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Standard Test Method for Volatile Content of Coatings¹

This standard is issued under the fixed designation D 2369; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense to replace Method 404 1.1 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method describes a procedure for the determination of the weight percent volatile content of solvent-based and water-reducible coatings. Test specimens are heated at $110 \pm 5^\circ\text{C}$ for 60 min.

NOTE 1—The coatings used in these round-robin studies represented air-dried, air-dried oxidizing, heat-cured baking systems, and also included multicomponent paint systems.

1.2 Sixty minutes at $110 \pm 5^\circ\text{C}$ is a general purpose test method based on the precision obtained with both solvent-based and water-reducible coatings (see Section 9). These coatings (single package, heat cured) are commonly applied in factories to automobiles, metal containers, flat (coil) metal and large appliances, and many other metal parts.

1.3 This test method is viable for coatings wherein one or more parts may, at ambient conditions, contain liquid coreactants that are volatile until a chemical reaction has occurred with another component of the multipackage system.

NOTE 2—Committee D-1 has run round-robin studies on volatiles of multicomponent paint systems. The only change in procedure is to premix the weighed components in the correct proportions and allow the solvent reduced specimens to stand at room temperature for 1 h prior to placing them into the oven.

1.4 This test method may not be applicable to all types of coatings such as some printing inks (see Test Method D 4713), and other procedures may be substituted with mutual agreement of the producer and user.

NOTE 3—If unusual decomposition or degradation of the specimen occurs during heating, the actual time and temperature used to cure the coating in practice may be substituted for the time and temperature specified in this test method, subject to mutual agreement of the producer and user. U.S. EPA Reference Method 24 specifies $110^\circ \pm 5^\circ\text{C}$ for 1 hr for coatings and test conditions for use with inks are specified in U.S. EPA Reference Method 24-A.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement see Note 6.*

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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2. Referenced Documents

2.1 ASTM Standards:

D 343 Specification for 2-Ethoxyethyl Acetate (95 % Grade)²

D 362 Specification for Industrial Grade Toluene³

D 1193 Specification for Reagent Water⁴

D 4713 Test Methods for Nonvolatile Content of Heatset and Liquid Printing Ink Systems⁵

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens⁶

E 180 Practice for Determining the Precision Data of ASTM Methods for Analysis and Testing of Industrial Chemicals⁷

2.2 Other Standards:

EPA Reference Method 24—Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings⁸

EPA Reference Method 24-A—Determination of Volatile Matter Content and Density of Printing Inks, and Related Coatings⁸

3. Summary of Test Method

3.1 A designated quantity of coating specimen is weighed into an aluminum foil dish containing 3 mL of an appropriate solvent, dispersed, and heated in an oven at $110 \pm 5^\circ\text{C}$ for 60 min. The percent volatile is calculated from the loss in weight.

4. Significance and Use

4.1 This test method is the procedure of choice for determining volatiles in coatings for the purpose of calculating the volatile organic content in coatings under specified test conditions. The inverse value, nonvolatile, is used to determine the weight percent solids content. This information is useful to the paint producer and user and to environmental interests for determining the volatiles emitted by coatings.

5. Apparatus

5.1 *Aluminum Foil Dishes*, 58 mm in diameter by 18 mm

² Discontinued; see 1982 Annual Book of ASTM Standards, Part 29.

³ Discontinued; see 1991 Annual Book of ASTM Standards, Vol 06.03.

⁴ Annual Book of ASTM Standards, Vol 11.01.

⁵ Annual Book of ASTM Standards, Vol 06.02.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Annual Book of ASTM Standards, Vol 15.05.

⁸ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.



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high with a smooth (planar) bottom surface. Precondition the dishes for 30 min in an oven at $110 \pm 5^\circ\text{C}$ and store in a desiccator prior to use. Use tongs or rubber gloves or both, to handle the dishes.

5.2 *Forced Draft Oven*, Type IIA or Type IIB as specified in Specification E 145.

NOTE 4—Be sure the shelves are level.

5.3 *Syringe*, 1-mL, capable of properly dispensing the coating under test at sufficient rate that the specimen can be dissolved in the solvent.

