



Designation: D6266 – 00a (Reapproved 2023)

Standard Test Method for Determining the Amount of Volatile Organic Compound (VOC) Released From Waterborne Automotive Coatings and Available for Removal in a VOC Control Device (Abatement)¹

This standard is issued under the fixed designation D6266; 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 This test method describes the determination of the amount of volatile organic compound (VOC) released from applied waterborne automotive coatings that is available for delivery to a VOC control device. The determination is accomplished by measuring the weight loss of a freshly coated test panel subject to evaporation or drying and by analysis of the VOC or water content in the coating.

1.2 This test method is applicable to the VOC released from application and baking operations after the paint has been applied in a simulation of a production process, or in an actual production facility.

1.3 Symbols and calculations from several other methods that determine VOC: Practice D3960, EPA 450/3-88-018 and EPA 450/3-84-019 have been incorporated into this test method. The majority of symbols and calculations used in this test method are unique because this test method deals uniquely with differences in weight of applied paint samples that have been subject to drying, curing or solvent addition.

NOTE 1—Training and knowledge of the product being evaluated are essential for obtaining meaningful data from this test method. It is recommended that several practice runs be performed, and the laboratories repeatability evaluated before performing this test on the test samples.

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

1.5 *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.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standard-*

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.

Current edition approved June 1, 2023. Published June 2023. Originally approved in 1998. Last previous edition approved in 2017 as D6266 – 00a (2017). DOI: 10.1520/D6266-00AR23.

ization 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

D343 Specification for 2-Ethoxyethyl Acetate (95% Grade) (Withdrawn 1980)³

D1186 Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base (Withdrawn 2006)³

D1193 Specification for Reagent Water

D1475 Test Method for Density of Liquid Coatings, Inks, and Related Products

D2369 Test Method for Volatile Content of Coatings

D2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings

D3960 Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings

D4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 U.S. EPA Standards:⁴

EPA 450/3-88-018 (Dated December, 1988) Environmental Protection Agency Protocol for Determining the Daily Volatile Organic Compound Emission Rate of Automobile and Light Duty Truck Topcoat Operations. This protocol

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>.

describes procedures for determining VOC emission reduction credit in abatement processes.

EPA Federal Reference Method 24 - (Ref. 40CFR, part 60, Appendix A) Determination of Volatile Matter Content, Water Content, Density, Volume Solids and Weight Solids, of Surface Coatings.

EPA 450/3-84-019 Procedures for Certifying Quantity of Organic Compound Emitted by Paint, Ink, and Other Coatings

5. Apparatus and Materials for the Analyses

NOTE 2—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 specification of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. The references to purity of water, unless otherwise indicated, shall be understood to mean Type II reagent grade water conforming to Specification D1193.

Sample Preparation:

5.1 *Thin Steel Panels*, with an area of 310 cm² (48 in.²) or smaller.

5.2 *Laboratory Balance*, with accuracy to 0.001 g (minimum).

5.3 *Laboratory Spray Booth*, for application of the test coating, with air flow representative of production conditions.

5.4 *Spray Application Equipment*, selected to represent that used in a production process or sufficiently similar that the equipment will produce comparable performance.

5.5 *Film Thickness Gage*, for measuring dry paint thickness on metal surfaces (see Test Methods D1186).

5.6 *Laboratory Force-Draft Bake Oven, Type-IIA or Type IIB*, as specified in Specification E145.

5.7 *Wide-Mouth Glass Jars*, with TFE-fluorocarbon-lined caps or polypropylene copolymer bottles, one for each sample.

5.8 *Smooth Aluminum Foil*, (grade may vary).

5.9 *Ultrasonic Cleaner unit*, with 0.95 L (1 qt) capacity minimum.

5.10 *Laboratory Scale Paint Shaker* (violent agitation) 3.8 L (1 gal) capacity.

5.11 *Wood tongue Depressor or Tweezers*, if desired to roll foil.

