



Designation: D7706 – 11

Standard Practice for Rapid Screening of VOC Emissions from Products Using Micro-Scale Chambers¹

This standard is issued under the fixed designation D7706; 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 practice describes a micro-scale chamber apparatus and associated procedures for rapidly screening materials and products for their vapor-phase emissions of volatile organic compounds (VOCs) including formaldehyde and other carbonyl compounds. It is intended to complement, not replace reference methods for measuring chemical emissions for example, small-scale chamber tests (Guide [D5116](#)) and emission cell tests (Practice [D7143](#)).

1.2 This practice is suitable for use in and outside of laboratories, in manufacturing sites and in field locations with access to electrical power.

1.3 Compatible material/product types that may be tested in the micro-scale chamber apparatus include rigid materials, dried or cured paints and coatings, compressible products, and small, irregularly-shaped components such as polymer beads.

1.4 This practice describes tests to correlate emission results obtained from the micro-scale chamber with results obtained from VOC emission reference methods (for example, Guide [D5116](#), Test Method [D6007](#), Practice [D7143](#), and ISO 16000-9 and ISO 16000-10).

1.5 The micro-scale chamber apparatus operates at moderately elevated temperatures, 30°C to 60°C, to eliminate the need for cooling, to reduce test times, boost emission rates, and enhance analytical signals for routine emission screening, and to facilitate screening of semi-volatile VOC (SVOC) emissions such as emissions of some phthalate esters and other plasticizers.

1.6 Gas sample collection and chemical analysis are dependent upon the nature of the VOCs targeted and are beyond the scope of this practice. However, the procedures described in Test Method [D7339](#), Practice [D6196](#) and ISO 16000-6 for analysis of VOCs and in Test Method [D5197](#) and ISO 16000-3 for analysis of formaldehyde and other carbonyl compounds are applicable to this practice.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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:*²

[D1356 Terminology Relating to Sampling and Analysis of Atmospheres](#)

[D1914 Practice for Conversion Units and Factors Relating to Sampling and Analysis of Atmospheres](#)

[D5116 Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products](#)

[D5197 Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air \(Active Sampler Methodology\)](#)

[D6007 Test Method for Determining Formaldehyde Concentrations in Air from Wood Products Using a Small-Scale Chamber](#)

[D6196 Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air](#)

[D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps](#)

[D7143 Practice for Emission Cells for the Determination of Volatile Organic Emissions from Indoor Materials/Products](#)

[D7339 Test Method for Determination of Volatile Organic Compounds Emitted from Carpet using a Specific Sorbent Tube and Thermal Desorption / Gas Chromatography](#)

¹ This practice is under the jurisdiction of ASTM Committee [D22](#) on Air Quality and is the direct responsibility of Subcommittee [D22.05](#) on Indoor Air.

Current edition approved March 1, 2011. Published March 2011. DOI: 10.1520/D7706-11.

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.

2.2 ISO Standards:³

ISO 16000-3 Determination of formaldehyde and other carbonyl compounds – Active sampling method

ISO 16000-6 Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas-chromatography using MS/FID

ISO 16000-9 Indoor air—Part 9: Determination of the emission of volatile organic compounds – Emission test chamber method

ISO 16000-10 Indoor air—Part 10: Determination of the emission of volatile organic compounds – Emission test cell method

2.3 Other Standard:⁴

US EPA Method TO-17 Determination of volatile organic compounds in ambient air using active sampling onto sorbent tubes

3. Terminology

3.1 *Definitions*—For definitions and terms commonly used for sampling and analysis of atmospheres, refer to Terminology **D1356**. For definitions and terms commonly used when testing materials and products for VOC emissions, refer to Guide **D5116**. For an explanation of general units, symbols and conversion factors, refer to Practice **D1914**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *micro-scale test chamber, n*—an environmental test chamber ranging in volume from a few milliliters to about 150 mL and designed to operate at moderately elevated temperatures that is used to measure vapor-phase organic emissions from small specimens of solid materials and products.

3.2.2 *control level, n*—a user-defined acceptance criterion for a micro-scale chamber test, for example, presence or absence of a target compound and/or a concentration or emission rate of a target compound, typically used in production quality control to indicate that the tested product sample likely will meet the corresponding acceptance criterion for a reference test.

