



Designation: D 5403 – 93 (Reapproved 2002)

Standard Test Methods for Volatile Content of Radiation Curable Materials¹

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 Coatings²
- E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens³
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Methods³
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

3. Terminology

3.1 Definitions:

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.

Current edition approved May 15, 1993. Published July 1993.

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

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

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 $110 \pm 5^\circ\text{C}$ 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.

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 study⁵ 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.