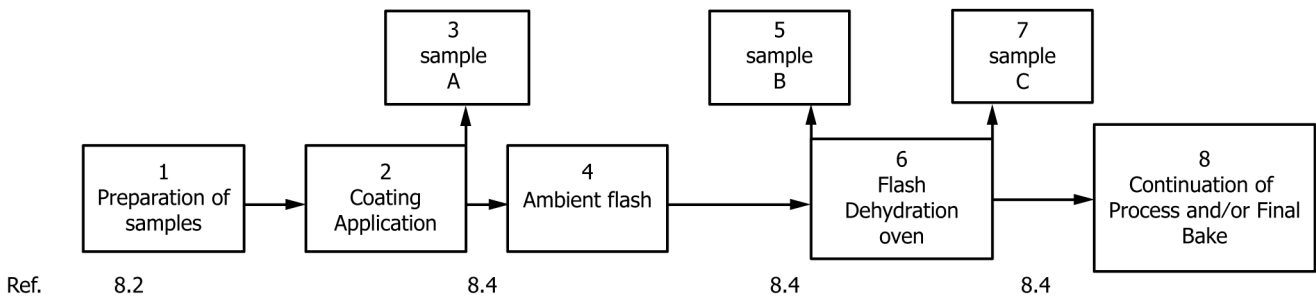
3. Summary of Test Method

3.1 This procedure measures the loss of VOC from a freshly coated surface by; (a) determining the difference in weight of a coated test panel before and after various steps in a process, (b) analyzing a sample of the applied coating for VOC or water content, or both, by gas chromatography (GC), or Karl Fisher, or both, before and after various steps in a process, and (c) calculating the VOC directly or after subtracting the water content. With these analyses, it is possible to relate the VOC loss to the volume of solids deposited on the test panel at each step of a process. The information obtained is used to determine the amount of VOC available for removal by the VOC control device at each step of a process (see Fig. 1).

4. Significance and Use

4.1 This test method provides basic engineering data that may be used to determine the amount of VOC delivered to the inlet of a VOC control device. The procedure is useful for establishing the quantity of VOC that is evolved from the coating in the flash zone or bake oven and available to be incinerated, although the same procedure can be followed when other forms of VOC abatement are used.

4.2 The total amount of VOC removed from the process by the VOC control device is a function of the amount available as given by this test method combined with the VOC removal efficiency of the control device.



Samples A, B, C include Foils for % NV, GC, and/or KF Analysis.

Karl Fisher (KF)	8.5
G. C. Analysis	8.6
% Non-Volatiles	8.7

FIG. 1 Test Panel Processing

Apparatus and Reagents for GC Analysis:

5.12 *Gas Chromatograph*, equipped with a flame ionization detector, electronic reporting integrator, capillary split injection port, and autosampler (where available).

5.13 *Chromatographic Syringe*, (10.0 µL).

5.14 *Analytical Balance*, accurate to 0.1 mg is needed, for this method.

5.15 *Sealable Vials*, (20 mL) scintillation.

5.16 *Medicine Droppers*.

5.17 *Analytical Column*—capillary, (30 meter) (0.25 mm) inside diameter,

5.17.1 film thickness, fused silica DB-5 or equivalent.

5.18 *Autosampler Vials*.

5.19 *Pipet, Volumetric*.

5.20 *Volumetric Flask*, for calibration standard and internal standard solutions.

5.21 *Bottles*, with good sealing caps for standard solutions.

5.22 *Solvents Standards*, expected to be found in the coating to be tested.

5.23 *Tetrahydrofuran (THF)*—HPLC grade, uninhibited.

5.24 *Cyclohexanol*—98 % or appropriate grade reagent.

5.25 *Water*.

5.26 *Acetone*—HPLC grade.

5.27 *Methanol*—HPLC grade.

5.28 *Dimethylformamide (DMF)*—HPLC grade.

5.29 *Chromatography Gases*—Helium of 99.9995 % purity or higher. Hydrogen of 99.9995 % minimum purity. Air, “dry” quality, free of hydrocarbons.

Apparatus and Reagents for KF Analysis:

5.30 *40 mL Volatile Organic Analysis (VOA) Vials*, with TFE-fluorocarbon lined caps.

5.31 *Methanol*—Low water grade (<0.008 % by K.F.)

5.32 *Karl Fisher Titrator*, or equivalent coulometric.

5.33 *Reagents*, appropriate for titrator.

