from the same place and angle for each test. Different adjustments may be necessary for the OTA and the various test dispensers, and the spray pattern from new dispensers should be determined prior to the test. One laboratory reports successful use of dispensers with the nozzle positioned 8 inches from the ceiling and 10 inches from the corner of the Peet-Grady Chamber.

Counts shall be made as to the number of flies "down" (paralyzed) at 5 and 10 minutes following insecticide application. These counts are especially important because with conventional formulations practically all flies "down" at 15 minutes fail to recover during the 24-hour observation period. At the end of 15 minutes the ports are opened and the chamber is ventilated by means of the exhaust fan while the flies are collected.

The "down" flies are picked up and transferred immediately to clean cages meeting the specifications of Section II, paragraph D. These flies may be counted when they are picked up or later, depending upon which time is more convenient. During the subsequent 24-hour recovery period, the cage is placed in the rearing room and supplied with an adequate quantity of 5 per cent sugar solution, arranged so that the top of the dish is not more than ¾ inch above the floor of the cage and the flies cannot drown in it. A gauze-wrapped ball of cotton saturated with 5 per cent sugar solution is also satisfactory.

The "up" (unparalyzed) flies in the chamber at the end of the 15-minute exposure period are counted and discarded.

After a test is completed all toxic residues must be removed from the chamber or, if allowed to remain, must be at sufficiently low levels so as not to affect test results. Where chamber surfaces permit many toxic residues can be removed by wiping with a clean cloth saturated with ethyl alcohol or ethyl alcohol containing 10% acetone.

C. Assembling the Data.

The number of "up" flies must be counted and recorded at the end of the 15-minute exposure period. The dead flies are counted 24 ± 1 hours later, preferably by removing them from the recovery cage. Only flies that show no sign of life upon being touched may be counted dead. If the "down" flies were counted as they were collected, the sum of the "down" and the "up" flies yields the total flies in the test. If the "down" flies were not counted as collected, the recovered flies are killed by placing the cage in an oven at 170°F. for a few minutes, after which they are counted. The sum of recovered and dead flies yields the "down" flies and this sum added to the "up" flies yields the total flies used in the test. The *Aerosol Test Knockdown Mortality* is the per cent dead of total flies. In the *Aerosol Test Knockdown Mortality* calculation, the "up" flies at the end of the 15-minute exposure period are considered to be alive at the end of the 24-hour observation period. The *Aerosol Test Knockdowns* are the per cent "down" of total flies at 5, 10 and 15 minutes.

The mortality and knockdown definitions are summarized in equation form as follows:

(1) Aerosol Test Knockdown Mortality =

$$\frac{\text{Dead "Down" Flies} \times 100}{\text{Total Flies}}$$

(2) Aerosol Test Knockdown, 5, 10 or 15 minutes =

$$\frac{\text{"Down" Flies} \times 100}{\text{Total Flies}}$$

IV. Conditions for Official Evaluation

1. The tests shall be conducted in accordance with the procedure previously described.

2. At least two cultures of flies, meeting Peet-Grady specifications, shall be used in making an official evaluation.

3. Cages showing a combined mortality and crippling greater than fifteen per cent on the day of test shall not be used.

4. In the small group procedure, using approximately 100 flies per test, no more than three unknown samples may be tested in conjunction with one OTA in any one series. Ten tests are run on the OTA and on each of the unknowns in parallel; that is, test each spray the same number of times on any one day. The samples of a series must be randomized in the order of testing.

5. The large group procedure using approximately 500 flies per test shall be conducted in the same manner as outlined for the small group procedure with the exception that five, rather than ten, tests are required.

6. The dosage of the OTA and experimental aerosol shall be 3.0 ± 0.5 grams per 1000 cubic feet, while that of the experimental pressurized space spray may be either 3.0 ± 0.5 grams or 6.0 ± 0. 5grams per 1000 cubic feet, depending upon whether efforts are being made to qualify the experimental pressurized space spray as a Grade A (3.0 ± 0.5 grams) or Grade B (6.0 ± 0.5 grams) insecticide. The experimental aerosol or pressurized space spray tested at 3.0 ± 0.5 grams dosage shall be reported as "meeting the standard" if its knockdown at 5, 10 and 15 minutes and 24-hour. *Aerosol Test Knockdown Mortality* is equal to, greater than, or not less than 5 percentage points below that of the OTA run in conjunction with the experimental insecticide. The experimental pressurized space spray tested at 6.0 ± 0.5 grams dosage shall be reported as "meeting the standard" if its knockdown at 15 minutes and 24-hour *Aerosol Test Knockdown Mortality* is equal to, greater than, or not less than 5 percentage points below that of the OTA run in conjunction with it (the OTA being tested at 3.0 ± 0.5 grams dosage).

7. The percentage knockdown and mortality of the insecticides, provided they "meet the standard," shall be designated by letter and shall be in accordance with the following grading system:

Grade	*Type of Insecticide*	*"Equal to" OTA at dosage of 3.0 ± 0.5 grams per 1000 cubic feet at dosage indicated below*
A	Aerosol or Pressurized Space Spray	3.0 ± 0.5 grams per 1000 cubic feet
B	Pressurized Space Spray	6.0 ± 0.5 grams per 1000 cubic feet

8. The *Official Test Aerosol* (OTA) is restricted to use in the above described procedure and the CSMA Cockroach Aerosol Test Method (14). It shall be used only as the reference insecticide in testing aerosol and pressurized insecticides against the house fly and aerosol insecticides against cockroaches.

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13. A report on Fleishmann's Active Dry Yeast for Bakers, Mimeo. Letter from D. F. Starr to C.S.M.A. Insecticide Committee members dated 1/7/53. Unpublished.
14. Cockroach Aerosol Test Method (latest reference, 1961 Blue Book or Proceedings of December 1959 C.S.M.A. Meetings).

Prepared and Presented by the Insecticide Division Scientific Committee
Reviewed and Accepted by the Aerosol Division Scientific Committee
This Method was adopted by CSMA Board of Governors. October 5, 1960.
It Supersedes CSMA Aerosol Test Method for Flying Insects adopted
by the Board of Governors, December 10, 1958, CSMA Proceedings, May, 1960.

LABORATORY SERVICE

Many commercial laboratories are properly equipped and qualified to check any aerosol or pressure package for a fixed charge.

The laboratory should report the following on the product samples submitted:

1. Labeling —
   Correct caution labeling for product.
   Type of shipping label required.
2. Complete test for flammability or combustibility in accordance with the "Flammability Tests for Pressurized Containers," and ICC Regulations.
3. "Fill" in accordance with minimum fill and safe fill requirements.
4. Pressure (Gage) at 70°F and 130°F.
5. Conduct tests outlined in the "CSMA Aerosol Guide."

Not less than three samples of each product shall be submitted.

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METHOD FOR DETERMINING PRESSURE DROP RATE

A revised method for the determination of pressure drop rate is outlined below.

To develop a satisfactory method for pressure drop rate, it is desirable that the procedure satisfy the following requirements.

(1) Method should be applicable to all food products packaged in pressurized containers.

(2) Method should be rapid and reproducible.

(3) Dispensing conditions encountered in actual usage should be simulated.

(4) Method should be such that any slight variances in valves and actuators are overcome.

(5) Method should be such that the final equilibrium pressure in the container can be correlated to the amount of product dispensed.

To satisfy the above conditions, a tentative method for pressure drop rate has been established.

METHOD

A. Storage Conditions

(1) Products normally refrigerated should be held at 40°F. for at least 24 hours before testing.

(2) Products normally stored at room temperature should be held at 70°F. for at least 24 hours before testing.

B. Weight Determination

(1) Weigh a replicate of three filled containers and record the weight in grams.

C. Pressure Determination

(1) Check equilibrium pressure in the container. A prepressurized gauge should be used to check the pressure in the container so as to minimize product or pressure loss.

(2) Products that require shaking, as indicated in directions on label, should be shaken as outlined in Section D—Shaking Procedure.

(3) Products that are dispensed without shaking should not be agitated before determining equilibrium pressure.

D. Shaking Procedure

(1) Grasp the can in an upright position and move the arm downward in an arc. As the arm moves downward, the can is inverted. Enough force should be exerted in the motion to throw the product to the top end of the can. The can is then brought back to the starting position.

(2) Repeat this cycle for a total of ten times, pausing for a few seconds between cycles.

E. Dispensing Procedure

(1) The container should be shaken as outlined above and the pressure recorded before each dispensing.

(2) Hold container in proper position for dispensing.

(3) Dispense a weighed amount at 5-minute intervals.

(4) The rate of dispensing should be regulated so that the product would be expelled in a given number of dispensings. (See enclosed Table I for the recommended number of dispensings.)

(5) The weight in grams of the product dispensed should be recorded after each dispensing.

(6) When the first gas discharge is observed, stop dispensing. Weigh the container and record the final equilibrium pressure.

F. Maximum Delivery

(1) Maximum Delivery should also be determined as outlined in the revised Method for Determination of Percent Product Retention; that is:

(a) "Hold can, without dispensing, in dispensing position (inverted for invert style valves, and upright for dip tube valves) for a period of two minutes.

(b) Do not shake can after standing.

(c) Dispense product until gas supply is exhausted.

(d) Weigh container."

G. Tare Weight of Container

(1) Cut open container, wash, dry and record weight in grams to obtain tare weight.

H. Determine Pressure Drop Rate As Follows:

(1) Tabulate weight of product in grams versus equilibrium pressure in container.

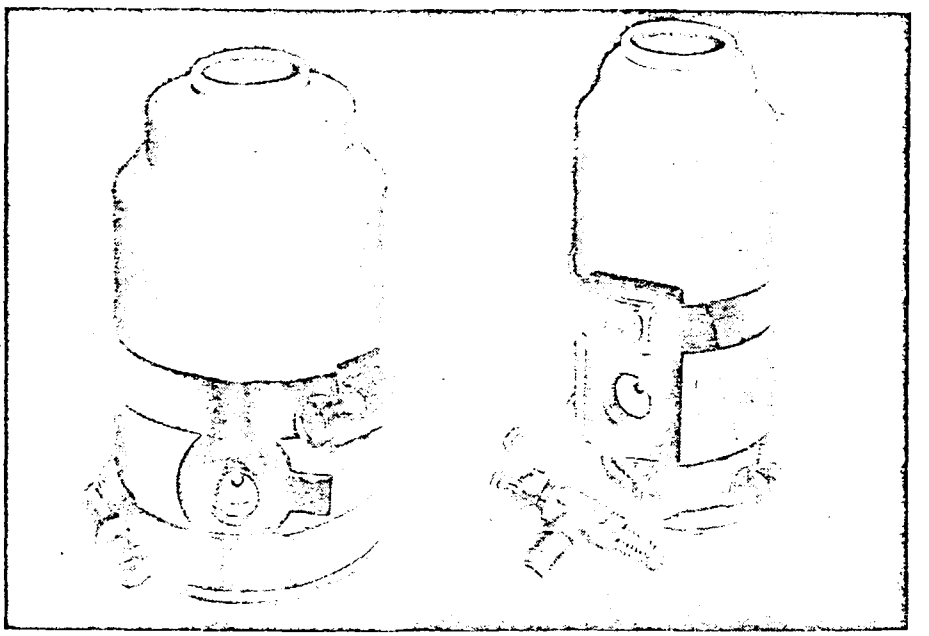
(2) Plot weight of product in grams on the axis of abscissas and the equilibrium pressure on the axis of ordinates to obtain a pressure drop graph.

