



# 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 reapproval. A superscript epsilon ( $\epsilon$ ) 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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 16 Terminology for Paint, Related Coatings, Materials, and Applications<sup>2</sup>
- D 1005 Test Method for Measurements of Dry Film Thickness of Organic Coatings<sup>2</sup>
- D 1212 Test Method for Measurement of Wet Film Thickness of Organic Coatings Using Micrometers<sup>2</sup>
- D 1356 Terminology Relating to Sampling and Analysis of Atmospheres<sup>3</sup>
- D 1914 Practice for Conversion Units and Factors Relating to Sampling and Analysis of Atmospheres<sup>3</sup>
- D 3686 Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)<sup>3</sup>
- D 3687 Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption Method<sup>3</sup>
- D 5116 Guide for Small Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products<sup>3</sup>
- D 5197 Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air (Active Sampler Methodology)<sup>3</sup>
- D 6196 Practice for Selection of Sorbents and Pumped Sampling/Thermal Desorption Analyses Procedures for Volatile Organic Compounds in Air<sup>3</sup>
- D 6345 Guide for Selection of Methods for Active, Integrative Sampling of Volatile Organic Compounds in Air<sup>3</sup>
- E 741 Test Method for Determining Change in a Single Zone by Means of Tracer Gas Dilution<sup>4</sup>

### 2.2 Other Referenced Document:<sup>5</sup>

Compendium of Methods for the Determination of Toxic

<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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 04.11.

<sup>5</sup> U.S. EPA, Center for Environmental Research Information, Cincinnati, OH, website <http://www.epa.gov/ttn/amtic/airtox.html>.

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 steel balls may be placed in the vial to aid in mixing prior to



use of the sample. Prepare a sufficient number of sample vials for all analyses and tests planned for the paint. Label the vials individually with a sample code and the date of preparation.

7.5 Store the vials of the paint samples and the original containers of the paint in the dark at room temperature.

7.6 Use the paint sample within the manufacturer's specified shelf-life time. VOC concentrations should be measured by GC/MS or GC/FID after extended periods of storage to verify that they have not changed during storage. Compare the concentrations of the VOCs to results from the original analyses performed immediately after the paint was obtained.

## 8. Procedures for Preparation of Test Specimens

8.1 Procure the substrate material that is appropriate for testing the emissions from the paint. The substrate should not emit any of the compounds that are to be quantified in the emissions from the paint or compounds that may interfere with quantification of the emissions from the paint. Cut the substrate material to an appropriate size (for example, 16 cm by 16 cm for a loading of 0.5 m<sup>2</sup>/m<sup>3</sup> in a 0.053 m<sup>3</sup> chamber). Use an appropriate cutting device to obtain smooth edges. If necessary, sand wood substrate to provide a smooth surface.

8.2 To minimize emissions from the cut edges, seal the edges. Techniques that can be used to seal the edges include: (1) coating the edges with technical grade liquid sodium silicate, (2) wrapping with aluminum foil, or (3) mounting the substrate in a stainless steel frame that covers the exposed edges.

8.3 The substrate material should be properly stored to remain clean, dry, and structurally sound.

## 9. Procedure for Paint Application

9.1 Prior to application of paint for emissions testing, one must perform practice applications using the same type of applicator (for example, a roller or brush) and substrate in order to develop a technique that will produce uniform and repeatable applications. The VOC emission characteristics will be influenced by the thickness of the paint applied, and care should be taken to ensure uniform paint application. If desired, wet and dry film thickness of paint applied can be determined by ASTM methods referenced in Section 2 to verify the uniformity of the application. The application should also be performed as quickly as possible to minimize VOC losses during preparation of the test specimen.

### 9.2 Paint Application Procedure:

9.2.1 Determine the target mass of paint to be applied from product data sheets or the spreading rate (ft<sup>2</sup>/gal) listed on the container label. Calculate the mass of paint needed using the spreading rate (ft<sup>2</sup>/gal) and the density of the paint. The density is generally listed on product data sheets or the MSDS. The mass to be applied can be calculated as follows:

$$M_p = \left( \frac{1}{SR} \right) \cdot (D_p) \cdot (A) \quad (1)$$

where:

$M_p$  = target mass of paint to be applied, g,

$SR$  = spreading rate, cm<sup>2</sup>/mL, generally listed on container or data sheet as ft<sup>2</sup>/gal,

$D_p$  = density of the paint, g/mL, and

$A$  = area of the substrate, cm<sup>2</sup>.