5.34 *Associated Glassware*, for the tests (pipetes, volumetric flasks, etc).

5.35 *Water*, for calibration of the test instrument.

Apparatus and Reagents for Solids Density (Test Methods D2369, D1475, D2697; EPA Federal Reference Method 24):

5.36 *Syringe*, 5 mL.

5.37 *Weighing or Bottle* with eye dropper.

5.38 *Test Tube*, with new cork stopper.

5.39 *Aluminum Foil Dish*, 58 mm (2.3 in.) in diameter by 18 mm (0.71 in.) high with a smooth bottom surface.

5.40 *Laboratory Force-Draft Bake Oven Type IIA or Type-IIB*, as specified in Specification E145.

5.41 *Analytical Balance*, with accuracy to 0.1 mg.

5.42 *Toluene*, minimum technical grade,

5.43 *Ethoxyethyl Acetate*, minimum technical grade, Specification D343.

6. Coating Materials

6.1 The coating materials used are to be in the “as applied condition,” for example, representative of the specific formulation used in the coating process to be evaluated.

7. Conditions

7.1 Prior to beginning the test, determine the following conditions that represent the production process:

- (1) Dry film thickness,
- (2) Process sequence flash times,
- (3) Air flow,
- (4) Percent solids content after dehydration, and
- (5) Temperature and humidity conditions for each significant step of the process.

7.1.1 With the information obtained, establish test parameters that represent the range of conditions found in the plant. Specific application parameters need not duplicate exact production conditions as long as the above parameters are controlled for this test.

7.2 Identify all locations in the process sequence in which flash zone/oven effluent is vented directly to a VOC control device. The number of locations will affect the number of panel weight measurements taken and the number of panels that need to be tested.

8. Procedures

8.1 *Parameters to Evaluate and General Method to Collect Samples:*

8.1.1 Parameters to be evaluated are as follows:

- (1) Determination of water content by Karl Fisher Titration (KF) or
- (2) Determination of organic solvent content by gas chromatography (GC), or both, and
- (3) Determination of volatiles and nonvolatiles (% NV) during flash/baking operations.

8.2 *Use of Panels and Foil:*

8.2.1 For each location identified in 7.2, prepare test panels in duplicate as a minimum or as agreed upon between the involved parties.

NOTE 3—Thin steel panels 101.6 mm by 304.8 mm (4 in. by 12 in.) are preferred. If spray area is limited, smaller panels such as 101.6 mm by 152.4 mm (4 in. by 6 in.) can be used. Foils should be 13 mm (½ in.) larger in size than the area to be sprayed for easiness in handling.

8.2.2 Specified time at which the samples need to be collected. The following is suggested as a guideline throughout the rest of this procedure:

- (1) Immediately after paint application, (Sample A)
- (2) Entrance to Dehydration Ovens, (Sample B)
- (3) Exit of Dehydration Oven, (Sample C)

Where only the dehydration oven is exhausted to the VOC Control Device for example calculations in Section 11.

8.2.3 *Preparation of Samples:*

8.2.3.1 Dry and label sufficient sheets of foil ($i=1, \dots, n$) for each test ($A_i; B_i; C_i$) to constant weight to remove residual moisture.

8.2.3.2 Record each foil weight ($F_{Ai}; F_{Bi}; F_{Ci}$).

8.2.4 Wrap or secure foils on panels so some area (minimum of 1/2 in. (13 mm) per side) remains unpainted for the future handling.

8.2.5 Weigh jars and lids prior to spraying. Record the data. Record each jar and lid weight ($J_{Ai}; J_{Bi}; J_{Ci}$).

8.2.6 Prepare as a minimum one additional steel panel per spray out (for film thickness verification) to be sprayed with the foiled panels. All panels can be sprayed simultaneously. Alternatively, the spraying could be broken into families of various panels for each one of the requested tests for % NV, KF, and GC.

NOTE 4—“Trip blanks” are analyzed for all parameters of interest. Trip blanks are often prepared by the laboratory and submitted to the sampling team when bottle ware is delivered. The trip blank accompanies all of the project samples through all custody changes in possession, coolers, and refrigerators. Trip blanks are *not* opened by the sampling team. The trip blanks provide information with respect to contamination that is “picked-up” during sample packaging, shipping and storage.