4. Principles

4.1 Micro-scale test chambers operate under the same mass transfer principles as conventional small-scale test chambers and cells for measuring emissions of VOCs including formaldehyde and other carbonyl compounds from materials and products (see Guide **D5116** and Practice **D7143**).

4.2 Clean gas (dry nitrogen or air) is supplied to a micro-scale chamber and passes over the exposed surface of the test specimen before reaching the exhaust point. The gas flow rate and temperature within the micro-scale chamber are controlled. As the gas passes over the test specimen, emitted compounds are swept away from the surface.

4.3 After the test specimen has equilibrated in the micro-scale chamber (typically for 20-40 minutes), a sampling device is connected to the outlet for collection of vapor-phase compounds exiting the chamber.

5. Summary of Practice

5.1 Micro-scale chambers can be used for rapid screening and quality control of VOC emissions from many materials and products. Compatible sources include (with examples): rigid materials (plastics, wood-based panels, hard surface flooring), compressible materials (textiles, foams, polymer sheeting), irregularly-shaped materials (polymer components, carpet), and wet-applied products in dried or cured form (for example, paints, coatings, adhesives, caulks, sealants).

5.2 Representative test specimens are prepared from material/product samples and are placed directly into micro-scale chambers. For samples that are heterogeneous, it is necessary to prepare and test replicate specimens. In some cases, it may be necessary to precondition samples or specimens prior to testing.

5.3 Micro-scale chambers typically are used for measuring area-specific emissions from surfaces. They also can be used to determine mass-, length- or unit-specific emission rates from variously shaped test specimens.

5.4 Chamber bodies are held at moderately elevated temperatures of 30°C to 60°C and typically are supplied with a controlled flow of clean, dry gas, either nitrogen or air.

5.5 Specific operating procedures are developed for each type of material or product. The key parameters of equilibration time, chamber temperature and inlet gas flow rate are optimized in an iterative process starting from typical conditions and then confirmed by the analysis of replicate specimens.

5.6 Gas samples for VOCs are collected at the exhaust of the micro-scale chamber. For ease of use, the entire gas flow exiting the chamber typically passes through the sampling device.

5.7 A number of gas sampling and analytical methods are compatible with micro-scale chambers. VOCs may be collected on sorbent tubes and analyzed by thermal desorption—gas chromatography (GC) with mass spectrometry (MS) and/or flame ionization detection (FID) to identify and quantify compounds as described in Test Method **D7339**, Practice **D6196**, ISO 16000-6 and US EPA Method TO-17. Formaldehyde and other carbonyl compounds may be sampled and analyzed as described in Test Method **D5197** and ISO 16000-3. Other analytical techniques such as direct-reading instruments may be used if applicable.

5.8 This practice describes tests that are used to correlate emission results obtained from micro-scale chambers to reference results from conventional emission test chambers and cells (that is, Guide **D5116**, Test Method **D6007**, Practice **D7143**). This relationship is then developed and validated to establish a ‘control’ level to evaluate whether the sample is likely to be compliant with guidelines or regulations for VOC emissions that are determined by a reference method—See Section 12.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁴ Available from United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

6. Significance and Use

6.1 Manufacturers increasingly are being asked or required to demonstrate that vapor-phase emissions of chemicals of concern from their products under normal use conditions comply with various voluntary or regulatory acceptance criteria. This process typically requires manufacturers to have their products periodically tested for VOC emissions by independent laboratories using designated reference test methods (for example, Test Method **D6007**, ISO 16000-9, and ISO 16000-10). To ensure continuing compliance, manufacturers may opt to, or be required to, implement screening tests at the production level.

6.2 Reference methods for testing chemical emissions from products are rigorous and typically are too time-consuming and impractical for routine emission screening in a production environment.

6.3 Micro-scale chambers are unique in that their small size and operation at moderately elevated temperatures facilitate rapid equilibration and shortened testing times. Provided a sufficiently repeatable correlation with reference test results can be demonstrated, appropriate control levels can be established and micro-scale chamber data can be used to monitor product manufacturing for likely compliance with reference acceptance criteria. Enhanced turnaround time for results allows for more timely adjustment of parameters to maintain consistent production with respect to vapor-phase chemical emissions.

6.4 This practice can also be used to monitor the quality of raw materials for manufacturing processes.

6.5 The use of elevated temperatures additionally facilitates screening tests for emissions of semi-volatile VOCs (SVOCs) such as some phthalate esters and other plasticizers.