As an example, for paint with a spreading rate of 98.2 cm<sup>2</sup>/mL (equivalent to 400 ft<sup>2</sup>/gal) and a density of 1.4 g/mL, the amount of paint to be applied to a 16 cm by 16 cm test substrate would be 3.65 g:

$$M_p = \left( \frac{\text{mL}}{98.2 \text{ cm}^2} \right) \left( \frac{1.4 \text{ g}}{\text{mL}} \right) (16 \text{ cm} \cdot 16 \text{ cm}) \quad (2)$$

9.2.2 Select a vial of paint for the test. Prepare the paint by vigorously shaking the vial for 5 min. Mixing must be done in the sealed vial to minimize loss of VOCs.

9.2.3 Wear cotton gloves during specimen preparation to prevent contamination by oils from the skin.

9.2.4 Weigh the substrate.

9.2.5 Pour the paint from the vial into a small, clean tray of appropriate size for the roller or brush being used for the application. Disposable trays can be made using clean aluminum foil. For a 7.6-cm wide paint roller, a tray that is 10 cm (4 in.) wide, 15 cm (6 in.) long, and 2 cm (0.75 in.) deep is appropriate.

9.2.6 Place the paint applicator into the tray and wet the surface uniformly with the paint. If a roller is used, apply the roller to the substrate starting at the center and roll outward to all edges until the surface is covered. Then rotate the substrate 90° and roll back and forth across the entire surface in this direction. Use a similar application procedure if using a brush to obtain a uniform coating on the substrate surface.

9.2.7 Immediately after application, weigh the substrate with the coating and record the weight. Calculate the weight of coating applied by the difference in the weight of the substrate before and after paint application. The final weight of paint applied should be within ±10 % of the target application amount. If the weight of coating is greater than ±10 % of the target mass, adjust the application procedure (for example, by changing the number of strokes or the amount of the paint in the brush, or both) and try again with a new substrate until desirable and repeatable applications can be achieved.

9.2.8 Each paint application process, including the weight measurement and calculation, should be finished within 3 min.

9.2.9 If a suitable balance is not available to weigh the substrate before and after application of the paint, an alternative method can be used involving weighing of the paint and applicator. Prior to preparation of the test specimen, pour the paint in the aluminum tray. Determine the combined weight of the tray, paint, and the applicator (roller or brush). Then apply the paint to the substrate. After the application is complete, weigh the paint tray and the applicator. Determine the amount of paint applied by calculating the weight difference.

## 10. Chamber Emissions Test Procedure

10.1 *Preparation of the Environmental Chamber Testing System*—Prior to each test, the chambers should be cleaned and operating parameters set.

10.1.1 Clean the environmental test chamber and all internal hardware by wiping the interior surfaces with an alkaline detergent, rinsing thoroughly with deionized water, and drying with clean laboratory tissue. Solvent such as acetone or methanol and/or chamber heating can be used when necessary.

Use the same procedure to clean all hardware on the chamber outlet and the sampling manifold.

10.1.2 Place the environmental test chamber in the environmental enclosure and set the enclosure temperature to the desired level. Close the chamber. Assemble the chamber outlet and manifold.

10.1.3 Adjust the water vapor concentration in the supply air to achieve the desired test conditions (for example, 50 % RH at 23°C). For water-based paints, % RH should be recorded throughout the test because the drying paint will affect the water vapor concentration. Ideally, in those cases the water vapor concentration in the outlet air (rather than the supply) should be controlled to 50 % RH. Start airflow to the chamber at the rate required for the desired air change rate (typically 0.5 or 1.0 ACH). For a 53-L chamber the airflow rate will be 442 mL/min for an ACH of 0.5 h<sup>-1</sup>. Measure the airflow rate at the inlet and the outlet of the chamber. If the inlet and outlet flow rates differ by more than 5 %, check for leaks.

10.1.4 Operate the empty chamber at desired test conditions for 24 h prior to use.

10.1.5 Collect background samples from the empty chamber at the end of the 24 h. Analyze the samples as soon as possible to ensure that the chamber background meets the criteria in 14.3.2 and is therefore ready for use. If the background VOC concentrations are too high, further cleaning or other measures may be required.

#### 10.2 Emissions Test Procedure:

10.2.1 Place the uncoated test substrate (and holder if used) into the chamber and condition for 24 h at the same temperature, RH, and ACH that will be used in the test.

