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Standard Test Methods for Volatile Content of Radiation Curable Materials[1]

This standard is issued under the fixed designation D 5403; 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.

1. Scope

1.1 These test methods cover procedures for the determination of weight percent volatile content of coatings, inks, and adhesives designed to be cured by exposure to ultraviolet light or to a beam of accelerated electrons.

1.2 Test Method A is applicable to radiation curable materials that are essentially 100 % reactive but may contain traces (no more than 3 %) of volatile materials as impurities or introduced by the inclusion of various additives.

1.3 Test Method B is applicable to all radiation curable materials but must be used for materials that contain volatile solvents intentionally introduced to control application viscosity and which are intended to be removed from the material prior to cure.

1.4 These test methods may not be applicable to radiation curable materials wherein the volatile material is water, and other procedures may be substituted by mutual consent of the producer and user.

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.* A specific hazard statement is given in 15.7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 2369 Test Method for Volatile Content of Coatings[2]

E 145 Specification for Gravity–Convection and Forced–Ventilation Ovens[3]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Methods[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Terminology

3.1 *Definitions*:

3.1.1 *cure*—the condition of a coating after conversion to the final state of cure as measured by tests generally related to end use performance and mutually agreeable to supplier and purchaser.

3.1.2 *ultraviolet (UV) curing*—conversion of a coating from its application state to its final use state by means of a mechanism initiated by ultraviolet radiation generated by equipment designed for that purpose.

3.1.3 *electron beam (EB) curing*—conversion of a coating from its application state to its final use state by means of a mechanism initiated by electron beam radiation generated by equipment designed for that purpose.

3.1.4 *processing volatiles*—loss in specimen weight under test conditions that are designed to simulate actual industrial cure processing conditions.

3.1.5 *potential volatiles*—loss in specimen weight upon heating at 110°C for 60 min after radiation curing.

3.1.5.1 *Discussion*—This value is an estimation of volatile loss that may occur during aging or under extreme storage conditions. Potential volatiles may also be referred to as residual volatiles.

3.1.6 *total volatiles*—sum of the processing volatiles and the potential volatiles.

4. Summary of Test Methods

4.1 A designated quantity of material is weighed before and after a cure step that simulates normal industrial processing. The test specimen is weighed again after heating at 110 ± 5°C for 60 min. The percent volatile is calculated from the losses in weight.

5. Significance and Use

5.1 These test methods are the procedures of choice for determining volatile content of materials designed to be cured by exposure to ultraviolet light or electron beam irradiation. These types of materials contain liquid reactants that react to become part of the film during cure, but, which under the test conditions of Test Method D 2369, will be erroneously measured as volatiles. The conditions of these test methods are similar to Test Method D 2369 with the inclusion of a step to cure the material prior to weight loss determination. Volatile content is determined as two separate components—processing volatiles and potential volatiles. Processing volatiles is a measure of volatile loss during the actual cure process.

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.55 on Factory Applied Coatings on Preformed Products.

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[2] *Annual Book of ASTM Standards*, Vol 06.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

Potential volatiles is a measure of volatile loss that might occur during aging or under extreme storage conditions. These volatile content measurements are useful to the producer and user of a material and to environmental interests for determining emissions.

6. Interferences

6.1 The degree to which the results of these procedures accurately measure the volatiles emitted during actual use is absolutely dependent upon proper cure during the test procedure. Although overcure will have little or no effect upon measured volatiles, undercure may lead to erroneously high values. Since various pieces of cure equipment may vary widely in efficiency, it is essential that dialogue between material manufacturer and testing laboratory establish a cure schedule appropriate both to the material to be tested and to the cure equipment to be used in the procedure.

TEST METHOD A

7. Scope

7.1 This test method is applicable to radiation curable materials with solvent content less than or equal to 3 %.

8. Apparatus

8.1 *Aluminum Substrate*, standard test panels (102 mm by 305 mm) or heavy gage (0.05 mm minimum) foil. Test panels are most convenient and may be cut into smaller pieces for ease of weighing. Precondition the substrate for 30 min at 110 ± 5°C and store in a desiccator prior to use.

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

8.3 *Ultraviolet Light or Electron Beam Curing Equipment*—There are several commercial suppliers of laboratory scale equipment that simulates industrial curing processes.[4]

9. Procedure

9.1 Mix the sample, if necessary, to ensure uniformity. Hand stirring is recommended to avoid the entrapment of air bubbles.

9.2 Weigh the preconditioned aluminum substrate, (8.1) to 0.1 mg (*A*). The size of the aluminum substrate must allow a minimum of 0.2 g of material to be applied at the supplier's recommended film thickness. Use rubber gloves or tongs, or both, to handle samples.