7. Apparatus

7.1 General Description:

7.1.1 The micro-scale chamber test apparatus comprises one or more micro-scale chambers, a means of incubating the micro-scale chamber(s) at controlled temperature, a regulated clean gas (nitrogen or air) supply system with optional humidification, gas sampling capabilities, and instrumentation for control, monitoring and recording of conditions.

7.1.2 A chamber is typically cylindrical in shape to accommodate an O-ring seal and ranges in total volume from a few milliliters to about 150 mL depending upon the mode of operation, that is, chamber mode for bulk sample emission testing and cell mode for surface emission testing of planar specimens. An example of a typical micro-scale chamber illustrating both modes of operation is shown in **Appendix X1**.

7.1.3 The chamber body and lid assembly are designed to be leak free.

7.1.4 A chamber body often has an integral sample holder that accommodates a planar test specimen such that the back surface and edges of the specimen are not exposed to the gas stream. The chamber body also can accommodate irregularly shaped materials for bulk emission testing.

7.1.5 The typical cylindrical shape, small exposed volume and associated high air change rate of the micro-scale chamber;

together with the positioning of the gas inlet and outlet perpendicular to the sample surface for a planar specimen (Annex A) are designed to optimize turbulence, eliminate still air and ensure thorough mixing of the gas within the chamber at the range of flows specified (see **7.4.4**). Typically all of the gas exiting the chamber outlet passes onto the gas sampling device, further ensuring representative sampling. Recovery tests can be used to demonstrate adequate mixing (see **8.7.1**).

7.2 Construction:

7.2.1 The micro-scale chamber body and associated lid are constructed of polished or inert-coated stainless steel.

7.2.2 The gasket or O-ring used to seal the lid to its body is low absorbing and low emitting at the operating temperature ($\leq 60^{\circ}\text{C}$) so it does not contribute significantly to background VOC concentrations. (see **10.2**) Gaskets and O-rings composed of fluoroelastomer polymers are suitable for this application.

7.2.3 The apparatus is designed for easy disassembly to facilitate cleaning. The chamber body is easily removed from the heater housing and the gasket or O-ring is easily removed from the body.

7.3 Heating:

7.3.1 The chamber body sits in a heater housing that can evenly heat the chamber body and maintain it at controlled temperatures between 30°C and 60°C with an accuracy of $\pm 1^{\circ}\text{C}$ and a precision of $\pm 2^{\circ}\text{C}$ at the set temperature. The micro-scale chamber lid and air/gas supply also are heated.

7.3.2 The apparatus may provide for elevated heating of chamber bodies to approximately 100°C as a cleaning procedure.

7.4 Gas Supply:

7.4.1 The apparatus includes a means of supplying clean gas, either dry nitrogen or air to the chambers. Either electronic or mechanical flow controllers are used. The flow rate to each chamber is individually controlled with an accuracy of $\pm 2\%$ and a precision of $\pm 3\%$ of the reading.

7.4.2 VOC levels in the gas supply are sufficiently low so that specified background levels can be achieved (see **10.2.2**).

7.4.3 For some applications, it may be necessary to provide a means of humidifying the inlet gas stream. Water used for humidification is of sufficient purity such that VOC background requirements (see **10.2.2**) can be achieved.

7.4.4 Inlet gas flow rates between approximately 30 and 500 mL/min are required depending upon application (see **8.4**).

7.5 Integrity:

7.5.1 During operation with or without a gas sampling device attached to the outlet, the chamber is considered sufficiently leak-free if the outlet gas flow rate is within 95 % of the inlet gas flow rate measured at the same temperature and humidity conditions.

7.6 Gas Sampling:

7.6.1 Gas samples are collected from the chamber by connecting a sampling device (sorvent tube for VOCs or 2,4-dinitrophenylhydrazine (DNPH) coated cartridge for formaldehyde and other carbonyl compounds) directly to the outlet of the chamber. In this case, all of the outlet gas flows through the sampling device and the sampling flow rate is the same as the chamber inlet gas flow rate.

7.6.2 Alternatively, gas exiting the chamber can be pulled through an attached sampling device using a flow controlled and calibrated pump system (see Guide D5116, Practice D7143, Practice D5337). If a pump system is used, the chamber outlet is open to atmospheric pressure and the sampling flow rate is set to a value less than the inlet flow rate such that the pressure in the chamber is not affected.