10.2.2 At the end of the 24 h, collect an air sample from the chamber outlet, using a sorbent tube to measure VOC background concentrations from the environmental test chamber loaded with the uncoated substrate. Collect an air sample from the chamber outlet on a DNPH-silica gel cartridge to measure background concentrations of carbonyl compounds. Analyze the chamber background samples as soon as possible after collection. Individual VOCs or carbonyl compounds that are targeted for quantification in the emissions from the paint should not be present in the samples at concentrations greater than the chamber background criteria in 14.3.2. The emissions from the substrate should not include any compounds that may interfere with quantification of the emissions from the paint.

10.2.3 After the substrate background levels have been determined to be acceptable, open the chamber and remove the test substrate. Maintain airflow to the chamber and close it during preparation of the test specimen to minimize contamination of the chamber with laboratory air.

10.2.4 Prepare the test specimen by applying the paint as described in Section 9. If the application is acceptable, immediately place the test specimen into the chamber and close the chamber. Record time that the chamber is closed as “Time Zero” for the start of the test. Total preparation time from chamber opening, painting, weighing, to sealing of the coated test specimen within the chamber should not exceed 5 min.

10.2.5 Operate the test chamber at desired conditions for a test period during which air samples are routinely collected from the chamber outlet.

## 11. Sample Collection

11.1 Throughout the emission test, air samples will be collected from the outlet of the chamber. Samples will be collected on either charcoal media or thermal desorption sorbent tubes for VOC analyses and on DNPH-silica gel cartridges for analyses of carbonyl compounds.

11.1.1 Set up the vacuum pump(s) and mass flow controllers/meters for collection of sorbent tube and DNPH-silica gel cartridge samplers.

11.1.2 Set the flow controllers for the required airflow rates for the sorbent tube and DNPH-silica gel samplers. The required airflow rates will be a function of the emission rates for individual compounds and the volume of air sample required to obtain VOC mass on the sampler that is within the calibration range of the analytical instrument. During early stages of a test, due to the relatively high VOC concentrations, samples may need to be collected over periods of only 5 to 10 min at a low flow rate in order to obtain accurate sample volumes. Higher airflow rates and longer sample collection periods will be required in later stages of the test as the concentrations of VOCs decrease in the chamber air. The total sampling flow rate must be less than 50 % of the airflow from the chamber (for example, less than 221 mL/min for a 53 L chamber operated at 0.5 ACH). Set airflow rates with a sampling tube or cartridge connected to the sampling line. Sample flow rates should not exceed manufacturer’s recommendations.

11.1.3 Collect air samples for VOCs and carbonyl compounds at predefined intervals during the test. The duration of the test and the frequency of sample collection will be determined by the objectives of the test. A high frequency of sampling will be required if the objective of the test is to develop or evaluate source emission models. A lower frequency of sampling may be appropriate for other test objectives. If resources are available, the recommended sampling times for collection of data for model development during a two-week test with alkyd paint are at 0.25, 0.5, 1, 1.5, 2, 4, 6, 8, 10, 12, 24, 48, 72, 96, 144, 192, 264, and 336 h following the start of the test. Recommended sampling times during a two-week test with latex paint are at 2, 4, 8, 12, 24, 48, 72, 96, 144, 192, 264, and 336 h following the start of the test. After sampling, carefully seal sample tubes and record sampler flow rates and duration.

11.1.4 Collect duplicate air samples simultaneously at a subset of time periods. Use separate flow controllers for each sampler. Adjust total flow to be less than 50 % of the chamber outlet airflow rate.

11.1.5 Store samples collected on sorbent media refrigerated at less than 4°C in a clean environment. Analyze samples as soon as possible after collection. Samples should be analyzed within 30 days as recommended in EPA methods referenced in Section 2. Analyses should be performed following methods referenced in Section 2 or other published methods that produce results that meet the project objectives for precision and bias.

11.1.6 Store samples collected on DNPH-silica gel cartridges refrigerated at less than 4°C. Analyze the samples within 30 days. Analyses should be performed following

methods referenced in Section 2 or other published methods that produce results that meet the project objectives for precision and bias.

## 12. Chemical Analysis

12.1 For VOCs collected on sorbent media for analysis by thermal desorption/GC/MS, analyze samples following guidance in Practice D 6196 and EPA Compendium Methods TO-15 and TO-17.