TABLE I

CONTAINER SIZE	TOTAL CAPACITY (Fluid Oz.)	FILL AVERAGE			RECOMMENDED DISPENSINGS		
		Foam	Stream	Spray	Foam	Stream	Spray
202 x 214	4.9	2	3	3	2	3	3
202 x 314	6.4	3	4	4	3	4	4
202 x 406	7.7	4	5	5	4	5	5
202 x 509	9.8	5	6	6	5	6	6
211 x 413	13.7	6	9	9	6	9	9
211 x 510	15.9	7	10	10	7	10	10
211 x 604	17.8	10	12	12	10	12	12

Adopted Food Aerosol Technical Sub-Committee, December 5, 1960
Approved Aerosol Division Scientific Committee, December 5, 1961
Approved Aerosol Division Executive Board, January 22, 1962

A SIMPLE CAN PUNCTURING DEVICE FOR AEROSOL LABORATORIES



Courtesy of "Freon" Products Division E. I. du Pont de Nemours & Co., Inc.

An inexpensive, simplified aerosol can puncturing device has been produced by our "Freon" Products Laboratory This new device can be made conveniently in two modifications costing about $3.50 and $2.50 respectively, compared to the $15 to $20 cost of the apparatus formerly used. Biggest advantages are the small size, easy assembly, and the ability to position the punctured can in any way most suitable to the job. Either device may be used with any commercially available aerosol can.

To make this new device a piece of 1" x 2" x ¼" steel is drilled and tapped for ¼" NPT straight thread in the center of a 1" x 2" face. Two Aero-Seal M44 hose clamps hold this steel plate to the side of a can, which is punctured by threading a "Fitzall" Can-O-Gas puncturing valve through the plate and into the can. The necessary seal is accomplished by the gasket furnished on the valve. Allowing one dollar for the manufacture of the steel plate, the device costs $3.53, the valve costing $1.45, and the hose clamps $.54 ea.

A less rugged device can be made by bending the prongs on the can clamps furnished with the Fitzall valve to a horizontal plane. The two opposed prongs may be held to a can by two Aero-Seal M32 hose clamps. Puncture is accomplished by threading the valve through the can clamps in the normal manner. The total cost of this unit is only $2.50.

PARTS LIST

"Fitzall" Valve. Part No. 5847, Mfrd. by Virginia Smelting Co., West Norfolk, Va.

Hose Clamps. Aero-Seal all stainless M32 and M44. Mfrd. by Breeze Corp., Inc., Union, N. J.

All parts obtainable at any refrigeration wholesaler.

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METHOD FOR DROP TESTING OF GLASS BOTTLE AEROSOLS

(Prepared by Project Committee on a Standard Drop Test of Glass Aerosol, Scientific Committee, Aerosol Division)

Scope

This method is intended to standardize the drop test procedure used in determining the acceptability of a bottle design in a glass container for packaging a given aerosol formulation.

Outline of Method

A. The arithmetic mean height of a specific glass aerosol bottle design is determined by dropping the bottles, loaded with the given formulation, according to the statistical procedure given below.

B. Dropping an additional group of similarly filled bottles at the arithmetic mean height provides a means of evaluating any potential bursting hazard of the final package, incorporating factors of bottle design, formulation, loading techniques, etc.

Apparatus

1. *Drop Surface*

The drop surface consists of a square steel plate ¼ in. thick and 12 in. on a side. This steel plate is imbedded in a square concrete pad 18 in. on a side and 2 in thick.

2. *Dropping Mechanism*

The test bottles are held above the drop surface by means of a vacuum system. Release of the vacuum allows the bottles to fall freely. The apparatus consists of a rubber stopper with a ½ in. hole connected to any suitable vacuum system, such as a vacuum pump, laboratory line vacuum, or water aspirator. The line connecting the stopper with the vacuum system is provided with a three-way stopcock for release of the vacuum. The rubber stopper is fastened by any suitable means above the dropping surface in such a position that the falling bottle will land as near as possible to the center of the plate.

Several rubber stoppers with ½ in. holes will be needed for the various shapes of bottles that will be tested. A stopper with a flat surface will serve for flat bottles, while stoppers with curved surfaces cut into them will be necessary in order to obtain a tight seal for bottles having other shapes.

Other mechanisms which give equivalent control of the drop positions may be substituted for the vacuum system.

3. *Ovens*

Air ovens are to be maintained at 70°F. and 100°F.

Procedure during Development Stage—Part A

Place the bottles to be tested in room temperature air (70°F. to 100°F.) for a minimum of three hours. Place each bottle in position against the rubber stopper at the terminal of the vacuum system. By opening the vacuum system to atmospheric pressure by means of the three-way stopcock, the vacuum on the bottle is released and the bottle is allowed to fall upon the drop surface. Care must be taken to position and to release the bottle properly so as to prevent twisting and rotation in the air as the bottle falls.

A total of 75 bottles are dropped from one given drop position at room temperature (70°F. to 100°F.). Drop each bottle only once. The drop schedule is based on a statistical design known as the "up and down" method

of sensitivity testing (1). If a bottle breaks* at a given height, the next bottle is dropped from a height one interval lower. If a bottle does not break, the next bottle is dropped from a height of one interval higher.

Drop the first bottle at 6 ft. If the bottle breaks, drop the next bottle at 4 ft. If that bottle breaks, drop the next bottle from 2 ft. If this bottle does not break, drop the next bottle at 4 ft. Again, if that bottle breaks, drop the next bottle at 2 ft. Follow this procedure throughout the testing of the 75 bottles. The interval between adjacent drop levels, d, is 2 ft.; i.e., drop levels of 2 ft., 6 ft., 8 ft., 10 ft., etc. It is required for statistical purposes that this schedule of drop heights be followed exactly. If, because of ceiling limitations, it is impossible to use very high drop heights, it will be permissible to stop at a maximum height of about 14 or 16 ft.

Record for each drop height the number of bottles which break and number of bottles which did not break. Tabulate these data as in Table I. In the example, it is seen that no bottles broke at 2 ft., 2 bottles broke at 4 ft., 11 at 6 ft., 22 at 8 ft., and 2 at 10 ft. If a bottle breaks at 2 ft., it is proper to record the next bottle as a "no break" at zero ft. and to set it aside as if it had been dropped.

TABLE I†

Summary of Drop Data

Height (ft.)	No. of Bottles Which Break	No. of Bottles Which Do Not Break
10	2	0
8	22	2
6	11	23
4	2	11
2	0	2
Total	37	38

In Table I, the total number of breaks (N-37) is one less than the total number of "no breaks". Therefore, the breaks are referred to as the less frequent event. If, however, there had been more breaks than "no breaks," the "no breaks" would be referred to as the less frequent event. The following arrangement of the data and the subsequent equations must be based on the less frequent event.

Record the data for the less frequent event (breaks) as shown in the example of Table II. The values of "i" are 0, 1, 2, 3, etc., where i = 0 is defined as the lowest height at which breaks occured (i.e. 4 ft.) and i = 1, 2, 3, etc., are at increasingly higher levels at which the breaks occurred. The "n_i" values are the frequency or number of breaks at each level of "i". Multiply "i" by "n_i", and obtain the values under the "in_i" column.

TABLE II

Drop Test Statistical Data

i	n_i	in_i
3	2	6
2	22	44
1	11	11
0	2	0
Totals	N = 37	A = 61

Add the "in_i" column to obtain the sum "A".

Using this value for A, calculate the arithmetic mean height, X, by the following formula:

(Formula 1)

$$X = y^1 \pm \left(\frac{A}{N} - \frac{1}{2}\right) d = 4 \pm \left(\frac{61}{37} - \frac{1}{2}\right) 2 =$$

$$4 + (1.65 - .50)_2 = 4 - 2.30 = 6.3 \text{ ft.}$$

where y^1 is the lowest height in feet at which the less frequent event occurred, N is sum of the n_i values in Table II, and d is the interval between drop levels (2 ft.). The plus sign is used when the calculation is based on "no breaks" and the minus sign when the calculation is based on breaks. In the example of Tables I and II, the less frequent event is "breaks"; therefore, a minus sign must be used.

In the initial testing of a new aerosol bottle (formulation, design, etc.), data are first collected to determine the weakest and most vulnerable position of the bottle. Generally, the number of drop positions is determined by the design of the bottle. One position is the bottom of the bottle. The other positions depend on the wall shape. If the bottle is spherical or cylindrical, i.e., symmetrically designed, there is only one drop position on the side. If, however, the bottle is elliptical or some other shape, nonsymmetrical with respect to its longitudinal axis, there are at least two side drop positions—one on the edge and one on the flat side.

*A break is defined as: bottle breaks and is no longer usable. In the case of plastic-coated bottles, it is considered as a break for the stastical definition if the glass breaks, even though the plastic coating remains intact.

† The values in Table I must meet this test: the number of breaks at any given height must be equal to (or more than or one less than) the number of "no breaks" at a height of one interval (d) lower.

Repeat the procedure just described previously for each of the remaining possible drop positions. Calculate X for each position. Select the weakest and most vulnerable position as that position corresponding to the lowest value of the arithmetic mean height (X_A).

It is apparent that there will be cases where an aerosol container may break on being dropped and still may not present a consumer hazard. Throughout this procedure, the potential hazard is assessed in relation to the extent of flying glass or other fragments which are projected from the broken container.

To assess this potential hazard, select at random 200 additional containers and place 100 in the 70°F. oven and 100 in the 100°F. oven for three hours minimum. Drop each container (only once) from a constant drop height which will now be set at a height equal to the arithmetic mean height (X_A) corresponding to the weakest position (or 6 ft., whichever is the lower height). Record the number of nonbreaks, the number of breaks, and the number of the breaks which project flying fragments from the container, as in the example of Table III for coated bottles.

TABLE III

	70°F	100°F
Total bottles dropped from arithmetic mean height X_A ft. (or · ft., whichever is lower)	100	100
Number of nonbreaks	50	50
Number of breaks	50	50
Number of the breaks which project flying fragments	2	4

With data corresponding to those in Table III, the loader, marketer, etc., will be in a position to decide for himself whether the formulation and the container constitute a package that meets his safety standards.

Procedure for Testing during Production—Part B

To insure that a bottle design and formulation which have met the specifications set forth in Part A continue to stay within specifications during production, the bottles as they come off the loading line are drop tested periodically.

The test consists of dropping at only one temperature (either 70°F. or 100°F.) a minimum sample of 100 bottles randomly selected from a given production run (one shift or one day), using the same procedure outlined in Table III above.

FACTORS AND PRINCIPLES USED IN DEVELOPING THE METHOD FOR TESTING GLASS BOTTLE AEROSOLS

The purpose of this section is to review the factors and principles considered in the proposed test method.