NOTE 5—Disposable syringes are recommended.

5.4 *Test Tube*, with new cork stopper.

5.5 *Weighing or Dropping Bottle*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁹ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

6.3 *Toluene*, technical grade, Specification D 362.

6.4 *2-Ethoxyethyl Acetate*, technical grade, Specification D 343.

7. Procedure

7.1 Mix the sample, preferably on a mechanical shaker or roller, until homogeneous. If air bubbles become entrapped, stir by hand until the air has been removed.

NOTE 6—For water reducible paint systems it is recommended that they be stirred by hand.

7.2 Using an appropriate weighing container (5.3, 5.4, or 5.5, with the syringe preferred for highest precision) weigh to 0.1 mg, by difference, the correct specimen weight according to the following:

Expected % Non Volatile	Expected % Volatile	Specimen Weight, % g
60 % or more	40 % or less	0.3 ± 0.1
Less than 60 %	More than 40 %	0.5 ± 0.1

The specimen is added to a tared aluminum foil dish (W_1) into which has been added 3 ± 1 mL of suitable solvent (6.2, 6.3 or 6.4). Add the specimen dropwise, shaking (swirling) the dish to disperse the specimen completely in the solvent. If the material forms a lump that cannot be dispersed, discard the specimen and prepare a new one. Similarly prepare a duplicate.

⁹ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

NOTE 7—Be sure to wipe the outer surface of the syringe clean after obtaining the specimen. Pull the syringe plunger up $\frac{1}{4}$ of an in. in order to pull the specimen away from the neck of the syringe. Cap and weigh. After dispensing the paint specimen do not wipe the tip of the syringe. Remove the specimen from the neck of the syringe by pulling up the plunger. Cap and reweigh.

NOTE 8—Use disposable (no talc) rubber gloves or polyethylene to handle the syringe.

NOTE 9—If the specimen cannot be dispersed in the solvents listed (6.2, 6.3, or 6.4) a compatible solvent may be substituted provided it is no less volatile than 2-ethoxyethyl acetate (6.4).

7.3 For multicomponent systems, weigh the components in the proper proportion into containers that can be capped. Mix the components together immediately, weigh the appropriate sample size from the table in 7.2, as specified into the aluminum weighing dishes containing 3 ± 1 mL of a suitable solvent. After dispersing, allow to stand for 1 h at room temperature (induction period) before placing the dishes into the oven.

NOTE 10—Other induction periods are used. See U.S. EPA Reference Method 24.

7.4 Heat the aluminum foil dishes containing the dispersed specimens in the forced draft oven (5.2) for 60 min at $110 \pm 5^\circ\text{C}$.

NOTE 11: **Precaution**—In addition to other precautions, provide adequate ventilation, consistent with accepted laboratory practice, to prevent solvent vapors from accumulating to a dangerous level.

7.5 Remove the dishes from the oven, place immediately in a desiccator, cool to ambient temperature, and weigh to 0.1 mg.

8. Calculation

8.1 Calculate the percent volatile matter, V , in the liquid coating as follows:

$$V = 100 - [(W_2 - W_1)/S] \times 100 \quad (1)$$

where:

W_1 = weight of dish,

W_2 = weight of dish plus specimen after heating, and

S = specimen weight.

8.2 The percent of nonvolatile matter, N , in the coating may be calculated by difference as follows:

$$N = 100 - V \quad (2)$$

9. Precision and Bias

9.1 The precision estimated for tests at 60 min at $110 \pm 5^\circ\text{C}$ are based on an interlaboratory study¹⁰ in which 1 operator in each of 15 laboratories analyzed in duplicate on 2 different days 7 samples of water-reducible paints and 8 samples of solvent-based paints containing between 35 and 72 % volatile material. The paints were commercially supplied. The results were analyzed statistically in accordance with Practice E 180. The within-laboratory coefficient of variation was found to be 0.5 % relative at 213 degrees of freedom and the between-laboratories coefficient of variation was 1.7 % relative at 198 degrees of freedom. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

9.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on

¹⁰ Supporting data are available from ASTM Headquarters. Request RR-D01-1026.