9.3 Apply a minimum of 0.2 g of test specimen to the aluminum substrate and reweigh to 0.1 mg (*B*). Prepare a total of three test specimens.

NOTE 1—The elapsed time between application and weighing should be no greater than 30 s. If the sample to be tested contains any reactive diluent with a vapor pressure at room temperature greater than 1.0 mm Hg (for example, styrene), the elapsed time between specimen application and weighing must be no greater than 15 s.

9.4 Cure the test specimen by exposure to UV or EB as prescribed by the supplier of the material.

NOTE 2—If there is any doubt as to the adequacy of the exposure for affecting proper cure (6.1), an additional sample can be tested utilizing 50 % additional exposure and the volatile content results compared. If the original exposure was adequate, there should be no difference in the results within the precision of the test method. If the results are different, the supplier of the material must be contacted and a revised cure schedule established.

9.5 Allow the test specimen to cool 15 min at room temperature and reweigh to 0.1 mg (*C*).

9.6 Heat the test specimen in a forced draft oven (8.2) for 60 min at 110 ± 5°C.

NOTE 3—Materials that can react with atmospheric moisture during post cure, that is, UV cationic-curable epoxy materials, may exhibit a weight gain during procedure in 9.6. If this occurs, the sample should be retested and allowed to post cure at room temperature for 48 h after procedure in 9.5, and then reweighed prior to procedure in 9.6. The weight after post cure should then be used as Weight *C* in the calculation of percent potential volatiles in 10.1.

9.7 Allow the test specimen to cool to room temperature in a desiccator and reweigh to 0.1 mg, (*D*).

10. Calculations

10.1 Calculate the weight percent volatiles as follows:

$$\text{Processing Volatiles} = 100\,[(B - C)/(B - A)] \quad (1)$$

$$\text{Potential Volatiles} = 100\,[(C - D)/(B - A)] \quad (2)$$

$$\text{Total volatiles} = \%\ \text{Processing Volatiles} + \%\ \text{Potential Volatiles}$$

where:

- A = weight of aluminum substrate, g,
- B = weight of aluminum substrate plus test specimen, g,
- C = weight of aluminum substrate plus test specimen after cure, g, and
- D = weight of aluminum substrate plus cured test specimen after heating.

11. Precision and Bias

11.1 *Interlaboratory Test Program*—An interlaboratory study[5] of volatile content of radiation cured materials (Test Method A) was conducted in accordance with Practice E 691 in nine laboratories with three materials, with each laboratory obtaining three test results for each material.

11.2 *Test Result*—The precision information given below for volatile content in weight percent is for the comparison of two test results, each of which is the average of three test determinations.

11.3 *Precision*:

	Percent
Processing Volatiles	
95 % repeatability limit (within laboratory)	0.9
95 % reproducibility limit (between laboratories)	1.6
Potential Volatiles	
95 % repeatability limit (within laboratory)	2.2
95 % reproducibility limit (between laboratories)	4.2

[4] A list of such suppliers may be obtained by contacting RadTech International N.A., 60 Revere Drive, Suite 500, Northbrook, IL 60062.

[5] *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.

Total Volatiles	
95 % repeatability limit (within laboratory)	2.3
95 % reproducibility limit (between laboratories)	3.9

The terms repeatability limit and reproducibility limit are used as specified in Practice E 177. The respective standard deviations among test results may be obtained by dividing the limit values by 2.8. The form of this precision statement is in accordance with Practice E 177, 31.1.

11.4 *Bias*—Since there is no accepted reference material or method, or laboratory suitable for determining the bias for the procedure in this test method for measuring the volatile content of radiation cured materials, no statement of bias is being made.

TEST METHOD B

12. Scope

12.1 This test method is applicable to all radiation curable materials that will cure properly at the designated specimen weight, which corresponds to a film thickness of 50 to 75 µm depending upon solvent content. Test Method B is the method of choice for all radiation curable materials with solvent content greater than 3 %.

12.2 This test method is not applicable to materials containing styrene due to its volatility at 50°C.

13. Apparatus

13.1 *Aluminum Foil Dish*, 58 mm in diameter by 18 mm in height 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.

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

NOTE 4—The shelves of the oven must be level.

13.3 *Syringe*, 1 mL, capable of properly dispensing the material under test at sufficient rate that the specimen can be dissolved in the solvent. Disposable syringes are recommended.

13.4 *Ultraviolet Light or Electron Beam Curing Equipment*—There are several commercial suppliers of laboratory scale equipment that simulates industrial curing processes.[5]

14. Reagents

14.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform 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.

