



Designation: D 3960 – 04

Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings¹

This standard is issued under the fixed designation D 3960; 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 Department of Defense.

1. Scope

1.1 This practice measures the volatile organic compound (VOC) content of solventborne and waterborne paints and related coatings as determined from the quantity of material released from a sample under specified bake conditions and subtracting exempt volatile compounds and water if present.

NOTE 1—The regulatory definition, under the control of the U.S. EPA, can change. To ensure currency, contact the local air pollution control agency.

1.2 This practice provides a guide to the selection of appropriate ASTM test methods for the determination of VOC content.

1.3 Certain organic compounds that may be released under the specified bake conditions are not classified as VOC, as they do not participate in atmospheric photochemical reactions. Such nonphotochemically reactive compounds are referred to as exempt volatile compounds in this practice.

NOTE 2—A list of the current US EPA approved exempt volatile compounds is found in Appendix X3, paragraph X3.1.1.1.

1.4 VOC content is calculated as a function of (1) the volume of coating less water and exempt volatile compounds, and (2) the volume of coating solids, and (3) the weight of coating solids.

2. Referenced Documents

2.1 ASTM Standards:²

- D 1475 Test Method for Density of Liquid Coatings, Inks, and Related Products
- D 2369 Test Method for Volatile Content of Coatings

¹ This practice 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.

- D 2697 Test Method for Volume Nonvolatile Matter in Clear or Pigmented Coatings
- D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings
- D 3792 Test Method for Water Content of Coatings by Direct Injection into a Gas Chromatograph
- D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings
- D 4017 Test Method for Water in Paints and Paint Materials by Karl Fischer Method
- D 4457 Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph
- D 5095 Test Method for Determination of the Nonvolatile Content in Silanes, Siloxanes and Silane-Siloxane Blends Used in Masonry Water Repellent Treatments
- D 5201 Practice for Calculating Formulation Physical Constants of Paints and Coatings
- D 5403 Test Methods for Volatile Content of Radiation Curable Materials
- D 6093 Test Method for Percent Volume Nonvolatile Matter in Clear or Pigmented Coatings Using a Helium Gas Pycnometer
- D 6133 Test Method for Acetone *p*-Chlorobenzotrifluoride, Methyl Acetate or *t*-Butyl Acetate Content of Solvent-Reducible and Water-Reducible Paints, Coatings, Resins, and Raw Materials by Direct Injection into a Gas Chromatograph
- D 6419 Test Method for Volatile Content of Non-Heatset Web Offset Printing Inks
- D 6438 Test Method for Acetone, Methyl Acetate, and Parachlorobenzotrifluoride Content of Paints and Coatings by Solid Phase Microextraction-Gas Chromatography
- D 6886 Test Method for Speciation of the Volatile Organic Compounds (VOCs) in Low VOC Content Waterborne Air-Dry Coatings by Gas Chromatography
- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals

2.2 Other Documents:

EPA Federal Reference Method 24—Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings³

EPA 450/3-84-019, U.S. Environmental Protection Agency Procedures for Certifying Quantity of Organic Compound Emitted by Paint, Ink, and Other Coatings³

EPA 450/3-83-013R, U.S. Environmental Protection Agency Glossary for Air Pollution Control of Industrial Coatings Operations³

3. Terminology

3.1 Definitions:

3.1.1 *exempt volatile compound*—organic compounds that do not participate significantly in atmospheric photochemical reactions.

3.1.2 *nonvolatile material*—the solid material remaining after volatiles have been removed from a coating under specified test conditions.

3.1.3 *volatile organic compound (VOC)*—any organic compound that participates in atmospheric photochemical reactions.

3.1.3.1 *Discussion*—The definition of VOC adopted by the U.S. EPA and a list of exempt volatile compounds are included in Appendix X3.

3.1.4 *volatile organic compound content (VOC content)*—the mass of VOC released from a coating under specified test conditions.

3.1.4.1 *Discussion*—VOC content is expressed in this practice as a function of: (1) the coating volume less water and exempt volatile compounds, and (2) the volume of coating solids and (3) the weight of coating solids.