A. Apparatus

1. *Drop Surface*

The selection of a ¼-inch steel plate imbedded in a concrete pad was made simply to obtain a good, solid, uniform surface which would give the maximum degree of reproducibility in the testing of a large number of bottles. It was found that concrete, marble, ceramic tiles, etc., chipped and spalled badly due to the impact of the bottles. In addition, there is an old "rule-of-thumb" guide which says that the object on which a test unit is to be dropped should have a mass in the order of 100 times the mass of the test unit. This is done in order that the energy of impact which is dissipated by the "drop surface" will have a minimum of effect on it.

2. *Dropping Mechanism*

The selection of a vacuum system to hold the test bottles in the required position prior to testing was the result of the desire to keep the necessary apparatus as simple and as readily accessible as possible. An additional feature of the system is that it reduces the possibility of giving a torque to the freely falling bottle.

3. *Temperature of Contents*

Temperatures of 70°F. to 100°F. were arbitrarily selected as reasonable temperatures the consumer might encounter with the container. The breakage of glass itself was not found to be affected within this temperature range. For the test purposes, these temperatures should be maintained by use of a constant temperature air oven rather than a hot water bath. The plastic coatings on clear coated bottles have been observed to turn "milky" in appearance after a short time in a water bath due to absorption of water. Presumably this absorption of water could temporarily or permanently alter the physical properties (tensile strength and elastic limit) of the coatings.

B. Test Procedure

1. *Position*

Many factors can influence the structural strength of a glass aerosol bottle. We will not elaborate on the factors, but satisfy ourselves

with the observation that the size, design, and shape of a bottle are very important with respect to its structural strength. It has arbitrarily been decided, therefore, to determine first the most vulnerable position of the bottle, and run subsequent tests in this drop position.

2. *Height*

The greatest distance between the floor and a shelf or cabinet, under ordinary home storage conditions, is taken as 6 ft. Therefore, 6 ft. was chosen as the height to begin the sensitivity testing.

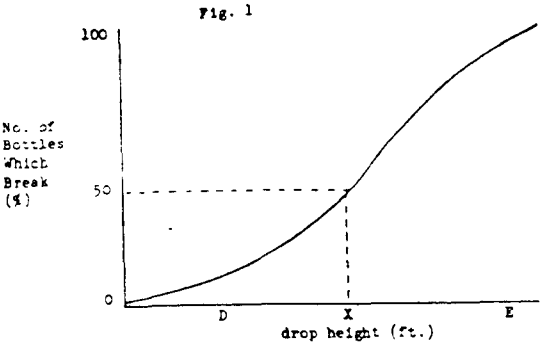
3. *Statistical Design*

The use of a statistical design enables us, through the mathematical laws of probability, to conduct the test with a relatively small number of bottles. The "sensitivity testing" procedure, on which the drop test is based, is commonly used in such fields as evaluating explosives, toxicity testing on animals, etc.

By conducting several thousand drop tests on glass bottle aerosols containers, a curve similar to that indicated below could be obtained for a given design and formulation:

At a low height, D, a small percentage of bottles will break at a high height, E, nearly all the bottles will break. At a height, X, 50% of the bottles will be expected to break. This value depends on an actual curve which has

Fig. 1



been laboriously plotted from a very large number of experimental drops.

However, from Formula 1:

$$X = y^1 + \frac{A}{N} = \frac{1}{2} d,$$

the arithmetic mean height X can be also calculated using only a relatively small number of drop samples. By means of this statistical equation, we have made use of the "S" shaped curve without having to plot it from a large body of experimental data.

C. References

1. Dixon, Wilfred J. and Massey, Frank J., Jr., *Introduction to Statistical Analysis*, Chapter 19, McGraw-Hill Book Company, Inc. (1951)

2. Eisenhart, Churchill, et al., *Selected Techniques of Statistical Analysis*, page 341. McGraw-Hill Book Company, Inc. (1947)

Adopted by the Project Committee on Standard Drop Test, May 17, 1958
Approved Aerosol Division Scientific Committee, May 20, 1958
Approved Aerosol Division Administrative Committee, September 8, 1958

PROCEDURE FOR MOISTURE DETERMINATION IN AEROSOL CONTAINERS

Moisture is determined in an aerosol container by back-titrating the sample, to which has been added Karl Fischer reagent, with a methanol-water solution. A ratio between the methanol and Karl Fischer is obtained and the Karl Fischer reagent is standardized against sodium tartrate dihydrate.

(1) Determination of ratio of Karl Fischer Reagent to Methanol-water solution.

Place approximately 60 ml. of anhydrous methanol in a closed glass container and add an excess (2 to 3 ml.) of Karl Fischer reagent. Keep agitating with a magnetic stirrer for about five minutes. Back titrate with methanol-water solution until the end point is reached. Then add an accurately measured amount (10 ml.) of Karl Fischer reagent and back titrate with the methanol-water solution until the end point is reached. Calculate the ratio X as follows, where

$$X = \frac{\text{ml. of Karl Fischer reagent (A)}}{\text{ml. of methanol-water solution (B)}}$$

A ratio close to 1 is recommended. (The methanol solution is Baker's Analyzed Anhydrous Methanol, approximately .05% water.)

(2) Standardization of Karl Fischer Reagent.

Place about 60 ml. of the anhydrous methanol in a closed glass container and add an excess (about 2 to 3 ml.) of Karl Fischer reagent. Keep agitating for five minutes with a magnetic stirrer. Back titrate with the methanol solution until the end point is reached. Add an accurately weighed amount (about .32 g.) of sodium tartrate, $Na_2C_4O_4 \cdot 2H_2O$, certified reagent, and add an excess (about 6 ml.) of Karl Fischer reagent. Keep agitating until the sodium tartrate is completely dissolved. Back titrate with the methanol-water until the end point is reached.

Calculate ml. of Karl Fischer reagent used to combine with the water in the sodium tartrate standard.

$$C = A - BX$$

where C is ml. of Karl Fischer used to combine with the water in the sodium tartrate standard.

A is ml. of Karl Fischer (as above)

B is ml. of methanol-water (as above)

$$y = \frac{W}{C}$$

where y is mg. of H_2O for 1 ml. of Karl Fischer; W is total mgs. of H_2O in sodium tartrate

(3) Titration of Unknown Sample.

Place about 60 ml. of the anhydrous methanol in a closed glass container and add an excess (about 2 to 3 ml.) of Karl Fischer. Keep agitating with a magnetic mixer for five minutes. Back titrate until the end point is reached. Add a weighed amount of the unknown and an excess of Karl Fischer reagent. Keep agitating for five minutes and back titrate with the methanol solution until the end point is reached.

$$\text{Water Content (weight per cent)} = \frac{A \times y \times 100}{\text{g. of unknown}}$$

Adopted Personal Products Standard Methods Sub-Committee, December, 1959
Approved Aerosol Division Scientific Committee, December, 1959
Approved Aerosol Division Executive Board, December, 1959

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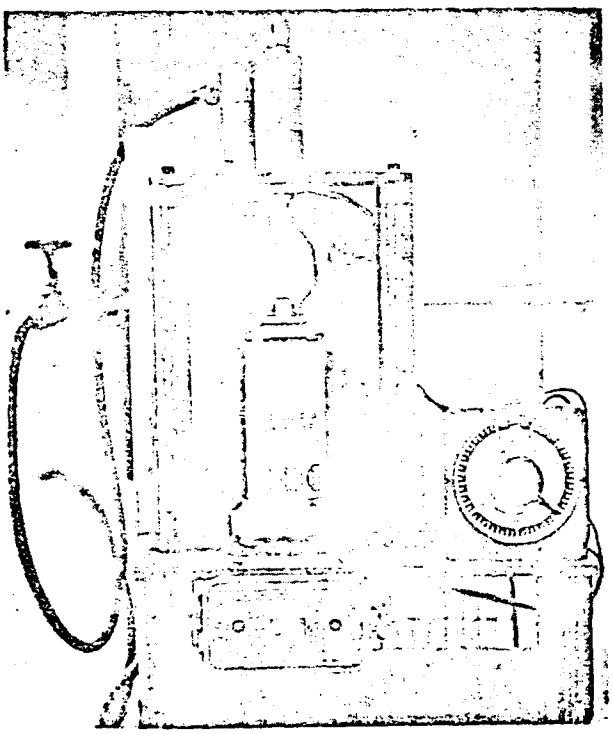
METHOD FOR AEROSOL VALVE DISCHARGE RATE

1. Weigh sample container and bring to required standard temperature.
2. Place sample into test stand, adjusting for proper height and setting required spray time.
3. Activate tester by pushing switch.
4. After sample has been sprayed automatically through set time period, remove and reweigh.
5. Change in weight is discharge per time set for spray.



Adopted Personal Products Standard Methods Sub-Committee, December, 1959
Approved Aerosol Division, Scientific Committee 1961
Approved Aerosol Division, Executive Board 1962

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PRODUCT:

Batch No.

Valve:
Button:
Total Delivery Rate:
Material Deposited:
Propellent Mixture:
Pressure at 70°F.:

Remarks:

Date:
Tested for:

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METHOD FOR SPRAY PATTERNS

Although several methods have been described in the literature for measuring particle size of aerosols, these methods are tedious, time-consuming, require elaborate equipment, or are difficult to carry out. The method herein described is a simple and quick technique for obtaining qualitative comparison of spray patterns. Furthermore, the equipment required can be put together for about $150.00.

The method is based on the impingement of the spray on a piece of paper that has been treated with a dye-talc mixture. A 5% mixture of the dye in talc is brushed onto a 60-pound Vellum paper stock. A small, but known, burst of spray is allowed to impinge upon the treated paper. Wherever the particles strike the paper, the dye goes into solution and is absorbed. The size of the dye spots are in direct relation to the size of the liquid particles when they come into contact with the paper. The dye used must be soluble in the spray particles. In the case of alcoholic sprays such as hair lacquers, a water-soluble dye such as DuPont Crystal Violet must be used; in the case of oil soluble sprays such as insecticides, an oil soluble dye, for example, DuPont Oil Red Powder, must be used.

Attached is a sample of the paper used for obtaining the spray pattern. Also included is a photograph of the equipment proposed by the Freon Products Laboratories for obtaining a given burst of the spray on the paper.

The apparatus is made up from the following:

1. General Electric A-C Motor Type SKH, Frame 48, 1/6 HP, 1725 RPM, 60 cycle, 115 volts, or equivalent.
2. Variable speed "Zero Max" torque converter, Model 142X, Revco Inc., Minneapolis, Minnesota.
3. One-inch diameter by 4" long connecting rod from torque converter to disc.
4. Rotating Disc made from 1/16" thickness aluminum or stainless steel, 5¾" radius; cut-out radial sector, 14 cm. at circumference; 15-cm. sliding shutter.
5. Mounting Board.
6. Slot on disc to accept 5" x 5" piece of paper.

