Designation: D2369 – 10 (Reapproved 2015)$^{\varepsilon 1}$

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.

ε^1 NOTE—Paragraph 9.3.2 was editorially corrected in June 2015.

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).

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 for 1 h for coatings.

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

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

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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] 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.

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 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 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 E145. The oven must be operating in accordance with Specification E145, since it is important to have proper air flow and good temperature control to ensure good precision.

NOTE 5—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 6—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.[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.

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,

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[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.

[6] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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
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]
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.

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.

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.

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.

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. The 1.5 % relative difference is not applicable to Method E.

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[7] 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 E180. 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 E180.

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 %

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1026. Contact ASTM Customer Service at service@astm.org.

TABLE 2 ILS Summary for Method E – % Volatile Material (V)[A]

Material	Average[B] V (%)	Repeatability Standard Deviation s_r	Reproducibility Standard Deviation s_R	Repeatability Limit r	Reproducibility Limit R
A	3.25	0.17	1.15	0.47	3.21
B	1.87	0.15	0.34	0.41	0.95
C	2.67	0.18	1.59	0.52	4.46
D	0.64	0.12	0.20	0.32	0.56
E	0.15	0.05	0.11	0.14	0.30

[A] The differences in the precision between the different materials (types of coatings) tested in this study are not due to the specific chemistry or class of material but are attributed to the specific formulations and components.

[B] The average of the laboratories' calculated averages.

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)	3.64
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.

9.5 *Precision for Multi-Component, >90 % Solids Systems*—The precision of this test was expanded to include data and analysis from Method E based on an interlaboratory study conducted in 2009. Each of eight laboratories tested five different materials, all multi-component, >90 % solids systems. Every "test result" represents the mean of duplicate determinations. Every laboratory reported three replicate test results for the analysis. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:D01-1152.[8]

9.5.1 *Repeatability Limit (r)*—Two test results, each the mean of duplicate determinations, obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

9.5.1.1 Repeatability limits for each of the materials tested are listed in Table 2.

9.5.2 *Reproducibility Limit (R)*—Two test results, each the mean of duplicate determinations, between different laboratories shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

9.5.2.1 Reproducibility limits for each of the metals tested are listed in Table 2.

9.5.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

9.5.4 Any judgment in accordance with statements 9.5.1 and 9.5.2 would have an approximate 95 % probability of being correct.

9.5.5 The precision statement for Method E for Multi-Component, >90 % Solids Systems was determined through statistical examination of 120 reported results, from eight laboratories, on five materials, which were identified as:

A – Two component amine cured epoxy clear coating
B – Two component amine cured epoxy pigmented coating
C – Two component urethane pigmented coating
D – Two component urethane waterproofing membrane coating
E – Two component amine cured epoxy/acrylate highway marking coating

9.6 *Bias*—At the time of the study, there was no accepted reference material or method suitable for determining the bias for this test method, therefore no statement on bias is being made.

10. Keywords

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

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1152. Contact ASTM Customer Service at service@astm.org.

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