15. Procedure

15.1 Mix the sample, if necessary, to ensure uniformity. Hand stirring is recommended to avoid the entrapment of air bubbles.

15.2 Weigh a preconditioned aluminum dish (13.1) to 0.1 mg (*A*). Use rubber gloves or tongs, or both, to handle sample dishes.

15.3 Using the syringe (see 13.3) weigh to 0.1 mg (*B*), by difference, 0.3 ± 0.1 g of test specimen into the foil dish to which has been added 3 ± 1 mL of acetone. Add the material dropwise, swirling the dish to disperse it completely in the acetone. If the material forms a lump that cannot be dispersed, discard the test specimen and prepare a new one. Prepare a total of three samples.

NOTE 5—Be sure to wipe the outer surface of the syringe clean after obtaining the test specimen. Pull the syringe plunger up ¼ of an inch to pull the material away from the neck of the syringe. Cap and weigh the syringe. After dispensing the test specimen, do not wipe the tip of the syringe. Remove the material from the neck of the syringe by pulling up the plunger. Cap and reweigh the syringe. Note that sample weight (*B*) equals initial weight syringe minus final weight syringe.

NOTE 6—Use disposable rubber gloves or polyethylene to handle the syringe.

NOTE 7—If the material is not compatible with acetone, tetrahydrofuran (THF) or a blend of acetone and THF may be substituted.

15.4 Heat the samples in the forced draft oven (see 13.2) for 30 min at 50 ± 2°C.

NOTE 8—This step is critical since a large amount of solvent present in the sample during cure will interfere with the cure process and an inadequate degree of cure may result, which could produce erroneous volatile results (see 6.1). If the material contains only very fast solvents, a lower temperature/shorter time may be substituted if it can be demonstrated that the conditions are adequate to remove at least 90 % of the original solvent in the composition. Any remaining solvent will be removed during the subsequent cure and heating steps. In the case of samples that contain volatile solvents for control of application viscosity, this step also simulates the industrial processing stage necessary to remove the solvent prior to cure.

15.5 Cure the test specimen by exposure to UV or EB as prescribed by the supplier of the material (see Note 2).

15.6 Allow the test specimen to cool for 5 min at room temperature and reweigh (*C*).

15.7 Heat the test specimen in the forced draft oven (see 13.2) for 60 min at 110 ± 5°C. (**Warning—**In addition to other precautions, provide adequate ventilation, consistent with accepted laboratory practice, to prevent solvent vapors from accumulating to a dangerous level.)

15.8 Allow test specimen to cool to room temperature in a desiccator and reweigh (*D*).

16. Calculations

16.1 Calculate the weigh percent volatiles as follows:

$$\text{Processing volatiles} = 100\,[(B - (C - A)]/B \quad (3)$$

$$\text{Potential volatiles} = 100\,[(C - D)/B] \quad (4)$$

$$\text{Total Volatiles} = \%\ \text{Processing Volatiles} + \%\ \text{Potential Volatiles}$$

where:

- A = weight of aluminum dish, g,
- B = weight of test specimen, g,
- C = weight of aluminum dish plus test specimen after initial heating and cure, g and
- D = weight of aluminum dish plus cured test specimen after final heating, g.

[6] Supporting data are available from ASTM International Headquarters. Request RR:D01-1083.

17. Precision and Bias [6]

17.1 *Interlaboratory Test Program*—An interlaboratory study of volatile content of radiation cured materials (Test Method B) was conducted in accordance with Practice E 691 in eleven laboratories with three materials, with each laboratory obtaining three test results for each material.

17.2 *Test Result*—The precision information given in 17.3 for volatile content in weight percent is for the comparison of two test results, each of which is the average of three test determinations.

17.3 *Precision*:

	Percent
Processing Volatiles	
95 % repeatability limit (within laboratory)	2.0
95 % reproducibility limit (between laboratories)	3.4
Potential Volatiles	
95 % repeatability limit (within laboratory)	1.1
95 % reproducibility limit (between laboratories)	4.7
Total Volatiles	
95 % repeatability limit (within laboratory)	2.0
95 % reproducibility limit (between laboratories)	5.1

The terms repeatability limit and reproducibility limit are used as specified in Practice E 177. The respective standard deviations among test results may be obtained by dividing the limit values by 2.8. The form of this precision statement is in accordance with Practice E 177, 31.1.

17.4 *Bias*—Since there is no accepted reference material, method, or laboratory for determining the bias for the procedure in this test method for measuring volatile content of radiation cured materials, no statement on bias is being made.

18. Keywords

18.1 electron beam curing; radiation curable material; radiation curing; ultraviolet curing; volatile content