4. Summary of Practice

4.1 Take a representative sample of the liquid coating in accordance with Practice D 3925. Mix thoroughly before taking specimens for individual tests. If air bubbles become entrapped, stir by hand until the air has been removed.

4.2 The volatile content, density, water content, volume solids and exempt solvent content of the coating are determined in accordance with designated methods and instructions. For multicomponent coatings, the components are first mixed in the appropriate ratios and the applicable values determined on the mixture. These values are combined using specified equations to calculate the VOC content of the coating.

NOTE 3—In Reference Method 24, the U.S. EPA defines a waterborne coating as any coating with more than 5 % water by weight in its volatile fraction, and requires/allows water determination for waterborne coatings only.

5. Significance and Use

5.1 This practice discusses applicable ASTM test methods used in the determination of the VOC content of paints and related coatings and provides equations for calculating the VOC content expressed as the mass of VOC: (1) per unit

volume of coating less water and exempt volatile compounds, and (2) per unit volume of coating solids and (3) per unit mass of coating solids.

5.2 Volatile organic compound content is used to compare the amount of VOC released from different coatings used for the same application, that is, to coat the same area to the same dry film thickness (assuming the same application efficiency).

5.3 VOC content data are required by various regulatory agencies.

5.4 Only the expression of VOC content as a function of the volume of coating solids gives a linear measure of the difference in VOC released from different coatings used for the same application.

NOTE 4—Thus assuming the same transfer efficiency, a coating with VOC content of 3 lb of VOC/gal of solids would release ½ the VOC that would a coating with 6 lb of VOC/gal of solids.

5.5 When VOC content is expressed as a function of the volume of coating less water and exempt solvents, the values obtained do not account for differences in the volume solids content of the coatings being compared: this expression, therefore, does not provide a linear measure of the difference in VOC emitted from different coatings used for the same application.

NOTE 5—Thus, a coating with VOC content of 3 lb of VOC/gal less water and exempt volatile compounds would release about 85 % less VOC than a coating with 6 lb of VOC/gal less water and exempt volatile compounds.

6. Nonvolatile and Volatile Content

6.1 Guide D 2832 includes suggested time/temperature drying schedules for the determination of the nonvolatile and volatile content of various types of coatings.

NOTE 6—For regulatory compliance testing, follow the method and conditions specified in the applicable regulation. Federal Reference Method 24 specifies the use of Test Method D 2369.

6.2 Test Method D 2369 includes a specific drying schedule and sample weight, and heating 1 h at 110 ± 5°C for the determination of the weight percent volatile content of solvent-borne and waterborne coatings.

6.2.1 For multicomponent coatings, Test Method D 2369 specifies the components should be mixed first, then the volatile content should be determined on the mixture. Test specimens are held in the aluminum dish for at least 1 h before baking.

NOTE 7—Other induction periods are used. See U.S. EPA Reference Method 24.

6.2.2 The nonvolatile content of silanes, siloxanes, and silane/siloxane blends used in masonry water repellent treatments is defined using Test Method D 5095. In this standard, applicable to both solvent and water reducible materials, the test specimen, containing an added catalyst, is allowed to stand at room temperature for 1 h prior to heating in an oven at 110° ± 5°C for 60 min.

NOTE 8—In VOC determinations, for 6.2.2 only, the density and water content (if applicable) are measured on the test material without the added catalyst.

³ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

6.2.3 The nonvolatile content of radiation curable coatings, inks, and adhesives is defined using Test Methods D 5403. These materials contain volatile reactive components that become nonvolatile after radiation curing. 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. 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 that are intended to be removed from the material to cure.

6.2.4 The volatile content of sheet-fed and coldset web offset printing inks is defined using Test Method D 6419. This standard is based on Test Method D 2369, but has tighter controls for specimen weight and oven temperature. Interlaboratory studies have shown this necessary to improve the precision of test results for these inks. It also allows the use of ink knives or taper knives for transferring the specimen to the aluminum dish from the sample container.