12.2 For VOCs collected on charcoal, follow procedures described in Practice D 3687 to extract the analytes from the charcoal media with carbon sulfide and analyze the liquid extract by GC.

12.3 For carbonyl compounds collected on DNPH-silica gel cartridges, follow the procedures described in Test Method D 5197. The test method involves extraction of the media with acetonitrile and analysis of the extract by HPLC.

## 13. Reporting Test Results

13.1 The report should include the following information:

13.1.1 *Test Objectives*—Provide a clear description of the purpose of the test.

13.1.2 *Equipment and Methods*—Provide a description of the small chamber test facility equipment, including chamber size and materials, environmental control and measurement systems, sampling and analysis equipment, and description of the test methods and protocols.

13.1.3 *Product Identification*—Provide the name, specific identifiers from the manufacturer and a brief description of the product, its application, and history.

13.1.4 *Test Conditions*—Provide data for the temperature, humidity, air change rate, dimensions of the test specimen, empty chamber and substrate background concentrations, mean air velocity above the surface of specimen, and mass applied.

13.1.5 *Samples Collected*—Provide a record of samples taken including sampling schedule, flow rate, volume, frequency, and type.

13.1.6 *Quality Assurance/Quality Control Results*—Describe the data quality objectives and quality control activities for the test. Provide results for quality control samples (replicates, blanks, controls).

## 14. Quality Assurance and Quality Control

14.1 A quality assurance and quality control (QA/QC) plan should be prepared and implemented to ensure the integrity of the measured and reported data obtained during the tests. This plan should encompass all facets of the measurement program from sample receipt to final review and issuance of reports. QA/QC activities applicable to tests to measure emissions from paint are described in Guide D 5116.

14.2 *Data Quality Objectives and Acceptance Criteria*—The QA/QC plan should be based on established data quality objectives and acceptance criteria. Recommended data quality objectives are listed in Table 1.

14.3 *Quality Assurance Activities and Quality Control Samples*:

**TABLE 1 Recommended Data Quality Objectives**

Parameter	Precision	Accuracy
Chamber temperature	0.5°C	0.5°C
Chamber relative humidity	5.0 %	5.0 %
Airflow rate	5.0 %	5.0 %
Amount of paint applied	10 %	10 %

14.3.1 Determine the accuracy of chamber test conditions and operating parameters (temperature and RH) by calibration of measurement devices with National Institute of Standards and Technology (NIST)-traceable primary sources. Determine the precision of the test conditions by continuous recording of the parameters. Failure to meet acceptance criteria requires immediate corrective action. Check instrument performance before and after each test by comparison of measurements with a reference device.

14.3.2 The empty chamber background concentrations of VOCs should be measured at the start of each test without the substrate in the chamber. The empty chamber background concentration shall meet the following criteria: (1) less than 10 g/m<sup>3</sup> or 1/6 of the lowest concentration to be measured, whichever is lower, for the sum of measurable VOCs, and (2) less than 2 g/m<sup>3</sup> or 1/6 of the lowest concentration to be measured, whichever is lower, for individual VOCs.

14.3.3 Field blanks, consisting of sorbent tubes and DNPH silica gel cartridges that are not used for sampling, should be analyzed to verify that the tubes and cartridges have not been contaminated during handling and storage. The blanks should be stored and handled in the same manner as the samples collected during the test. The number of blanks analyzed should represent approximately 10 % of the number of samples collected for each type of sampling media; a minimum of 2 blanks for each type is recommended.

14.3.4 Precision of the sampling and analysis methods should be determined by collection of replicate samples. Duplicate samples should be collected concurrently at selected time periods during the test. The number of duplicates for each type of sampling media should represent approximately 10 % of the number of samples collected. Due to the limited volume of air available, duplicate samples can be collected for only one type of media during a sampling period, especially when low VOC concentrations dictate high sample volumes. Each sampling and analytical method used must have detection limits lower than the acceptable background levels stipulated in 14.3.2.

14.3.5 The accuracy and precision of the small chamber test method can be measured prior to start of a test program. The precision of the chamber test method, to include the combined variability of test specimen preparation, chamber operation, sampling, and analysis, can be measured by performing multiple chamber tests with the same source and substrate. Variation in test data from a single chamber and among chambers can also be established by use of standardized sources such as permeation tubes for determining organic concentrations and calculated emission rates.

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