In order to know the exact amount of material which is sprayed from the container, it is necessary to know the rate of rotation of the disc, the dimensions of the cut-out sector, and the delivery rate of the valve. The sector must be pie shaped, i.e., the edges must follow the radii of the disc. Using the equation below, it is then possible to calculate exactly how many grams are sprayed through the disc.

$$\frac{SD}{2\pi rR} = \text{Grams}$$

S — Slit width at circumference in cm.

D — Delivery rate of valve in grams per second.

R — Rate of revolution of disc in revolutions per second.

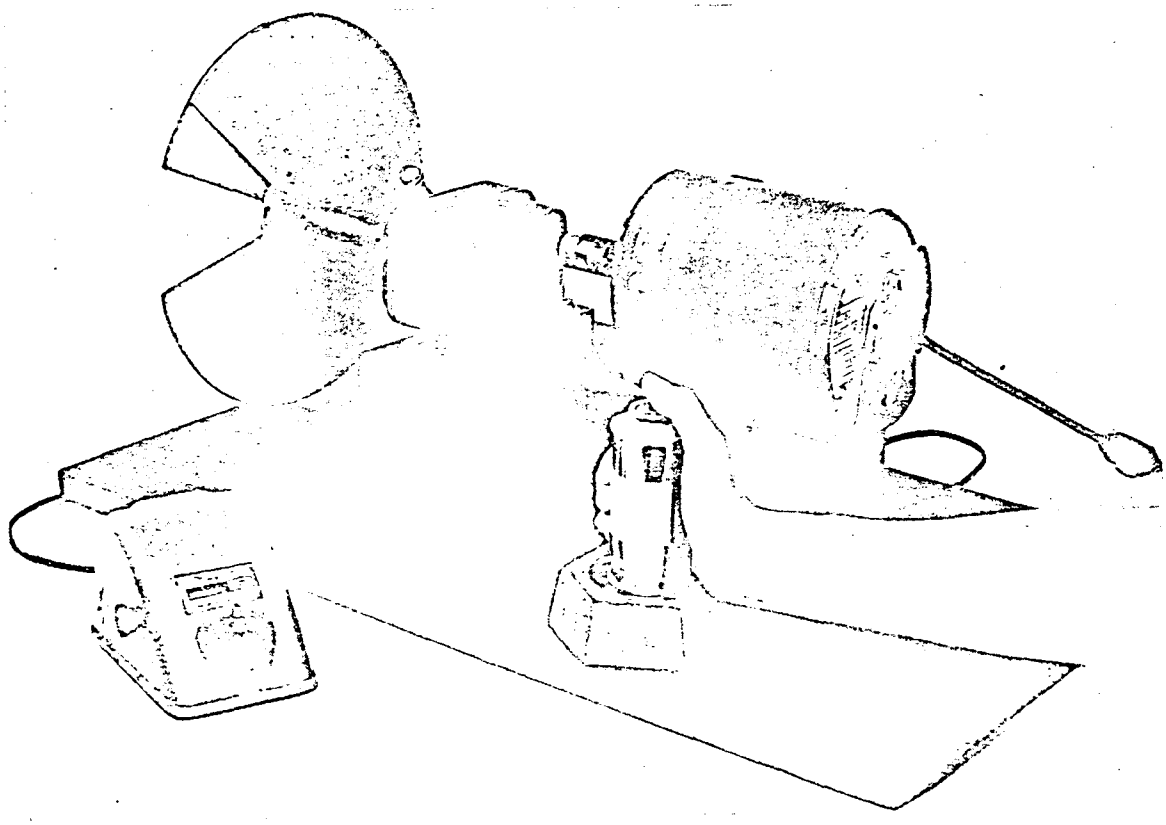
r — radius of disc in centimeters (14.5 cm.)

The disc must revolve slowly enough so that it is possible to depress and release the aerosol valve before the lot in the disc has made a second revolution in front of the spray. A small slit width and high rate of revolution permit only a small amount of spray to pass, while a large slit width and slow rate of revolution permit a large amount of spray to pass.

Patterns made from aerosols containing only fast drying solvents can be kept indefinitely and serve as a permanent record, whereas patterns made from aerosols containing oils or solvents which dry slowly change with time and must be photographed for a permanent record. The slow or non-drying oil with dissolved dye gradually spreads in the paper, thereby distorting the pattern obtained.

This spray pattern technique is particularly useful for comparing spray characteristics obtained with different valves, actuators and formulations. The method is rapid, easily done, and can be visually evaluated.

Adopted Personal Products Standard Methods Sub-Committee, May, 1959
Approved Aerosol Division, Scientific Committee 1961
Approved Aerosol Division, Executive Board 1962



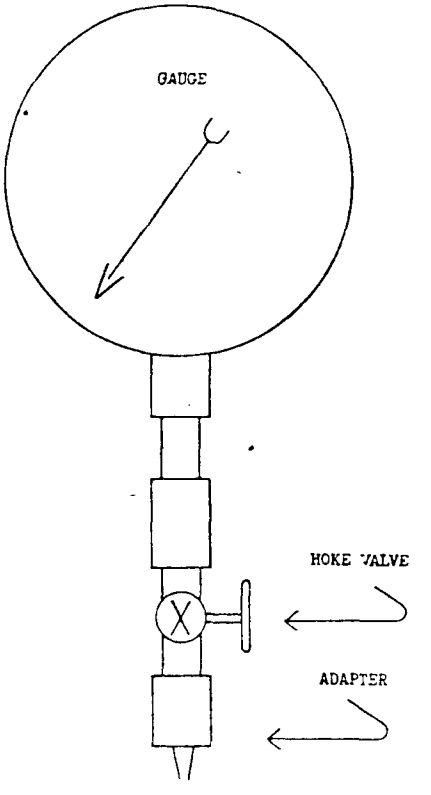
RAPID METHOD FOR PRESSURE DETERMINATION

The following method is suggested as a rapid technique for quality control, formula development and etc. where speed is necessary and a high degree of accuracy is not essential. For more accurate measurements, the reader is referred to the "Tentative Method for Internal Pressure Determination of Aerosol Products in Light Weight Containers", adopted December, 1956, by the Aerosol Scientific Committee and subsequently by the Aerosol Division Administrative Committee of CSMA.

Apparatus required:

1. Pressure gauge—range 0 to 160, stainless steel construction, with 1≟ graduations preferred. This gauge should be attached to ⅛" Hoke needle valve with all connections leak proof. To needle valve, attach a suitable adaptor* to fit the aerosol valve(s) on the containers to be analysed (see diagram).
2. Standard for above gauges. It is recommended that a suitable standard check accurate to plus or minus 0.5 psi be set up in the laboratory for quick calibrations of test gauges. Gauges in constant use should then be checked about once each week or at any time after a gauge has been subjected to accidental shock such as dropping, etc.
3. Constant temperature water bath accurate to at least plus or minus 1°F.
4. Prepressurizing gas supply (compressed air, N_2, CO_2, etc. satisfactory).

*Adaptors may be obtained from Modern Machine Shop, 123 N. Hazel St., Danville, Illinois. These were designed for use in measuring pressure of glass aerosols (see Ttentative Method for Determining Internal Pressure of Glass Aerosols).



Procedure:

Measurements should be made in groups of at least three and the average reading taken. Immerse cans in constant temperature water bath. Immersion time required depends on initial can temperature; if it is within roughly 10°F. of bath temperature, 15 minutes (agitated every 5 minutes) will suffice. For water based products having higher specific heat, large cans (over 12 ounces) or cans varying widely in temperature, at least 45 minutes (preferably 60 minutes) is required. Prepressurize gauge as nearly as possible to anticipated container pressure, agitate cans.

attach to valve on container, press open, and open Hoke valve. Record reading, close Hoke valve, remove and repeat with second and third cans. Record exact bath temperature.

Discussion:

Care should be taken to avoid handling containers with bare hands, etc. after they have reached constant temperature, since pressure is a function of container temperature. Care should be also taken to be certain there are no leaks in guage apparatus. This can be checked by occasionally making a measurement with attachment under water. "Bleeding" or removal of gas from vapor phase before checking pressure is not permissible if the true pressure in the container is desired.

For non food products the gauge apparatus may be cleaned between product uses by forcing suitable solvents into the gauge then venting. This should be repeated several times until gauge is free of contamination. When changing from chlorinated solvents to water base products and visa versa the above is particularly important to avoid possible contamination.

A separate device should be used for food products only. Suitable prepressurizing gasses for food products should be limited to any of the approved and commonly employed food propellant gasses such as nitrogen, carbon dioxide or nitrous oxide that are not liquefiable at the temperatures and pressures used. Compressed air is not recommended because of its oxygen content.

Care should be taken to daily clean gauges by repeated injection and venting of an approved cleaning solution containing a bacteriastat. After cleaning, the gauge and apparatus should be rinsed with clean water and purged with the prepressurizing gas prior to use.

Approved Sub-Committee on Pressure Determination, March, 1960
Approved Aerosol Division Scientific Committee, May 17, 1960
Approved Aerosol Division Executive Board, January 17, 1960

PRE-MARKETING CHECKLIST FOR FOOD AEROSOLS

The following list of factors relative to the various phases of packaging pressurized foods has been compiled by the Food Aerosol Liaison Committee of the Aerosol Division, Chemical Specialties Manufacturers Association.

I. FORMULATION

A. Product Characteristic

1. Viscosity
2. Particle size and resultant limitations.
3. Specific gravity
4. Product pH
5. Product composition
   a. Moisture
   b. Sugar content
   c. Salt content
6. Color requirements
7. Flavor requirements

B. Effect of Temperature on Non-Volatile product

1. Flow characteristics of product at the following temperatures:
   a. 40°F.
   b. 70°F.
   c. 130°F.
   d. 212°F.
2. Effect of Temperature on Product Stability
   a. 70°F.
   b. 100°F.
   c. 130°F.
   d. 180°F.
3. Effect of Temperature on Product Quality—Carmelization, etc.