6.2.5 Test Method D 6886 is a direct gas chromatographic method for the determination of individual and total volatile organic compounds in low VOC content waterborne latex air-dry coatings. This method is intended primarily for analysis of waterborne coatings in which the material VOC content is below 5 weight percent. It is the only method for the speciation of VOCs in low VOC content waterborne latex air-dry coatings. This method provides a direct determination of weight percent VOC content, in contrast to the indirect determination (total volatiles minus water) of weight percent VOC of waterborne coatings presented in Practice D 3960.

7. Water Content

7.1 To determine the water content of coatings two test methods are available:

7.1.1 In Test Method D 3792, a paint specimen is diluted with dimethyl formamide, an internal standard (2-propanol) is added, and an aliquot of the mixture is injected directly into a gas chromatograph.

7.1.2 Test Method D 4017 offers three options for determining water content by Karl Fischer titration.

7.1.2.1 A specimen is dissolved in pyridine or another suitable solvent and titrated in the presence of a buffer, 1-ethyl piperidine. The use of newer non-pyridine titration reagents is also allowed.

7.1.2.2 The water in a latex paint is first extracted into anhydrous methanol, then an aliquot of the methanol extract is titrated with non-pyridine reagent in methanol solvent (see Appendix X1 of Test Method D 4017).

7.1.2.3 The specimen is dispersed in methanol solvent using a homogenizer accessory, then directly titrated with non-pyridine reagent (see Appendix X2 of Test Method D 4017).

7.1.3 With multicomponent coatings, the components are first mixed in the appropriate ratios, then water content is determined using Test Methods D 3792 or D 4017.

8. Density

8.1 The density of the paint or coating at 25°C is determined in accordance with Test Method D 1475. Although both the

pycnometer and weight-per-gallon cup are covered by the test method, and the former is more accurate and precise, the weight-per-gallon cup is recommended because of its speed and ease of use.

8.2 With multicomponent coatings, first mix the components in appropriate ratios in sufficient quantity to determine the weight-per-gallon using Test Method D 1475.

9. Exempt Volatile Compounds

9.1 In Test Method D 4457 an internal standard (1-propanol) is added to the test specimen, and then the specimen is injected directly into a gas chromatograph.

9.2 In Test Method D 6133 an internal standard is added to the whole paint, and the mixture injected directly into the gas chromatograph.

9.3 In Test Method D 6438 an internal standard is added to whole paint, followed by solid phase microextraction (SPME) headspace sampling and subsequent injection into a gas chromatograph.

9.4 With multicomponent coatings, the exempt volatile compound content is determined on the mixture of the components.

10. Calculation of VOC Content

10.1 In this practice VOC content is expressed in three ways: (1) as the mass of VOC per unit volume of the coating less water and exempt volatile compounds, (2) as the mass of VOC per unit volume of coating solids, and (3) the mass of VOC per unit mass of coatings solids. The following equations should be used to calculate VOC content and may be used for coatings both “as supplied” and “as applied” (see Note 8).

NOTE 9—For compliance with VOC regulations, the VOC content should be calculated after any thinning or dilution (“as applied”). Instructions for VOC calculations of such diluted coatings are available in EPA 450/3-84-019.

10.2 *VOC Content Expressed as the Mass of VOC per Unit Volume of Coating Less Water and Exempt Volatile Compounds:*

10.2.1 *General Expression:*

$$VOC = \frac{[(\text{weight percent of total volatiles less water less exempt volatile compounds})(\text{density of coating})]}{[100 \% - (\text{volume percent of water}) - (\text{volume percent of exempt volatile compounds})]} \quad (1)$$

or

$$VOC = \frac{(W_o)(D_c)}{100 \% - V_w - V_{ex}} \quad (2)$$

$$= \frac{(W_v - W_w - W_{ex})(D_c)}{100 \% - (W_w)(D_c/D_w) - (W_{ex})(D_c/D_{ex})}$$

where:

VOC = VOC content in g/L of coating less water and exempt volatile compound (see Note 9),

W_o = weight of organic volatiles, % ($W_v - W_w - W_{ex}$),

W_v = weight of total volatiles, % (100 % - weight % nonvolatiles, see Test Method D 2369),

W_w = weight of water, % (see Test Methods D 3792 or D 4017),