



Designation: D 6803 – 02

# Standard Practice for Testing and Sampling of Volatile Organic Compounds (Including Carbonyl Compounds) Emitted from Paint Using Small Environmental Chambers<sup>1</sup>

This standard is issued under the fixed designation D 6803; 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 approval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice provides procedures for preparing test samples of alkyd primer, alkyd paint, latex primer, or latex paint applied to building materials such as gypsum wallboard, wood, or engineered wood products and procedures for sampling volatile organic compounds (VOCs) emitted from those test samples. Emissions are sampled from small environmental chambers operated under controlled conditions.

1.2 This practice describes procedures for preparation of test specimens by application of primer or paint to common building materials. Use of the procedures described in this practice for tests with other application methods or substrates may affect the results and not meet the criteria recommended in the practice.

1.3 This practice describes procedures for collection of VOCs on sorbent tubes and carbonyl compounds on silica gel treated with 2,4-dinitrophenylhydrazine (DNPH) that require analytical methods for measurement of individual organic compound concentrations. This practice does not describe the detailed procedures of analytical methods, but refers to published methods for these analyses.

1.4 This practice describes procedures for testing and sampling VOCs emitted from paint under controlled conditions. The test conditions, when combined with analytical data, can be used to calculate emission rates. This practice does not recommend a method for the calculations.

1.5 Values stated in the International System of Units (SI) are to be regarded as the standard.

1.6 *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.*

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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## 2. Referenced Documents

### 2.1 *ASTM Standards:*<sup>2</sup>

**D 16** Terminology for Paint, Related Coatings, Materials, and Applications

**D 1005** Test Method for Measurements of Dry Film Thickness of Organic Coatings

**D 1212** Test Method for Measurement of Wet Film Thickness of Organic Coatings Using Micrometers

**D 1356** Terminology Relating to Sampling and Analysis of Atmospheres

**D 1914** Practice for Conversion Units and Factors Relating to Sampling and Analysis of Atmospheres

**D 3686** Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)

**D 3687** Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method

**D 5116** Guide for Small Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products

**D 5197** Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air (Active Sampler Methodology)

**D 6196** Practice for Selection of Sorbents and Pumped Sampling/Thermal Desorption Analyses Procedures for Volatile Organic Compounds in Air

**D 6345** Guide for Selection of Methods for Active, Integrative Sampling of Volatile Organic Compounds in Air

**E 741** Test Method for Determining Change in a Single Zone by Means of Tracer Gas Dilution

### 2.2 *Other Referenced Document:*<sup>3</sup>

<sup>2</sup> 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.

<sup>3</sup> U.S. EPA, Center for Environmental Research Information, Cincinnati, OH, website <http://www.epa.gov/ttn/amtic/airtox.html>.

**Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air**, Compendium Methods TO-15 and TO-17, EPA/625/R-96-010b, January 1999, (NTIS No. PB99-172355)

### 3. Terminology

3.1 *Definitions*—For definitions and terms used in this practice, refer to Terminology **D 1356**, Terminology **D 16**, and Practice **E 355**. For definitions and terms related to test methods using small-scale environmental chambers, refer to Guide **D 5116**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air change per hour (ACH)*—the volume of clean air brought into the chamber in 1 h divided by the chamber volume measured in identical volume units, normally expressed in air changes per hour ( $\text{h}^{-1}$ ).

3.2.2 *alkyd paint*—also referred to as oil paint, it is a paint that contains drying oil or oil varnish as the basic vehicle ingredient.

3.2.3 *chamber loading ( $\text{m}^2/\text{m}^3$ )*—the exposed surface area of the test specimen coated with paint divided by the environmental test chamber volume.

3.2.4 *clean air*—air that does not contain any individual VOC at a concentration in excess of  $2 \mu\text{g}/\text{m}^3$  and does not contain greater than  $10 \mu\text{g}/\text{m}^3$  for the sum of the VOCs measurable in the sample. The air should be conditioned to remove particulates and ozone.

3.2.5 *environmental enclosure*—a temperature controlled enclosure of sufficient size to contain the environmental test chamber(s) and allow adequate access to it to conduct the testing.

3.2.6 *environmental test chamber*—a chamber constructed of inert materials into which a material can be placed and tested to determine the VOC emission rate under controlled environmental conditions.

3.2.7 *latex paint*—a paint containing a stable aqueous dispersion of synthetic resin, produced by emulsion polymerization, as the principal constituent of the binder. Modifying resins may be present.

3.2.8 *primer*—the first of two or more coats of a paint.