C. Compatibility of Product with Compressed Propellent

1. Critical solubility range
2. Degree of agitation required in terms of length of shaking cycle
3. Effect of compressed propellants on product characteristics and quality
4. Equilibrium pressure characteristics related to:
   a. Product Fill versus Headspace
   b. Temperature (40°F—200°F.)
5. Effectiveness of Product for the purpose intended, as determined by commercial techniques.
6. Determination of weight loss in grams of gas, or in pounds per square inch gauge

D. Compatibility of Product with Liquified Gas Propellent*

1. Critical solubility range
2. Emulsificability characteristics
3. Weight ratio of propellant required for desired form of dispensing
4. Pressure characteristics as the following temperatures:
   a. 40°F.
   b. 70°F.
   c. 130°F.
   d. 200°F.
5. I.C.C. Regulations
6. Effectiveness of Product for the purpose intended as determined by commercial techniques

E. Standards of Identity for the Product

*Section D added in assumption that liquified gas propellents will receive clearance for food use, either individually or in combination with other gases

II. VALVE REQUIREMENTS

A. Valve

1. Performance of valve with formulated product
   a. Dispensing characteristics as related to valve structures and type
      aa. Spray
      bb. Foam
      cc. Ribbon
      dd. Stream (non-aerated)
   b. Body orifice sizes as related to the following factors:
      aa. Delivery rate
      bb. Particle size of product
      cc. Type of dispensing characteristics desired
      dd. Dropping rate
   c. Valve action
   d. Product retention
   e. Degree overrun (where applicable)
   f. Dip tube length and ability to anchor in container
   g. Means to prevent cavitation (where applicable)
   h. Marking of valve cup by manufacturer to insure proper alignment of actuator with position of tube to insure adequate removal of product
   i. Effect of crystallization of sugars on valve performance
   j. Effect of temperature upon valve components and gasketing material

B. Actuator

1. Particle size limitations
2. Orifice size as related to particle size and type of dispension characteristics desired
3. Configuration and grouping of mechanical spinners or other means to insure breakup of spray-type product
4. Ability of actuator to perform required function
   a. Achievement of desired spray pattern and velocity in terms of:
      aa. Particle size
      bb. Spray pattern width at predetermined distances
      cc. Coverage of area to which product applied
   b. Achievement of desired application of streams (non-aerated), ribbon and foam type valves to perform required function in terms of:
      aa. Uninterrupted delivery
      bb. Non-clogging
      cc. Non-drip
      dd. Low percent product retention
      ee. Effect of possible crystallization of sugars
      ff. Ease of application
   c. General Factors
      aa. Ease of attachment of actuator to valve stem
      bb. Ease of removal for cleaning by housewife and ease of reinstallment after cleaning
      cc. Adaptability of actuator for use with other products

III. CONTAINER REQUIREMENTS

A. Effect of formulation on Container Components

Test pack determinations for obtaining storage data at various predetermined temperatures and storage times at predetermined positions. Test packs should be conducted for all products prior to test marketing so as to determine container and product shelf life, including the following factors:

1. Effect on internal enamel systems and or plain tin plate
2. Effect on side seam structures at higher storage temperatures
3. Effect of product on a drawn dome and seamed areas of dome
4. Effect of product on external enamel systems and lithography

B. Effect of formulation on valve and actuator components

1. Degree of corrosion on valve cup (internal and external) and protection required
2. Effect on valve with intermittent application
3. Effect on actuator with intermittent application
4. Effect of storage period on delivery rate
5. Determination of gas loss in terms of weight (grams) during storage period on pre-weighed containers
6. Effect of storage on spray pattern

C. Effect of container and storage temperatures and time on product formulation

1. Changes in pressure characteristics of product plus compressed propellant
2. Alterations in product color
3. Alterations in products pH
4. Alterations in product flavor, pick up of objectionable tin off-flavors, and other deleterious changes
5. Effect on product overrun in foam products
6. Effect on product stability and viscosity
7. Precipitation of solids during storage
8. Possible crystallization of sugars in stream type actuators
9. Product retention

D. Possible Toxicity Considerations of valve or container components

1. Feasibility of use of release agents or lubrication systems

IV. BACTERIOLOGICAL REQUIREMENTS VERSUS PRODUCT FORMULATION

A. Factors of Product Composition related to bacteriological stability of product

1. pH
2. Moisture
3. Salt content
4. Sugar content
5. Total solids

B. Microbiological Flora normally associated with product

1. Thermal death time characteristics

C. Method of preservation required to insure against bacterial contamination, assuming a normal bacterial load

1. Fill temperature
2. Storage temperatures at which the product will be handled and merchandised
3. Length or terms of process required to insure adequate bacteriological shelf-life

D. Method and position of gassing operation in terms of possible recontamination of heat-treated product

1. Gassing of product prior to cooling operation
   a. Pressure-temperature characteristics of propellant
2. Gassing of product cooled to, or filled at, room temperature
   a. Sterilization of gassing heads and valve stems prior to gassing operation

E. Use of asceptic filling techniques

V. COMPLETED PRODUCTS USE TEST

A. Effectiveness of product application, based on comparison of pressurized product with a related product currently on the market either in the pressurized form or in a conventional package

B. Development of instructions and label declarations (directions and mandatory precautions)

C. Development of carton according to specifications of I.C.C. Regulations

D. Development of overcap to give protection to product on shelf

E. Ease of operation

F. Absence of spillage and/or drip from actuator

G. Possible adverse effect on materials and implements in the kitchen

VI. REGULATORY CONSIDERATIONS

A. Food and Drug Administration—Federal Food, Drug and Cosmetic Act

B. Interstate Commerce Commission (Freight Tariff No. 11)

C. Federal Trade Commission

D. Department of Agriculture

E. Post Office Department

F. Chemical Specialties Manufacturing Association

G. State and local regulatory agencies

Adopted Food Aerosol Liaison Committee, December 8, 1958
Approved Aerosol Division Scientific Committee, December 8, 1958
Approved Aerosol Division, Administrative Committee, December 8, 1958

METHOD FOR DETERMINING OVERRUN OF FOOD AEROSOLS SUCH AS WHIPPED CREAM

The term overrun is that common to the ice cream industry indicating the relative volume of a product before and after aeration. For food aerosols as whipped cream, it indicates the relation between the liquid volume of the cream industry indicating the relative volume product. This relationship can be expressed in the following formula:

$$\frac{\text{Volume Dispensed Cream-Volume Liquid Cream Mix}}{\text{Volume Liquid Cream Mix}} \times 100 = \%\ \text{overrun}$$

The volume and density of the liquid mix, that is the cream plus flavoring, sugar, etc., is determined previous to filling into the aerosol container. The filled container is closed, gassed, shaken and stored at 40° F. for at least 24 hours before testing. Some creams or toppings may require longer storage to reach equilibrium pressures and full overrun.

Dispense at 40° F. into a measuring cup with a wide opening and straight sides which permits filling with a minimum of air pockets. The cream should be dispensed in a manner to avoid air pockets.

Before dispensing, shake vigorously by hand for 5 seconds. Holding in the proper position, dispense about 6 fluid ounces of whipped product, shake again and dispense 6 more ounces. Repeat until contents have been dispensed.

To correct for product retained in the container, weigh the contents remaining after complete dispensing, convert to volume, and subtract from the original liquid volume before inserting in the formula above.

An alternate method provides determination of overrun on a weight basis of a portion of the product. The formula used here follows:

$$\frac{\text{Wt. of Liquid Cream Mix-Wt. of Same Volume of Dispensed Cream}}{\text{Weight of Same Volume of Dispensed Cream}} \times 100 = \%\ \text{overrun}$$

In this method, the preparation is the same as the first above. A 4 oz. drinking cup is filled with liquid cream and weighed. A similar cup is then filled from the shaken aerated container and weighed. Apply formula.

This latter method permits comparison of overrun on various portions of dispensed product. The overrun tends to decrease as various portions are dispensed. Thus a whipped cream which dispensed 500 to 550% on the first portion may average about 400% for all portions.

Results should be expressed to the nearest 10%.

Adopted Food Aerosol Technical Sub-Committee, December 5, 1960
Approved Aerosol Division Scientific Committee, December 5, 1961
Approved Aerosol Division Executive Board, January 22, 1962

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METHOD FOR THE DETERMINATION OF PER CENT PRODUCT RETENTION IN PRESSURIZED FOODS

1. Storage conditions.

Products normally refrigerated should be held at 40°F. for at least 24 hours before testing.

Products normally stored at room temperature should be held at 70°F. for at least 24 hours before testing.

2. Weigh filled container to nearest gram or .1 ounce.

3. Check pressure in container. Approximate pressure measurement with a Bourdon type gauge is satisfactory. Invert the container for pressure measurement of containers with dip tube valves. For more accurate determination of pressure, refer to CSMA standard method.

4. Dispensing procedure.

Follow directions for use of container, shaking if specified. It is suggested that shaking be standardized as follows: Grasp the can in an upright position and move the arm downward in an arc. As the arm moves downward, the can is inverted. Enough force should be exerted in this motion to throw product to the top end of the can. The can is then brought back to starting position. Repeat this cycle for a total of ten times, pausing for a few seconds between cycles.

Hold container in proper position for dispensing. Dispense a weighed amount at five-minute intervals. The rate of discharge should be regulated so as to approximate a rate encountered in actual usage. The container is shaken before each dispensing (except for last dispensing when maximum delivery is being determined).

The amount dispensed at each interval should be such that the net weight of the product would be expelled in six dispensing. One-half hour is, therefore, required for total dispensing.

5. Delivery to first gas discharge.

When first gas discharge is observed, stop dispensing and weigh container.

6. Maximum delivery.

Hold can (without dispensing) in dispensing position (inverted for invert style valves and upright for dip tube valves) for a period of two minutes. Do not shake can after standing. Dispense product until gas supply is exhausted. Weigh container.

7. Cut open container, wash and dry. Weigh all component parts to obtain tare weight.

8. Calculate per cent retention for delivery to first gas discharge and for maximum delivery.

Adopted Food Aerosol Technical Sub-Committee, December 5, 1960
Approved Aerosol Division Scientific Committee, December 5, 1961
Approved Aerosol Division Executive Board, January 22, 1962

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CHECKING PACKAGED COMMODITIES

A MANUAL FOR WEIGHTS AND MEASURES OFFICIALS

National Bureau of Standards
Handbook 67 — Issued March 20, 1959

May be purchased
Superintendent of Documents
U. S. Government Printing Office
Washington 25, D.C.

Price 35 cents

EXCERPTS

CHECKING PREPACKAGED COMMODITIES

Malcolm W. Jensen

A manual for State and local weights and measures officials, describing a method for controlling various types of prepackaged commodities.

1. INTRODUCTION

There is presented here a method of control of prepackaged commodities (commodities put up in packages in advance of being offered for sale) for use by State and local weights and measures officials—a method based on two concepts:

(1) Variations in quantities of packages are not permitted to such extent that the averages of the quantities in the packages comprising a lot, shipment, or delivery is below the quantity stated, and an unreasonable shortage in any individual package is not acceptable, even though overages in other packages in the same lot, shipment, or delivery compensate for such shortages. (This is the basic quantity requirement of the Model Regulation for Prepackaged Commodities adopted by the National Conference on Weights and Measures and of the Federal Food and Drug Administration.)

(2) Perfection in either mechanical devices or human beings has not yet been attained; thus the existence of imperfection *must* be recognized and allowances for such imperfection must be made. These allowances are recognized in the "average" concept.

5. POSITION FOR PACKAGE-CHECKING OPERATION

After the announcement of his presence, the official should select a suitable position for his package-checking operations. The principal requirement of the site is convenience—both to the inspector and to the store personnel and customers. If one that is in the customer area of the store yet out of the way of normal customer traffic can be found, it will be quite proper to perform the tasks in the view of the public. This tends to inform the casual onlookers as to one important phase of the weights and measures program. Such activity also will represent good public relations for the store, if the packages being checked are found to be accurately labeled.

6. SAMPLE SELECTION

(The word "sample" will be used herin to designate the small group of packages, usually 10, selected to represent a lot, shipment, or delivery. In a storage area such as is found on the premises of a manufacturer, packer, distributor, and in some cases a retailer, the total inventory of a single item of merchandise in a

single size may be found to contain 2 or more lots, each identifiable by a lot symbol. In these instances it is advisable to sample one or more of the individual lots and take action on such individual lot, independent of other lots of the same type package.)

