Designation: ~~D2369 – 20~~ D2369 – 24

Standard Test Method for Volatile Content of Coatings[1]

This standard is issued under the fixed designation D2369; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. 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 U.S. 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~~ 110 °C ± 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~~110 °C ± 5 °C is a general purpose test method based on the precision obtained with both solventborne and waterborne coatings (see Section 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 D5095 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 D5403 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 D6419 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~~ 110 °C ± 5 °C for 1 h for coatings.

[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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NOTE 4—Practice D3960 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 standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.10 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water
D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings
D3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings
D5095 Test Method for Determination of the Nonvolatile Content in Silanes, Siloxanes and Silane-Siloxane Blends Used in Masonry Water Repellent Treatments
D5403 Test Methods for Volatile Content of Radiation Curable Materials
D6419 Test Method for Volatile Content of Sheet-Fed and Coldset Web Offset Printing Inks
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Standards:*

EPA Reference Method 24 Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings [4]

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~~110 °C ± 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*[5], 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~~110 °C ± 5 °C and store in a desiccator prior to use. Use tongs or rubber gloves, or both, to handle the dishes.

[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* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

[5] Available from Fisher, Disposable Aluminum Dishes (code 08–732–106), www.fishersci.com; McAlister Bicknell, Aluminum Weighing Dishes (code 10676–0000), www.mbcoct.com; Cole-Parmer, Disposable Aluminum Weigh Dishes (code EW-01017–44), www.coleparmer.com.

5.3 *Forced Draft Oven,* ~~Type IIA or Type IIB as specified in Specification~~Proper air flow and temperature control are critical for ~~E145. The oven must be operating in accordance with Specification~~ good precision, while adequate ~~E145, since it is important to have proper air flow and good temperature control to ensure good precision.~~air exchange minimizes accumulation of flammable solvent vapors. The forced draft oven must operate in accordance with the following specifications:

~~NOTE 5—Be sure the shelves are level and dampers are open.~~

5.3.1 *Temperature Uniformity:* the consistency of heating throughout the oven chamber. The oven must achieve a temperature uniformity of ≤ 4.0 °C at 110 °C and temperature controller stability of ≤ 1.0 °C. Temperature controller stability is the difference between the load temperature and setpoint temperature over time.

5.3.2 *Time Constant:* the time required for the oven to return to setpoint temperature after opening oven door. The oven must recover to the setpoint temp of 110 °C within 5 minutes of leaving oven door open for 30 seconds.

5.3.3 *Air Exchanges:* how often fresh laboratory air is introduced into the oven chamber. The oven must operate at ≥ 50 air exchanges per hour.

5.4 Each specification should be certified by the oven manufacturer, confirmed by the user, or tested by a qualified third party. Evaluation of oven specifications should be performed according to the procedures outlined in Specification E145.

NOTE 5—Be sure the shelves are level and dampers are open.

5.5 *Syringe,* ~~1-mL without~~ 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 6—Disposable syringes with caps are recommended.

5.6 *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.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D1193.

6.3 *Toluene,* water or appropriate solvent.

7. Procedure

7.1 Take a representative sample of the liquid coating (each component) in accordance with Practice D3925. Mix thoroughly by hand before taking specimens.

7.2 For multi-component coatings, weigh each component in the proper proportion into a container that can be capped. Mix the components together thoroughly by hand before extracting specimens. Tightly close the container to prevent loss of volatile materials.

NOTE 7—Manufacturers often specify mix ratios by volume. To minimize errors, prepare the mixture gravimetrically using the measured density of each

[6] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

component to convert volume to mass. The calculated mass and prepared mass should differ by no more than 0.1 % relative.

7.3 Weigh the preconditioned aluminum foil dish (see 5.2) and record the weight to the nearest 0.1 mg (W_1). Use disposable (no talc) rubber or polyethylene gloves, tweezers or forceps to handle the dish.

7.4 To facilitate dispersing or spreading the specimen, a metal paper clip may be placed (partially unfolded) in the aluminum dish and weighed with the dish. If a paper clip is used, it must remain with the dish throughout the remainder of the procedure.

7.5 Add to the aluminum foil dish the appropriate type and amount of solvent according to Table 1.

7.6 Draw the coating specimen into the syringe. Remove the syringe from the specimen and then pull the plunger tip up 6 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. Place the filled syringe on the scale and tare the scale. Use disposable (no talc) rubber or polyethylene gloves to handle the syringe.

7.7 Remove the cap and dispense from the syringe into the dish the target specimen weight as specified in Table 1. If solvent is used in the dish add the specimen dropwise to the solvent-containing dish. The paper clip may be used to help disperse the coating specimen in the solvent. If the material forms a lump that cannot be dispersed, discard the specimen and prepare a new one. If no solvent is used (see Table 1, Method E), spread out the specimen in the dish with the paper clip to cover the bottom of the dish completely with as uniform thickness as possible.

7.8 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 place the syringe on the balance (that was tared with the syringe before the specimen was dispensed) and record the weight to the nearest 0.1 mg as the Specimen Weight (S_A).

7.9 Repeat steps 7.3 – 7.8 to prepare a duplicate specimen for each sample.

NOTE 8—Duplicate determinations must meet the relative percent difference requirements detailed in 8.2.

7.10 For multi-component coatings, after the specimens are prepared, allow them to sit at ambient conditions for a prescribed induction time according to Table 1 before placing the dishes in the oven.

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

7.12 Remove each dish from the oven, place immediately in a desiccator, cool to ambient temperature, weigh to the nearest 0.1 mg and record this weight (W_2) for each specimen.

TABLE 1 Summary of Methods

Coating Type	Method A – One Component Waterborne	Method B – One Component Solventborne	Method C – Multi-Component Waterborne	Method D – Multi-Component Solventborne	Method E – Multi-Component >90 % Solids
~~Solvent Type and Amount~~	~~3 ± 1 ml water (6.2)~~	~~3 ± 1 ml solvent (6.3)~~	~~3 ± 1 ml water (6.2)~~	~~3 ± 1 ml solvent (6.3)~~	~~none~~
Solvent Type and Amount	3 ml ± 1 ml water (6.2)	3 ml ± 1 ml solvent (6.3)	3 ml ± 1 ml water (6.2)	3 ml ± 1 ml solvent (6.3)	none
~~Specimen Weight~~	~~0.3 ± 0.1 g if expected result is =<40 % volatile (>=60 % non-volatile)~~ ~~0.5 ± 0.1 g if expected result is >40 % volatile (<60 % non-volatile)~~				~~see[A]~~
Specimen Weight	0.3 g ± 0.1 g if expected result is =<40 % volatile (>=60 % non-volatile) 0.5 g ± 0.1 g if expected result is >40 % volatile (<60 % non-volatile)				see[A]
Induction Time	N/A	N/A	1 hr (see[B])	1 hr (see[B])	24 hr

[A] Specimen weight to be representative of how the product is used (the lowest thickness which the manufacturer's literature recommends) where: Weight (g) = Thickness (mm) × 3.14 × [Dish Diameter^2 (mm^2)/4] × Density (g/cc)/1000. For example: the appropriate specimen weight for a coating with a density of 1 g/cc placed in a 50 mm diameter dish at a thickness of 0.5 mm calculates to 1.0 g.
[B] Other induction periods are used. See EPA Reference Method 24.