3.2.9 *spreading rate*—the area covered by a unit volume of coating material, frequently expressed as square feet per gallon. It may also be referred to as *coverage* or *coverage rate* on paint container labels.

3.2.10 *test specimen*—a specimen of the paint applied to a substrate such as gypsum wallboard, wood, or engineered wood products.

3.2.11 *volatile organic compound (VOC)*—an organic compound with saturation vapor pressure greater than  $10^{-2}$  kPa at  $25^\circ\text{C}$ .

### 4. Summary of Practice

4.1 This practice describes procedures for testing and sampling emissions of VOCs, including formaldehyde and other carbonyl compounds, from paint applied to building materials such as gypsum wallboard, wood, and engineered wood products. Emissions tests are conducted using small environmental chambers operated in a dynamic mode with continuous flow of humidified VOC-free air through the chambers. The

environmental chambers are operated at designated conditions of airflow rate, temperature, and relative humidity. The VOCs in the emissions are sampled by adsorption on an appropriate single, or multiple sorbent media that can be analyzed by thermal desorption and combined gas chromatography/mass spectrometry (GC/MS) or GC/flame ionization detection (GC/FID). Formaldehyde and other carbonyl compounds are collected on silica gel coated with DNPH reagent that can be analyzed by high performance liquid chromatography (HPLC).

4.2 This practice describes the procedures for handling and storage of paint, setup of the small environmental test chambers, preparation of test specimens, chamber performance tests, sampling and reporting.

### 5. Significance and Use

5.1 Latex and alkyd paints are used as coatings for walls, wooden trim, and furnishings in occupied buildings. Paint may be applied to large surface areas and may be applied repeatedly during the lifetime of a building. VOCs are emitted from paint after application to surfaces.

5.2 There is a need for data on emissions from paint. The data can be used to compare emissions from different products. The data may be used to assist manufacturers in reducing or eliminating VOC emissions from their products. The data may be used to predict concentrations of VOCs in a room or building when used with appropriate indoor air quality models.

5.3 Standard test practices and procedures are needed for the comparison of emissions data from different laboratories.

### 6. Apparatus

6.1 This practice requires the use of an environmental chamber testing facility and air sample collection systems.

6.2 *Environmental Chamber Testing Facility*, consisting of an environmental test chamber, a controlled-temperature environmental enclosure, a system for supplying clean and conditioned air to the chamber, and fittings and manifolds on the chamber outlet for collection of air samples. All materials and components in contact with the test specimen or air prior to sample collection should be chemically inert and accessible for cleaning. Suitable materials include stainless steel and glass. All gaskets and flexible components should be made from chemically inert materials. General guidance for design, construction, configuration, and validation of an environmental test chamber facility is provided in Guide **D 5116**.

6.2.1 *Environmental Test Chamber*, constructed of inert materials of sufficient size to hold the test specimen. Small environmental test chambers may range in size from a few litres to  $5 \text{ m}^3$ . Procedures recommended in this practice have been evaluated using environmental chambers with a volume of  $0.053 \text{ m}^3$ . This volume is used in the practice for discussion and illustrative purposes. Chambers of different size and shape may be used if the standard environmental test chamber conditions can be maintained and chamber performance can be demonstrated. The chamber should be equipped with an opening large enough for loading the test specimen and for cleaning the chamber. The chamber will be equipped with a port to supply air to the chamber, an air outlet from the chamber, and ports for temperature and relative humidity probes. The chamber may be equipped with a fan to promote

mixing in the chamber and to achieve the desired air velocity across the surface of the test specimen. The performance of the chamber should be evaluated prior to use to determine airtightness, surface adsorption effects, air mixing, and air velocity at the surface of the substrate. Some of the chamber performance (that is, air mixing and air velocity) should be evaluated with an uncoated test substrate in place. The chamber performance should be tested and demonstrated following the guidelines presented in Guide **D 5116**.

6.2.2 *Environmental Enclosure*, of sufficient size to accommodate the environmental test chamber and capable of maintaining the desired temperature within 0.5°C.

6.2.3 *Clean Air Supply System*, capable of supplying a controlled flow of clean, humidified air into the test chamber, as described in Guide **D 5116**. The system should incorporate hardware for removing particles, ozone, and VOCs from the air supplied to the chamber(s). Concentrations of VOCs and aldehydes measured at the chamber inlet should not exceed 2 µg/m<sup>3</sup> for any single compound or 10 µg/m<sup>3</sup> for the sum of all measurable VOCs in the sample. The relative humidity (RH) of the air supplied to the chamber(s) should be controlled to the desired set point within ±5 % RH. The flow rate of the air supplied to the chamber should be controlled within ±5 % of the set-point airflow rate. Ideally, the chamber system will be designed such that a positive pressurization of the chamber of approximately 10 Pa relative to the environmental enclosure will be maintained and monitored at all times during the test.