With the location selected, it is advisable to decide, at least tentatively, the lots of prepackaged items that are to be checked (for example, hamburger, chuck roasts, pork chops, calf liver, sliced American cheese, Swiss cheese, cereal, canned beans, salt, and the like) and select the samples from those lots. There are two important considerations in the selection of samples. First, the sample should be of sufficient number to represent properly the lot from which it is taken, yet not so many as to require for a single lot a disproportionate amount of checking time; and second, the samples should be selected from various places in the lot—top, bottom, center, right, left, front, rear—again so that the lot is properly represented. Under normal conditions a sample of 10 will be adequate. A larger sample does not increase the reliability of the sample in an amount proportional to the increase. (An exception in the nature of a larger sample for very large lots is explained in Step 5 of the Checking Procedure, page 9.)

If practicable, all samples should be selected before the weighing of any is begun. This provides for checking the counter "as found," and avoids any possibility of packages being added to or removed from a lot that is to be checked during the time another lot is being checked, and thus disturbing the as-found condition.

7. SCALE TEST

Once the samples have been selected, the scale to be used in the checking procedure is made ready. If the packages are of such size that the equal-arm scale is to be used, the scale must be placed on a firm support and should be leveled. The scale, itself, should be tested in the new environment. (A simple test is appropriate, such as a careful observation of zero-load indication, one or two equal loads on each pan, one small load to test the tower indicator and side bar, and a test for sensitiveness.) A test not only will assure the inspector that his device is operating properly; it will also convince any observers as to the care exercised by the weights and measures official in the conduct of his duties.

If the packages are large, the store scale that is to be used in the checking should be selected, both as to its physical condition and as to its convenience from the standpoint of the store personnel, and should be examined as to its appropriateness for the checking procedure. Such a scale obviously should be a "sealed" scale. It should be checked carefully for sensitiveness and should be used only if it is sufficiently sensitive to indicate clearly weight in the amount that errors are to be defined. Once the scale has been selected, it should not be released to commercial service until the inspector's use of it has been completed.

8. CHECKING PROCEDURE

8.1 Random Packages

(See also Section 9).—The checking procedure is designed to determine whether the average quantity of contents of the packages in a lot is at least equal to the average declared quantity, and also whether there exist any "unreasonably" large errors in the package labeling. This procedure develops such information through the determination of errors in individual packages. The step-by-step procedure for checking random packages follows:

Step 1. Checkweighing

(a) Equal-Arm Scale. Weigh each package of the sample representing a single lot by placing on one pan of the scale the package and on the other pan the tare, as represented by similar packaging material (essentially uniform packaging materials having been used for similar packages), and standard weights equal to the declared weight. Read the error as shown on the tower indicator, or tower indicator plus side bar graduations if the error is greater than the tower capacity, to the nearest 1/16 ounce.

(b) "Substitution" Method. First "balance in" on the load-receiving element of the scale to be used, standard weights in small denominations sufficient in total weight to equal the largest plus error that might be expected in the packages to be weighed. Determine carefully the weight of the packaging material, and then place on the load-receiving element of the scale standard weights in an amount equal to the tare weight plus the labeled weight. Note the exact indication of the scale (either automatically indicated or indicated by poise placement

—with or without counterpoise weights—as the case may be). Remove these standard weights from the load-receiving element and place thereon a package to be weighed. Restore precisely the previously noted scale indication by adding or removing standard weights. The weights thus added or removed indicate the package error—*short* (minus) if weights are *added*, *over* (plus) if weights are *removed*.

Because some shortages in package weight are caused by the leaking of fluids from the commodity, and because certain packages are sufficiently watertight that they will hold such leaked fluid, it will be advisable to make special observation in certain instances. If a package containing a commodity suspected of leaking is transparent, and if any tray, cup, or other absorbent packaging material apparently has not absorbed any moisture, the package may be turned upside down so that any fluid will run to the transparent top and be easily seen. If fluid is apparent inside the package, or if the packaging material appears to be or to have been wet and soggy, the package should be opened and the net weight determined directly.

Step 2. Recording

(See also Section 11).—Record the labeled weight and the error in 1/16 ounce for each small package, or in an appropriate denomination for each large package. The zero errors (recorded as 0) and the plus errors are listed in one column, the minus errors in a second column. (See example, Step 5.)

Step 3. Unreasonable errors

Circle errors that are "unreasonably" large, either plus or minus. The decision as to the unreasonableness of an error, though of necessity arbitrary, must be made and may be predicated, to a certain extent, on knowledge. Consideration should be given to (1) the allowable error in the commercial device employed in the packaging process, (2) the possible error in the scale used to check the packages, (3) anticipated reasonable human errors in both operations, and (4) the susceptibility of the packaged commodity to accurate weight control at the time of packaging. The table that follows is suggested for both random and standard-pack packages that contain items of such a nature that they are susceptible of precise weight control. Standard-pack packages of such commodities as apples, potatoes, and the like cannot be controlled as precisely as can packages of commodities such as peas, corn, sugar, salt, and flour; consequently the inspector must exercise greater liberality in the determination of the reasonableness or unreasonableness of errors in packages containing large individual elements.

(It will be noted that the suggested plus allowances are twice the suggested minus allowances at each "labeled quantity." This is an acknowledgment that packers must be allowed to overfill such packages as are susceptible of moisture loss.)

UNREASONABLE MINUS OR PLUS ERRORS

Labeled Quantity	*Minus Error Greater Than*	*Plus Error Greater Than*
0 to 2 ounces	1/8 ounce	1/4 ounce
2– to 8 ounces	3/16 ounce	3/8 ounce
8 ounces– to 2 pounds	1/4 ounce	1/2 ounce
2+ to 4 pounds	5/16 ounce	5/8 ounce
4+ to 7 pounds	3/8 ounce	3/4 ounce
7+ to 14 pounds	1/2 ounce	1 ounce
14+ to 24 pounds	3/4 ounce	1½ ounces
24+ to 36 pounds	1 ounce	2 ounces
36+ to 51 pounds	8 ounces	1 pound
51+ to 101 pounds	2 pounds	4 pounds

The figures offered above are suggested for the determination of the "reasonableness" of errors in individual packages; they should not be used as tolerance figures.

Step 4. Action based on unreasonable errors

Action should be taken with respect to the packages with unreasonable errors (either + or —); the following is suggested:

(a) If one package of the sample of 10 packages has an unreasonablly large *minus* error, that package may be ordered repacked or relabeled, or may be held to constitute a violation of the statute and taken as evidence, at the discretion of the inspector.

(b) If there are in the sample of 10 packages 2 or more packages with unreasonably large *minus* errors, the *entire lot* should be held in violation, *without further calculation*. Appropriate action with respect to ordering off sale, prosecution, or the like should be taken. (See 10. Official Action.)

(c) If 3 or less of the sample of 10 packages have unreasonably large *plus* errors, these should be called to the attention of the market operator or the person responsible.

(d) If there are in the sample of 10 packages 4 or more packages with unreasonably large

plus errors, this should be considered to show poor packaging practice, without further calculation. This situation should be called to the attention of the store operator, who should be instructed as to more precise weighing.

Step 5. Determination of average error

Average errors should be determined for those lots on which conclusions have not been reached under (b) and (d) in Step 4 above. The average error is determined as follows:

(a) As in the example below, add the plus (+) errors, on the one hand, and the minus (—) errors, on the other hand—excluding from the sums the circled figures which represent unreasonably large errors. (The unreasonably large errors, both plus and minus, are excluded from the average, because they are acted upon individually and because their inclusion could destroy or alter the packaging "pattern." For example, a sample could show 9 packages each with a minus error of 1/16 ounce and one package with a plus error of 8/16 ounce. If the large plus error is included, the average error is zero. Actually the "pattern" is minus 1/16 ounce per package, and this is evident when the "unreasonably" large plus error is excluded from the average.)

EXAMPLE

Error in 1/16 oz

—	0, +
3	0
1	2
(8)	0
	2
4	1
	0
	1
	6

(b) Calculate the average error by (1) subtracting the smaller sum (plus errors or minus errors) from the larger sum, (2) giving the result the sign (+ or —) of the larger sum (in the example above: $-6 - 4 = +2$), and (3) dividing the result by the number of items not circled (or the total number of items, including the zeros, included in the sums). Thus, in the example, *average error* = +2/9.

This figure is the number of 16ths ounce that the "average" package of the sample (representing the lot being checked) deviates from zero error, and the sign indicates whether this average error is plus (overweight) or minus (short weight). This "average" is, of course, exclusive of those packages having unreasonably large errors.

Under many circumstances the inspector will be in a position at this point to declare whether or not the lot under examination conforms to the requirements of the law. In certain instances when a very large lot—say 200 packages or more—is being checked, a further step is advisable. If the average error found in the sample of 10 representing the very large lot is plus, zero, or significantly minus, a decision on the lot is quite proper. If, however, the average error in the sample of 10 representing a very large lot is just barely minus, the inspector will want to convince himself that his small sample is truly representative. In this case 40 more packages should be selected *at random* from the *same* lot. These 40 packages should be weighed individually, the "unreasonably" large errors, plus and minus, circled and eliminated, and an average error calculted for the sample of 50 (the original 10 and the additional 40). Action should be taken on the lot according to the average error on the sample of 50, regardless of the magnitude of such average error.

Although the calculation designated (b) above is not necessary to establish the primary fact that the average net quantity of contents is or is not below the label quantity, this having been established at the conclusion of the computation designated (a), it is well for the inspector to complete the calculation of the average error in order that his report to his superior may be complete, in order that he may properly inform the packer as to the reason for any official action, and so that any record taken to court may be almost self-explanatory. (For action, if average quantity of contents is less than the declared quantity, see 10. Official Action.)

(It is advisable that all calculations made by the inspector be made on the official report form in order that these may be checked for accuracy later in the office.)

TEST METHOD

AEROSOL FOAM-TYPE PRODUCTS

On October 23, 1964, the National Bureau of Standards issued a draft of a step-by-step procedure developed to parallel Steps 1 through 5 found in Section 8.2 of Handbook 67 for use with non-food, foam-type aerosol products in standard-pack packages:

Standard-Pack Packages, Aerosol Foam-Type Products

(Packaged aerosol foam-type products should be checked at a temperature between 70° to 80°F.)

Step 1. Select a sample of 10 or more identical packages (identical as to labeled weight, brand, and commodity). Remove any covers or caps not required for dispensing the product.

Step 2. Check the gross weight of each package to determine the lightest and heaviest package in the sample. Record the gross weight of the lightest and heaviest package.

Step 3. Following instructions on the container, prepare the lightest package for the checking procedure. If shaking is specified shake the container with a wrist-twisting motion for 15 seconds at the approximate rate of one complete cycle per second.

Step 4. Exhaust the lightest container by holding the valve wide open for 35 minutes. During this exhausting procedure the container should be held in the proper position (generally upright or inverted) as specified in the instructions on the package. (A lightweight, portable, test stand equipped with an adjustable valve-button depressor may be used for this operation.)

