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

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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 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.

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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization 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

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)

D5337 Practice for Setting and Verifying the Flow Rate of Personal Sampling Pumps

D6007 Test Method for Determining Formaldehyde Concentrations in Air from Wood Products Using a Small-Scale Chamber

D6196 Practice for Choosing Sorbents, Sampling Parameters and Thermal Desorption Analytical Conditions for Monitoring Volatile Organic Chemicals in Air

D7143 Practice for Emission Cells for the Determination of Volatile Organic Emissions from Indoor Materials/Products

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.

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

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

U.S. 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 250 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 min to 40 min), 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 U.S. 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

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), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

likely to be compliant with guidelines or regulations for VOC emissions that are determined by a reference method—See Section 12.

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 250 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 (**Appendix X1**) 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 should be low absorbing and low emitting at the operating temperature (≤ 60 °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 disassembly to facilitate cleaning. The chamber body is removed from the heater housing and the gasket or O-ring is 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 ± 1 °C and a precision of ± 2 °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 ± 2 % and a precision of ± 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 mL/min 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 (sorbert 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.

NOTE 1—Temp and humidity have an effect on HCOH emissions.

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 min to 40 min; 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 (Table 1) is five or ten minutes longer than the time taken for the signals of the compound(s) of interest to stabilize.

NOTE 2—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 h to 30 h) 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 mL/min 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 min or 60 min 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,

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

Initial Test Conditions for Routine Use of Micro-Scale Chamber		
Test Parameter	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

below the point at which additional components are observed. If the temperature has been altered by more than 5 °C, reconfirm the equilibration time (see 8.2.2).

NOTE 3—Be aware that the relative humidity of the gas stream exiting the chamber also may be affected by temperature changes depending upon the moisture content of the test specimen.

8.4 Gas Flow Rate:

8.4.1 *Typical Use*—Micro-scale chambers are generally designed such that all of the gas flow entering the chamber passes through the gas sampling device attached to the chamber outlet. To accommodate this mode of sampling and the volume requirements of the sampling devices (see Practice D6196 and Test Method D5197), typically set gas flow rates at 30 mL/min to 100 mL/min for VOCs and 150 mL/min to 500 mL/min for formaldehyde and other carbonyls.

8.4.2 Procedure Development:

8.4.2.1 Optimize the flow rate parameter using an equivalent iterative procedure to those described above. Conduct the test in duplicate. Set the optimized micro-scale chamber temperature (see 8.3.2.2). Set the gas flow rate to 50 mL/min for VOCs and 250 mL/min for formaldehyde. At the end of the equilibration time (see 8.2.2), collect and analyze a 10 min or 60 min gas sample (see Table 1).

8.4.2.2 For VOCs, repeat the test by lowering or increasing the gas flow rate through the chamber in approximate 20 mL/min increments over the 30 mL/min to 100 mL/min range until the ratio of the least volatile compounds to the most volatile compounds of interest stabilizes (<10 % difference relative to the previous measurement), or matches that from an emission test by a reference method.

8.4.2.3 Use elevated (for example, 150 mL/min to 500 mL/min) gas flow rates and substantially longer sampling times to achieve adequate analytical sensitivity for micro-scale chamber tests of formaldehyde and SVOC emissions.

8.4.2.4 If the gas flow has been adjusted by more than 20 mL/min, reconfirm the equilibration time (see 8.2.2).

8.5 *Gas Sampling Time*—Initially, use a gas sampling time of 10 min to 15 min for VOCs and 60 min to 90 min for formaldehyde. Adjust the times as necessary to achieve the desired sensitivity.

8.6 *Monitoring and Data Acquisition*—Monitor temperature, gas flow rate and humidity (if required) throughout a test with appropriate sensing devices. Electronically acquire and record the data.

8.7 Verification of Parameters:

8.7.1 Analyte Recovery:

8.7.1.1 Once the test parameters have been optimized for a given material or product, measure the recovery of the compounds of interest. Re-evaluate analyte recovery for each new micro-scale chamber apparatus that is commissioned and periodically as required by the laboratory's quality management system.