6.2.4 *Environmental Measurement System*, consisting of hardware and software to measure and record the temperature, RH, and airflow rates during operation of the test system. A system for continuous recording of the data is recommended.

6.3 *Air Sampling Systems*, consisting of sorbent tubes and DNPH-silica gel cartridges, a sampling manifold, vacuum pumps, and airflow controllers/meters. Airflow controllers should control the airflow rate through the sampling system to within 5 % of the specified value. All system components between the chamber and the sampling media should be constructed of chemically inert materials.

6.3.1 A glass or stainless steel manifold should be connected to the outlet of the chamber for collection of air samples. The manifold should be designed for collection of multiple samples simultaneously. The exhaust from the manifold should be vented into a laboratory fume hood or other appropriate exhaust device to prevent contamination of the air in the laboratory or environmental enclosure.

6.3.2 Vacuum pumps should be used to draw air through the sorbent tubes. The required airflow rate is a function of the type of sampler used, the size of the chamber, and the air change rate. The total airflow rate through the samplers should not exceed 50 % of the flow rate from the chamber outlet. For collection of VOCs on sorbent tubes, the pump should be capable of maintaining a constant flow in the range of 10 to 200 mL/min. For collection of air samples on DNPH-silica gel cartridges, the pump should be capable of maintaining a constant flow in the range of 100 to 500 mL/min.

6.3.3 For collection of VOCs during the emissions test, tubes containing single or multiple sorbents may be used. The sorbents may be porous polymers or graphitized carbon blacks.

Select an appropriate single or multi-layered sorbent tube following the procedures in Practice **D 6196**, Guide **D 6345**, and EPA Method TO-17. Recommendations on the use of sorbent tubes from manufacturers or suppliers should be followed in selecting the sampling airflow rate and sampling period to avoid breakthrough of VOCs through the sorbent tube. The required air sampling volume at each collection time point should be determined through consideration of the safe sampling volume (SSV, see Practice **D 6196**) of the VOC with the lowest retention volume, concentrations to be measured, and detection limits of the analytical method.

6.3.4 For collection of VOCs during the first 10 to 20 h following application of alkyd primer or paint, charcoal sorbents (Practice **D 3687**) may be used due to the high concentrations of VOCs in the chamber air.

6.3.5 For collection of formaldehyde and other carbonyl compounds, DNPH-silica gel cartridges should be used following the Test Method **D 5197**.

6.3.6 An airflow meter/controller should be used to control and measure the airflow rate during sample collection. The controller may consist of a precision flow control valve, a critical orifice, or a mass flow controller. The measurement device may consist of soap film bubble meter, calibrated high precision rotameter, or mass flow meter. A mass flow meter/controller is recommended for use during sample collection. All flow measurements should be referenced to standard temperature and pressure.

## 7. Procedures for Paint Selection, Handling, and Storage

7.1 Procedures for selection of the paint to be tested are a function of the objectives of the tests. Paint may be procured from clients, manufacturers, distributors, or retailers. Record pertinent information upon receipt of the paint including date of acquisition, source of the paint, manufacturer, container size, lot number, and other relevant information on the label. Obtain and review the Material Safety Data Sheet (MSDS) for the paint. At least two containers of the same lot number of paint should be procured (one for testing and one to archive).

7.2 Upon receipt of the paint, it should be split into storage vials for handling and testing. The paint should be mixed in the original container on a paint shaker before the split.

7.3 Split the paint into aliquots. Special care should be taken to minimize the loss of volatile compounds during the process. Paint containers should not be left open except when required for transfer to storage vials. The size of the aliquot and storage vial is a function of the amount of paint required for the test. Vials of 40 to 60 mL volumes hold sufficient paint for GC/MS analyses of the liquid product or preparation of test specimens of 256 cm<sup>2</sup> area for chamber tests. Store paint in clean amber glass vials that can be sealed with caps that have Teflon liners. Clean vials with alkaline detergent, rinse thoroughly with deionized water, then dry before use. Individual vials of paint are used for testing to minimize losses of volatile compounds during handling and preparation of test specimens. Repeated opening of a large container of paint will result in losses of VOCs.

7.4 Pour the mixed paint into the vials, filling the vial to near the top to minimize the volume of headspace and loss of VOCs when the vial is opened. Two or more clean stainless