Step 5. Rinse and dry the exterior of the container. (If the nozzle is removable, remove for cleaning and drying, and then replace the nozzle.)

Step 6. Weigh the empty container to determine the wet tare. (The wet tare is defined as the weight of the container plus any product that is not expelled during the exhausting procedure.)

Step 7. Determine the regeneration allowance by reference to the following table. (The regeneration allowance is the difference in the weight of product delivered through normal consumer usage and the weight of the product delivered through the accelerated procedure as outlined in Step 4, and as determined by laboratory investigation.) The regeneration allowance also may be computed by multiplying the labeled weight by the regeneration factor (which has been determined to be 0.02 for foam-type products). Thus, if the labeled weight is 6¼ oz. (6.25 oz.), obtain the regeneration allowance by multiplying the labeled weight by 0.02 (i.e., 6.25 oz. x 0.02 = 0.125 oz. = $^{2}/_{16}$ oz.).

Labeled Weight of Package	*Regeneration Allowance (label wt. x factor of 0.02)*
Zero to less than 1 $^{9}/_{16}$ oz.	Zero
1 $^{9}/_{16}$ oz. to less than 4 $^{11}/_{16}$ oz.	$^{1}/_{16}$ oz.
4 $^{11}/_{16}$ oz. to less than 7 $^{13}/_{16}$ oz.	$^{2}/_{16}$ oz.
7 $^{13}/_{16}$ oz. to less than 10 $^{15}/_{16}$ oz.	$^{3}/_{16}$ oz.
10 $^{15}/_{16}$ oz. to less than 14 $^{1}/_{16}$ oz.	$^{4}/_{16}$ oz.
14 $^{1}/_{16}$ oz. to less than 1 lb. 1 $^{3}/_{16}$ oz.	$^{5}/_{16}$ oz.
1 lb. 1 $^{3}/_{16}$ oz. to less than 1 lb. 4 $^{5}/_{16}$ oz.	$^{6}/_{16}$ oz.
1 lb. 4 $^{5}/_{16}$ oz. to less than 1 lb. 7 $^{7}/_{16}$ oz.	$^{7}/_{16}$ oz.
1 lb. 7 $^{7}/_{16}$ oz. to less than 1 lb. 10 $^{9}/_{16}$ oz.	$^{8}/_{16}$ oz.

Step 8. Subtract the regeneration allowance from the wet tare to obtain the corrected wet tare.

Step 9. Subtract the corrected wet tare from the gross weight to obtain the adjusted net weight of the lightest package.

Step 10. If the adjusted net weight of the lightest package at least equals the declared net weight it may be reasonable to assume that the lot is satisfactory.

Step 11. If the adjusted net weight of the lightest package is less than the declared weight it will be necessary to treat the 10 packages as a sample of the lot and proceed to weigh them individually to determine individual errors. For this procedure it will be essential to arrive at an average corrected wet tare weight to be added to the labeled net weight of the package to determine a "standard" gross weight with which the packages will be compared.

In order to arrive at a representative average corrected wet tare weight for the sample, Steps 3 through 8 must be repeated with the heaviest package to obtain its corrected wet tare weight. The average of the two corrected wet tare weights may then be accepted as the tare weight for the weighing of individual packages. In rounding off this average always round off to the lower figure, (i.e., the average of $2^{10}/_{16}$ oz. and $2^{11}/_{16}$ oz. is $2^{10}/_{16}$ oz.). (The inspector is cautioned that the tare of a single package is not considered acceptable as an average corrected wet tare, and also that no "permanent" or "reference" record of tares is acceptably reliable.)

Step 12. With standard weights in an amount equal to the "standard" gross weight for the sample packages on one side of the scale (or as the "standard" gross weight in the "substitution" procedure if an equal-arm scale is not used), weigh the remaining packages of the sample and record the error of all sample packages. Exclude, by circling, any errors (±) that are unreasonably large and determine an average error for the sample (see Steps 1, 2, 3, 4, and 5 of 8.1, Handbook 67).

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TEST METHOD

AEROSOL PRODUCTS — LOW VISCOSITY

On February 23, 1965, the National Bureau of Standards issued a step-by-step procedure developed to parallel Steps 1 through 5 of Section 8.2 of Handbook 67 for use with low viscosity aerosol products in standard-pack packages:

Standard-Pack Packages, Aerosol Products (Low Viscosity)

(Low viscosity packaged aerosol products should be checked at a temperature between 70° to 80°F. The fumes released when emptying the containers may be toxic and/or flammable. The exhausting procedure should be conducted in a well-ventilated area or outdoors. No smoking should be permitted in the test area. The containers should not be punctured or subjected to temperatures in excess of 110°F.)

Step 1. Select a sample of 10 or more identical packages (identical as to labeled weight, brand, and commodity.) Remove any overcaps not required for dispensing the product.

Step 2. Check the gross weight of each package to determine the lightest and heaviest package in the sample. Record the gross weight of the lightest and heaviest package.

Step 3. Following instructions on the container, prepare the lightest package for the checking procedure. If shaking is specified the shaking should be done according to the directions on the container. If no directions as to how the can should be shaken are given, shake the container with a wrist-twisting motion for 15 seconds at the approximate rate of one complete cycle per second.

Step 4. Exhaust the lightest container by holding the valve actuator depressed until no additional product or gas is expelled. During this exhausting procedure the container should be held in the proper position (generally upright) as specified in the instructions on the package. (A lightweight, portable, test stand equipped with an adjustable valve-actuator depressor may be used for this operation. If any product remains, this should be expelled as completely as possible by holding the container in the hand with the valve-actuator depressed and alternately inverting the container and then restoring to the original test position at approximately ten second intervals until no additional product is delivered. In cases where the can becomes chilled during the initial exhausting period, it is very important to hold the can in the hand during the inverting procedure or permit the container to warm up to 70° to 80° F before concluding the evacuation with the inverting procedure.

A container with a metered valve cannot be emptied by holding the valve-actuator depressed. (A metered valve is defined as a valve that permits only a predetermined amount of product to be expelled each time the valve-actuator is depressed.) The container will have to be emptied by alternately depressing and releasing the valve-actuator by hand until no additional product or gas is expelled.

Step 5. Rinse and dry the exterior of the container. (If the valve-actuator is removable, remove for cleaning and drying, and then replace.)

Step 6. Weigh the empty container to determine the wet tare. (The wet tare is defined as the weight of the container plus any product that is not expelled during the exhausting procedure.)

Step 7. Determine the test allowance by reference to the following table. (The test allowance is defined as the difference in the amount of product delivered through normal consumer usage and the amount of the product delivered through the procedure outlined in Step 4, as determined by laboratory investigation.)

Labeled Weight of Package	*Test Allowance*	
	Fractional	*Decimal*
Zero to less than 1½ oz.	Zero	Zero
1½ oz to less than 3 oz.	1/16 oz.	0.06
3 oz. or higher	2/16 oz.	0.13

Step 8. Subtract the test allowance from the wet tare to obtain the corrected wet tare.

Step 9. Subtract the corrected wet tare from the gross weight to obtain the adjusted net weight of the lightest package.

Step 10. If the adjusted net weight of the lightest package at least equals the declared net weight it may be reasonable to assume that the lot is satisfactory.

Step 11. If the adjusted net weight of the lightest package is less than the declared weight it will be necessary to treat the 10 packages as a sample of the lot and proceed to weigh them individually to determine individual errors. For this procedure it will be essential to arrive at an average corrected wet tare weight to be added to the labeled net weight of the package to determine a "standard" gross weight with which the packages will be compared.

In order to arrive at a representative average corrected wet tare weight for the sample, Steps 3 through 8 must be repeated with the heaviest package to obtain its corrected wet tare weight. The average of the two corrected wet tare weights may then be accepted as the tare weight for the weighing of individual packages. In rounding off this average always round off to the lower figure (i.e., the average of 2 1/16 oz. and 2 1/16 oz. is 2 0/16 oz.) (The inspector is cautioned that the tare of a single package is not considered acceptable as an average corrected wet tare, and also that no "permanent" or "reference" record of tares is acceptably reliable.)

Step 12. With standard weights in an amount equal to the "standard" gross weight for the sample packages on one side of the scale (or as the "standard" gross weight in the "substitution" procedure if an equal-arm scale is not used), weigh the remaining packages of the sample and record the error of all sample packages. Exclude, by circling, any errors (±) that are unreasonably large and determine an average error for the sample (see Steps 1, 2, 3, 4, and 5 of 8.1., NBS Handbook 67.)

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TEST METHOD

AEROSOL PRODUCTS — FOOD

On June 11, 1965, the National Bureau of Standards issued a step-by-step procedure developed to parallel Steps 1 through 5 of Section 8.2 of Handbook 67 for use with aerosol food products in standard-pack packages:

Standard-Pack Packages, Aerosol Products (Food)

(Packaged aerosol food products should be at a temperature of 70° to 80°F. when checked unless refrigerated. Refrigerated products should be at a temperature between 37° and 43°F. when checked.)

Step 1. Select a sample of 10 or more identical packages (identical as to labeled weight, brand, and commodity). Remove any overcaps not required for dispensing the product.

Step 2. Check the gross weight of each package to determine the lightest and heaviest package in the sample. Record the gross weight of the lightest and heaviest package.

Step 3. Following instructions on the container, prepare the lightest package for the checking procedure. If shaking is specified, the shaking should be done according to the directions on the container. If no directions as to how the can should be shaken are given, shake the container with a wrist-twisting motion for 15 seconds at the approximate rate of two complete cycles per second.

Step 4. Exhaust the lightest container by holding the valve wide open for 30 minutes. During this exhausting procedure the container should be held in the proper position (generally, upright or inverted) as specified in the instructions on the package. (A lightweight, portable test stand equipped with an adjustable valve-button depressor may be used for this operation.

Step 5. Rinse and dry the exterior of the container. (If the valve-actuator is removable, remove for cleaning and drying, and then replace.)

Step 6. Weigh the empty container to determine the wet tare. (The wet tare is defined as the weight of the container plus any product that is not expelled during the exhausting procedure.)

Step 7. Determine the test allowance by reference to the following table. (The test allowance is an allowance for the difference in the amount of product delivered through normal consumer usage and the amount of the product delivered through the procedure outlined in Step. 4.)

Labeled Weight of Package		*Test Allowance*	
		Fractional	*Decimal*
Zero to less than	$1^{9}/_{16}$ oz.	Zero	Zero
$1\ ^{9}/_{16}$ oz. to less than	$4^{11}/_{16}$ oz.	$^{1}/_{16}$ oz.	0.06 oz.
$4\ ^{11}/_{16}$ oz. to less than	$7^{13}/_{16}$ oz.	$^{2}/_{16}$ oz.	0.13 oz.
$7\ ^{13}/_{16}$ oz. to less than	$10^{15}/_{16}$ oz.	$^{3}/_{16}$ oz.	0.19 oz.
$10\ ^{15}/_{16}$ oz. to less than	$14\ ^{1}/_{16}$ oz.	$^{4}/_{16}$ oz.	0.25 oz.
$14\ ^{1}/_{16}$ oz. to less than 1 lb.	$1\ ^{3}/_{16}$ oz.	$^{5}/_{16}$ oz.	0.31 oz.
1 lb. $1\ ^{3}/_{16}$ oz. or higher		$^{6}/_{16}$ oz.	0.38 oz.

Step 8. Subtract the test allowance from the wet tare to obtain the corrected wet tare.

Step 9. Subtract the corrected wet tare from the gross weight to obtain the adjusted net weight of the lightest package.

Step 10. If the adjusted net weight of the lightest package at least equals the declared net weight, it may be reasonable to assume that the lot is satisfactory.

Step 11. If the adjusted net weight of the lightest package is less than the declared weight, it will be necessary to treat the 10 packages as a sample of the lot and proceed to weigh them individually to determine individual errors. For this procedure it will be essential to arrive at an average corrected wet tare weight to be added to the labeled net weight of the package to determine a "standard" gross weight with which the packages will be compared.

In order to arrive at a representative average corrected wet tare weight for the sample, Steps 3 through 8 must be repeated with the heaviest package to obtain its corrected wet tare weight. The average of the two corrected wet tare weights may then be accepted as the tare weight for the weighing of individual packages. In rounding off this average, always round off to the lower figure (i.e., the average of $2^{10}/_{16}$oz. and $2^{11}/_{16}$ oz. is $2^{10}/_{16}$ oz.). (The inspector is cautioned that the tare of a single package is not considered acceptable as an average corrected wet tare, and also that no "permanent" or reference" record of tares is acceptably reliable.)

Step 12. With standard weights in an amount equal to the "standard" gross weight for the sample packages on one side of the scale (or as the "standard" gross weight in the "substitution" procedure if an equal-arm scale is not used), weigh the remaining packages of the sample and record the error of all sample packages. Exclude, by circling, any errors (±) that are unreasonably large and determine an average error for the sample (see Steps 1, 2, 3, 4, and 5 of 8.1., NBS Handbook 67).

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TEST METHOD

AEROSOL PRODUCTS — HIGH VISCOSITY

On June 11, 1965 the National Bureau of Standards issued a step-by-step procedure developed to parallel Steps 1 through 5 of Section 8.2 of the Handbook for use with high viscosity aerosol products in standard-pack packages:

Standard-Pack Packages, Aerosol Products (High Viscosity)

(High viscosity aerosol products should be at a temperature between 70° to 80°F. when checked. The fumes and the product released when emptying the containers may be toxic and/or flammable. The exhausting procedure should be conducted in a well-ventilated area or outdoors. No smoking should be permitted in the test area. The containers should not be punctured or subjected to temperatures in excess of 110°F.)

Step 1. Select a sample of 10 or more identical packages (identical as to labeled weight, brand, and commodity). Remove any overcaps not required for dispensing the product.

Step 2. Check the gross weight of each package to determine the lightest and heaviest package in the sample. Record the gross weight of the lightest and heaviest package.

Step 3. Following directions on the container, prepare the lightest package for the checking procedure. If shaking is specified, the shaking should be done according to the directions on the container. If no specific directions as to how the can should be shaken are given, shake the container with a brisk wrist-twisting motion for one minute at the approximate rate of two complete cycles per second. If the sample contains a ball agitator, this shaking procedure should continue for one minute after the ball has shaken loose.

Step 4. Exhaust the lightest container by holding the valve-actuator depressed until the visual spray pattern is interrupted. During this exhausting procedure the container should be held in the proper position (generally upright) as specified in the directions on the package. (A lightweight, portable test stand equipped with an adjustable valve-actuator depressor used in conjunction with a receiving vessel may be used for this operation.

As soon as the visual spray pattern is interrupted, release the actuator. Allow the container to warm up to 70° to 80°F. before concluding the evacuation. Agitate the container with a swirling motion for 30 seconds. Hold the container at approximately a 45° angle, with the valve-actuator depressed, and rotate the container to maintain the visual spray as long as possible. (This will ensure contact of the dip tube with any remaining liquid in the container.) Continue this procedure until no additional liquid or gas is expelled.

Step 5. Rinse with a suitable solvent and dry the exterior of the container. (If the valve-actuator is removable, remove for cleaning and drying, and then replace.)

Step 6. Weigh the empty container to determine the wet tare. (The wet tare is defined as the weight of the container plus any product that is not expelled during the exhausting procedure.).

Step 7. Determine the test allowance by reference to the following table. (The test allowance is an allowance for the difference in the amount of product delivered through normal consumer usage and the amount of the product delivered through the procedure outlined in Step 4.)

Labeled Weight of Package	*Test Allowance*	
	Fractional	*Decimal*
Zero to less than 1 9/16 oz.	Zero	Zero
1 9/16 oz. to less than 5 oz.	1/16 oz.	0.06 oz.
5 oz. to less than 8 oz.	3/16 oz.	0.13 oz.
8 oz. to less than 12 oz.	5/16 oz.	0.19 oz.
12 oz. or higher	4/16 oz.	0.25 oz.

Step 8. Subtract the test allowance from the wet tare to obtain the corrected wet tare.

Step 9. Subtract the corrected wet tare from the gross weight to obtain the adjusted net weight of the lightest package.

Step 10. If the adjusted net weight of the lightest package at least equals the declared net weight, it may be reasonable to assume that the lot is satisfactory.

Step 11. If the adjusted net weight of the lightest package is less than the declared weight, it will be necessary to treat the 10 packages as a sample of the lot and proceed to weigh them individually to determine individual errors. For this procedure it will be essential to arrive at an average corrected wet tare weight to be added to the labeled net weight of the package to determine a "standard" gross weight with which the packages will be compared.

In order to arrive at a representative average corrected wet tare weight for the sample, Steps 3 through 8 must be repeated with the heaviest package to obtain its corrected wet tare weight. The average of the two corrected wet tare weights may then be accepted as the tare weight for the weighing of individual packages. In rounding off this average always round off to the lower figure (i.e., the average of 2 10/16 oz. and 2 11/16 oz. is 2 10/16 oz.). (The inspector is cautioned that the tare of a single package is not considered acceptable as an average corrected wet tare, and also that no "permanent" or "reference" record of tares is acceptably reliable.)

Step 12. With standard weights in an amount equal to the "standard" gross weight for the sample packages on one side of the scale (or as the "standard" gross weight in the "substitution" procedure if an equal-arm scale is not used), weigh the remaining packages of the sample and record the error of all sample packages. Exclude, by circling, any errors (±) that are unreasonably large and determine an average error for the sample (see Steps 1, 2, 3, 4, and 5 of 8.1.1., NBS Handbook 67).

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INDUSTRY SPECIFICATIONS FOR FABRICATED AEROSOL CANS

Presented and passed as Modified in Total at the 51st Annual Meeting of C.S.M.A. in 1964, and revised at the 52nd Annual Meeting of C.S.M.A. in 1965.

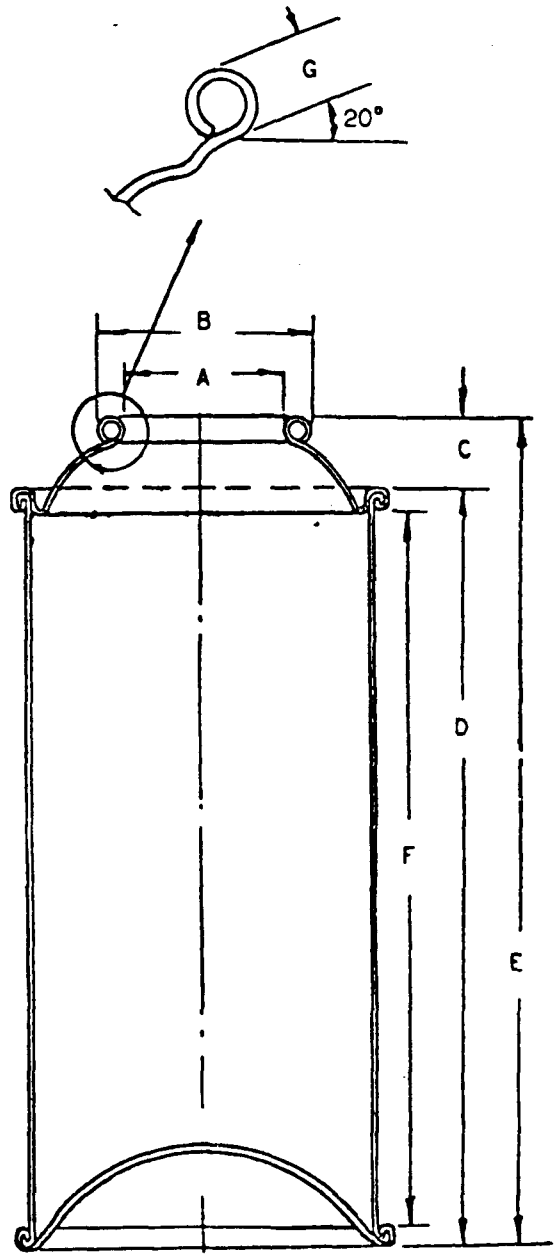
Sales Code Diameter and Height of all Fabricated Aerosol Cans Covered by Industry Specifications to date:

202 Diameter	207.5 Diameter	211 Diameter	300 Diameter
202 x 214	207.5 x 413	211 x 407.5	300 x 709
202 x 314	207.5 x 509	211 x 413	
202 x 406	207.5 x 605	211 x 510	
202 x 509	207.5 x 701	211 x 604	
		211 x 612	
		211 x 713	

Dimensions included in Industry Specifications to Date:

A. Inside Diameter of 1" Cup Opening.
B. Outside Diameter of 1" Cup Opening.
C. Height of Curl Opening above Double Seam.
D. Height over Double Seam.
E. Overall Height of Container.
F. Height Between Double Seams.
G. Thickness of Curl Around the One Inch Opening.
(Gauges for measuring Dimension G are available through C.S.M.A.)

Nominal Dimensions	A ±.004	B ±.010	C ±.016	D ±.031	E ±.047	F Min.	G ±.007
202 x 214	1.000	1.226	.396	Sales Code Height — .010 [See Example]	Item C Plus Item D [See Example]	Sales Code Height — .300 [See Example]	.130
x 314	1.000	1.226	.396				.130
x 406	1.000	1.226	.396				.130
x 509	1.000	1.226	.396				.130
207.5 x 413	1.000	1.226	.798				.130
x 509	1.000	1.226	.798				.130
x 605	1.000	1.226	.798				.130
x 701	1.000	1.226	.798				.130
211 x 407.5	1.000	1.226	.798				.130
x 413	1.000	1.226	.798				.130
x 510	1.000	1.226	.798				.130
x 604	1.000	1.226	.798				.130
x 612	1.000	1.226	.798				.130
x 713	1.000	1.226	.798				.130
300 x 709	1.000	1.226	.798				.130



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To determine items D, E & F proceed as follows:

EXAMPLE

211 x 413

The Sales Code Height is 413 or $4\frac{13}{16}$ [4.812].

$D = 4.812 - .010$
$= 4.802$
$E = C + D$
$= .798 + 4.802$
$= 5.600$
$F = 4.812 - .300$
$= 4.512$