NOTE 5—The “field blank” is a portion of the sampling matrix that is carried through the entire analytical scheme. The field blank is treated exactly as the actual sample is treated. For example, the field blank vial is opened and closed when the corresponding sample vial is opened and closed. It is important that the volume/weight of the field blanks be the same as that of the samples.

8.3 Spraying Samples:

8.3.1 Apply basecoat using an automated device (preferred for consistency) to target film build simulating assembly plant processing conditions.

8.4 Collection of Samples:

8.4.1 At the sampling points specified in 8.2.2, the foil samples for KF and GC analyses need to be placed in jars with a specified solvent (8.5 and 8.6). The samples for % NV just need to be weighed at the sampling point. Then, they are baked at final bake condition and re-weighed.

8.4.2 Roll up foil *paint side out* for KF (8.5) and GC (8.6).

8.4.3 Place coated foil (8.4.1) immediately into a jar of size depending upon foil size. Weigh jars, lids, and coated foils. Record weights ($K_{Ai}; K_{Bi}; K_{Ci}$) including field blanks (see 8.2.6).

8.4.4 Fold foils *paint side in* for % NV (8.7).

8.5 Water Content by KF:

8.5.1 Water Content Solvent Preparation:

8.5.1.1 Analyze a sample of the bulk methanol for water content by the Karl Fischer Method in accordance with Test Method D4017 to check purity of reagent.

8.5.1.2 Fill each jar (as described in 8.4.1.1) with 30 mL of methanol. Foil should be completely submerged in methanol.

8.5.1.3 Reweigh jars, lids, coated foils and methanol and record weights ($L_{Ai}; L_{Bi}; L_{Ci}$).

8.5.1.4 Place the capped jars containing methanol and foil strips into an ultrasonic cleaning unit until sample is uniformly dispersed into the methanol.

8.5.2 Sample Analysis:

8.5.2.1 Follow the procedure for KF Titration in accordance with Test Method D4017.

8.5.2.2 Fill a 5.0 mL disposable syringe with a well mixed representative portion of the methanol from one of the glass sample jars. Perform this step in a low humidity room or chamber.

8.5.2.3 Weigh and transfer enough of each sample into the Karl Fischer titration vessels so that at least 10 mL of KF reagent will be required to reach the endpoint. Repeat for each sample and all blank vials.

8.5.3 Calculation of % Water in the Paint Sample on a Wt./Wt. Basis:

8.5.3.1 Determine the weight of sample paint on the foil P_i as follows:

$$P_i = K_i - (F_i + J_i) \quad (1)$$

where:

i = foil i ,
 K_i = weight of jar, lid, foil and paint (4.1.1),
 F_i = weight of foil (8.2.3.2), and
 J_i = weight of jar and lid (8.2.5).

8.5.3.2 Determine the weight of methanol used for Sample M_i .

$$M_i = L_i - K_i \quad (2)$$

where:

i = foil i ,
 L_i = weight of jar, lid, foil, paint and methanol, and
 K_i = weight of jar, lid, foil and paint.

8.5.3.3 Calculate the % water (corrected for % water in the field blank) in the paint sample on a weight/weight basis as follows:

$$\% \text{ H}_2\text{O (wt./wt.)} = \quad (3)$$

$$\frac{[(\% \text{ water in sample}) \times (M_i + P_i) - (\% \text{ water in field blank}) (M_i)]}{P_i}$$

where:

i = foil i ,
 M_i = weight of methanol,
 P_i = weight of paint specimen, and
 $\% \text{ Water}$ = percent water from 8.5.2.

8.5.3.4 Calculate the % VOC in the paint sample on a weight/weight basis as follows:

$$\% \text{ VOC (wt./wt.)} = \% V - \% \text{ H}_2\text{O (wt./wt.)} \quad (4)$$

where:

$\% V$ = % volatiles as determined in 8.7.

8.6 Procedure for GC Analysis:

8.6.1 Chromatographic Conditions:

8.6.1.1 Set up the instrument according to manufacturer’s instructions using the following parameters: