Designation: D 2369 – 03~~4~~

Standard Test Method for Volatile Content of Coatings[1]

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 standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method describes a procedure for the determination of the weight percent volatile content of solventborne and waterborne coatings. Test specimens are heated at 110 ± 5°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 ± 5°C is a general purpose test method based on the precision obtained with both solventborne and waterborne 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.~~ 9).

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 multi-package system.

NOTE 2—Committee D01 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 specimens to stand at room temperature for 1 h prior to placing them into the oven.

1.4 Test Method D 5095 for Determination of the Nonvolatile Content in Silanes, Siloxanes and Silane-Siloxane Blends Used in Masonry Water Repellent Treatments is the standard method for nonvolatile content of these types of materials.

1.5 Test Methods D 5403 for Volatile Content of Radiation Curable Materials is the standard method for determining nonvolatile content of radiation curable coatings, inks and adhesives.

1.6 Test Method D 6419 for Volatile Content of Sheet-Fed and Coldset Web Offset Printing Inks is the method of choice for these types of printing inks.

1.7 This test method may not be applicable to all types of coatings. Other procedures may be substituted with mutual agreement between the producer and the 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 between the producer and the user. The U.S. EPA Reference Method 24 specifies 110 ± 5°C for 1 h for coatings.

NOTE 4—Practice D 3960 for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings describes procedures and calculations and provides guidance on selecting test methods to determine VOC content of solventborne and waterborne coatings.

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

1.9 *This standard does not purport to address all of the safety concerns, 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 7.4.1.

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[1] This test method is under the jurisdiction of ASTM Committee D01 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:* [2]

D 1193 Specification for Reagent Water

D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

D 3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings

D 5095 Test Method for Determination of the Nonvolatile Content in Silanes, Siloxanes and Silane-Siloxane Blends Used in Masonry Water Repellent Treatments

D 5403 Test Methods for Volatile Content of Radiation Curable Materials

D 6419 Test Method for Volatile Content of Sheet-Fed and Coldset Web Offset Printing Inks

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

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals

2.2 *Other Standards:*

EPA Reference Method 24—Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings[3]

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 ± 5°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 weight percent solids content (nonvolatile matter) may be determined by difference. 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 *Analytical Balance*, capable of weighing ±0.1 mg.

5.2 *Aluminum Foil Dishes*, 58 mm in diameter by 18 mm high with a smooth (planar) bottom surface. Precondition the dishes for 30 min in an oven at 110 ± 5°C and store in a desiccator prior to use. Use tongs or rubber gloves, or both, to handle the dishes.

5.3 *Forced Draft Oven*, Type IIA or Type IIB as specified in Specification E 145. The oven must be operating in accordance with Specification E 145, since it is important to have proper air flow and good temperature control to ensure good precision.

NOTE 45—Be sure the shelves are level and dampers are open.

5.4 *Syringe*, 1-mL without needle, but equipped with caps, capable of properly dispensing the coating under test, at a sufficient rate so that the specimen can be dissolved in the solvent.

NOTE 56—Disposable syringes with caps are recommended.

5.5 *Paper Clips*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

6.3 *Toluene*, water or appropriate solvent.

7. Procedure

7.1 Take a representative sample of the liquid coatings in accordance with Practice D 3925. Mix thoroughly before taking specimens for individual tests. If air bubbles become entrapped, stir by hand, until the air has been removed.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards*, ~~Vol 11.01.~~ volume information, refer to the standard's Document Summary page on the ASTM website.

~~*Annual Book*~~

[3] Available from Superintendent of ~~ASTM Standards, Vol 06.01.~~ Documents, U.S. Government Printing Office, Washington, DC 20402.

[4] ~~*Annual Book*~~*Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of ~~ASTM~~ reagents not listed by the American Chemical Society, see *Analar Standards*, ~~Vol 06.02.~~ for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

NOTE 67—For waterborne paint systems it is recommended that they be stirred by hand.

NOTE 78—Paint samples tend to settle in the syringe. Obtain a freshly stirred sample for each test run.

7.2 Using a syringe weigh to 0.1 mg, by difference, the correct specimen weight according to the following:

Expected % Nonvolatile	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 or 6.3). 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.

NOTE 89—Draw the specimen into the syringe. Remove the syringe from the specimen, pull the plunger tip up 6.4 mm (¼ in.) in order to pull the specimen away from the neck of the syringe. Wipe the outer surface of the syringe to remove excess material and cap the syringe. Weigh to the nearest 0.1 mg. After dispensing the 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 to the nearest 0.1 mg.

NOTE 910—To facilitate dispersing the sample completely in the solvent, a metal paper clip may be placed (partially unfolded to use as a stirrer) in the preconditioned aluminum dish before weighing. The clip may then be used as a stirrer to completely disperse the specimen into the solvent. Be sure to leave the paper clip in the dish throughout the remainder of the procedure (through 7.5).

7.2.1 Similarly prepare a duplicate.

7.2.2 The result obtained is based on the mean of the results obtained in 7.2 and 7.2.1.

NOTE 101—Use disposable (no talc) rubber gloves or polyethylene to handle the syringe.

NOTE 112—If the specimen cannot be dispersed in the solvents listed (6.2 or 6.3), a compatible solvent may be substituted provided it is no less volatile than 2-ethoxyethyl acetate.

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 123—Other induction periods are also used. See U.S. EPA Reference Method 24.

7.4 Heat the aluminum foil dishes containing the dispersed specimens in the forced draft oven (5.3) for 60 min at 110 ± 5°C.

7.4.1 **Warning:** 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:

$$V_A = 100 - [((W_2 - W_1)/S_A) \times 100] \quad (1)$$

where:
V_A = % volatiles (first determination),
W_1 = weight of dish,
W_2 = weight of dish plus specimen after heating,
S_A = specimen weight, and
V_B = % volatiles (duplicate determination, calculate in same manner as V_A).

$$V = (V_A + V_B)/2 \quad (2)$$

8.2 Report *V*, the mean of the duplicate determination if relative percent difference is 1.5 % or less. If relative difference between V_A and V_B is greater than 1.5 %, repeat the duplicate determinations.

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

$$N = (N_A + N_B)/2 \quad (3)$$

where:
N_A = $100 - V_A$, and
N_B = $100 - V_B$.

N_A represents first determination and N_B represents duplicate determination.

9. Precision and Bias

9.1 The precision estimated for tests at 60 min at 110± 5°C are based on an interlaboratory study[5] in which 1 operator in each of 15 laboratories analyzed in duplicate on 2 different days 7 samples of waterborne paints and 8 samples of solventborne 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 df and the between-laboratories coefficient of variation was 1.7 % relative at 198 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

9.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 1.5 % relative.

9.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 4.7 % relative.

9.2 *Bias*—Bias has not been determined.

9.3 The precision results for multicomponent systems are based on an interlaboratory study in which one operator in each of five laboratories analyzed in duplicate on two different days, four samples of commercially supplied solventborne and waterborne multicomponent systems. The results were analyzed statistically in accordance with Practice E 180.

9.3.1 *Repeatability for Solventborne Multicomponent Systems*:

Coefficient of variation	0.5 %
Degrees of freedom	6
Factor (based on 95 % confidence level)	3.46
Precision	1.74 %

Two results, each the mean of duplicate determinations obtained by the same operator on different days, should be considered suspect if they vary by more than 1.74 % relative.

9.3.2 *Reproducibility for Solventborne Multicomponent Systems*:

Coefficient of variation	1.46 %
Degrees of freedom	5
Factor (based on 95 % confidence level)	3.46
Precision	5.31 %

Two results, each the mean of duplicate determinations obtained by operators in different laboratories, should be considered suspect if they vary by more than 5.31 % relative.

9.3.3 *Repeatability for Waterborne Multicomponent Systems*:

Coefficient of variation	0.53 %
Degrees of freedom	6
Factor (based on 95 % confidence level)	3.46
Precision	1.84 %

Two results, each the mean of duplicate determinations obtained by the same operator on different days, should be considered suspect if they vary by more than 1.84 % relative.

9.3.4 *Reproducibility for Waterborne Multicomponent Systems:*

Coefficient of variation	0.94 %
Degrees of freedom	5
Factor (based on 95 % confidence level)	3.64
Precision	3.43 %

Two results, each the mean of duplicate determinations obtained by operators in different laboratories, should be considered suspect if they vary by more then 3.43 % relative.

9.4 *Bias*—Since there is no accepted standard for volatile content in coatings, bias cannot be determined.

10. Keywords

10.1 multicomponent paints; nonvolatile determination; VOC baking temperature; VOC in paints; volatile determination; volatiles

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[5] Supporting data are available from ASTM Standards, Vol 14.04. International Headquarters. Request RR: D01-1026.