8. Test Conditions—Optimizing Parameters and Verifying Performance

8.1 Parameter optimization establishes the conditions that result in acceptable correlation between the emissions of VOCs measured in a micro-scale chamber and those measured by a reference method while minimizing the time needed to perform the test. The key parameters for micro-scale chamber tests are equilibration time, chamber temperature, inlet gas flow rate and vapor sampling time. These parameters are optimized in an iterative manner starting from typical conditions for each material/product source of interest. Suggested initial conditions for routine use of the micro-scale chamber for VOC and formaldehyde sampling are summarized in Table 1.

8.2 Equilibration Time:

8.2.1 *Typical Use*—The equilibration time is the time between initiating gas flow after placing the test specimen into an empty micro-scale test chamber and the beginning of gas sampling. This and the other test parameters are specific to the source being tested. For many source types, equilibration times are typically 20 to 40 minutes; but this value can vary substantially depending upon the source and the compound(s) of interest.

8.2.2 Procedure Development:

8.2.2.1 Experimentally determine equilibration times by first placing the specimen into a micro-scale chamber and operating the chamber at typical use conditions (see Table 1). Conduct these and the subsequent tests in duplicate with two chambers operating simultaneously or with one chamber operated sequentially.

8.2.2.2 Initiate gas sampling of emitted VOCs (or formaldehyde) immediately after starting the test. Collect a minimum of six sequential samples, for example one sample every ten minutes for an hour. Plot the concentration of each compound of interest against time to evaluate the change in emissions. If the duplicate specimens are not identical with respect to exposed surface area (or mass), calculate specific emission rates and plot these rates. The minimum equilibration time under these conditions is five or ten minutes longer than the time taken for the signals of the compound(s) of interest to stabilize.

NOTE 1—Typically, the rate of change of the signal (concentration or

emission rate) for individual compounds will be fast initially and then stabilize or level off (that is, <10 % difference relative to the previous measurement).

8.2.2.3 Observe the difference between the duplicate tests. Duplicate results should agree within approximately ±25 % depending upon sample homogeneity (see 8.7.2 and 11.2).

8.2.2.4 Repeat the test if optimized operating parameters (for example, temperature) for a given material/product are subsequently established at values different from those used to determine the equilibration time.

8.2.2.5 For SVOCs, extend the overall period (for example, from 1 to 30 hours) and increase the gas sampling time; for example, collect a sample every hour on the first day, leave overnight and then collect three additional consecutive hourly gas samples the following day. If necessary, run the test at a higher temperature to reduce equilibration and overall test times.

8.3 Temperature:

8.3.1 *Typical Use*—Use moderately elevated temperatures ranging from 30°C to 60°C to speed up micro-scale emission tests of specimens of dry materials/products.

8.3.2 Procedure Development:

8.3.2.1 Optimize the temperature parameter using an equivalent procedure to that used for establishing the equilibration time. Conduct the test in duplicate. Typically, start the process with the chamber temperature at 40°C. If an optimum inlet gas flow rate has not been determined at this stage, use a gas flow rate of approximately 50 or 250 mL/min (see Table 1).

8.3.2.2 At the end of the equilibration time (see 8.2.2.2), collect and analyze a 10- or 60-minute gas sample (see Table 1). Compare the results to the emission profile/data obtained from a reference emission test. If some of the VOCs observed in reference test are missing and/or if the signals are weak and difficult to measure, repeat the test by raising the chamber in 10°C increments within the 30°C to 60°C range until the signal strengths increase and all compounds of interest are observed. Alternately, if additional compounds are observed that are not seen in the reference test, repeat the test by lowering the chamber temperature in 5°C intervals until the significant differences are no longer present.

8.3.2.3 Check that the duplicate measurements agree within approximately ±25 % depending upon sample homogeneity (see 8.7.2 and 11.2).

8.3.2.4 Compare the emission profiles obtained at each temperature with those from a reference emission test. Keep the temperature of the micro-scale chamber test below the point where significant differences begin to appear, that is, below the point at which additional components are observed.

TABLE 1 Suggested Initial Conditions for Routine Use of the Micro-Scale Chamber for Measuring Emissions of VOCs and Formaldehyde

Test Parameter	Initial test conditions for routine use of micro-scale chamber	
	VOCs	Formaldehyde
Equilibration time range (min)	20–40	20–40
Chamber temperature (°C)	40	40
Inlet gas flow rate range (mL/min)	50	250
Gas sampling time (min)	10	60