8.7.1.2 Determine the recovery of organic vapors present at high concentration by introducing a known mass of a target analyte (for example, toluene or n-decane) into the micro-scale chamber onto an inert sample holder such as a clean PTFE or glass disk with a well in the center. Compare the amount of the

compound collected at the chamber exhaust over a given time to the amount introduced. The mass of vapor experimentally recovered should be 100 % ± 20 % of the introduced mass.

8.7.1.3 Use alternate procedures (for example, permeation or diffusion-controlled sources) to demonstrate recovery of organic vapors present at low ($\mu\text{g}/\text{m}^3$) concentrations.

8.7.1.4 Temperature of chambers (see Appendix X2).

8.7.2 *Repeatability*—Once the test parameters have been optimized, test three to six replicate specimens of the same sample to evaluate repeatability. The calculated emission rates of the target analytes among replicates should agree within ±25 %. If dealing with a heterogeneous material, repeat the test on three to six replicate specimens at least three times and calculate the variability of the average emission rates for the replicate sets (see also 9.1 and 11.2).

9. Sample Collection and Preparation of Test Specimens

9.1 Because specimen sizes utilized in micro-scale chambers are relatively small, the homogeneity of the material/product must be considered and accommodated in the design of a sample collection plan. The details of designing a sampling plan are outside of the scope of this practice; however, in many cases, it is necessary to collect and analyze multiple specimens to obtain a representative emission value for a larger sample such as a board product or roll goods.

9.2 If information is being sought for a finished product, samples are typically collected from the end of the normal manufacturing line. If tests are not to be performed immediately, wrap samples in impermeable, non-emitting, and non-absorbing packaging. Guidance on sample collection, storage and preparation of test specimens is given in Guide D5116 and Practice D7143.

9.3 A specimen for bulk emissions testing often is weighed or otherwise measured into a suitable inert container that is inserted into the micro-scale chamber or it may be weighed/measured and placed directly into the body of the chamber (see Appendix X1).

9.4 A test of surface emissions is achieved using the integral specimen holder to seal the edges and back of the test specimen (see Appendix X1). Alternately, the edges and back of the specimen can be sealed with aluminum tape, provided emissions from the tape do not compromise micro-scale chamber background levels at the temperatures used (10.2.2).

10. Preparation of Apparatus

10.1 *Suggested Procedures for Cleaning Chamber Components:*

10.1.1 Remove the gaskets or O-rings and clean the chamber components using a dilute alkaline detergent followed by three separate rinses with distilled water. Alternately, clean the chamber components with methanol followed by three distilled water rinses. Avoid abrasives and high pH. Dry thoroughly.

10.1.2 If the apparatus is designed to accommodate elevated temperature, the chamber can be cleaned without disassembly. Raise the chamber temperature to approximately 100 °C with elevated flow of clean gas sufficiently long to reduce background artifacts to acceptable levels at normal operating conditions.

10.2 Chamber Background Measurement:

10.2.1 Measure chamber background levels prior to each use or at a frequency dictated by the laboratory's quality management system. For field use, it is recommended that a blank measurement be carried out at the beginning of the day and before switching to a different type of material/product sample.

10.2.2 Background measurements are performed under normal operating conditions but without a specimen in the chamber. Background levels for VOCs of interest should be 10 %, or less, of the lowest levels of interest for actual samples.

11. Micro-Scale Chamber Test Procedures

11.1 Chamber Operation:

11.1.1 Turn on the gas supply and adjust the flow rate to the desired setting.

11.1.2 Heat the micro-scale chamber to the required temperature.

11.1.3 Open the chamber body and quickly insert the test specimen. Immediately seal the chamber body. This time marks the initiation of the test, t_0 .

11.1.4 Check the gas flow rate at the chamber exhaust. The value measured at the exhaust should be at least 95 % of the inlet flow rate (see 7.5).

11.1.5 Leave the chamber operating at stable conditions for the appropriate equilibration period.

11.1.6 Connect the sampling device to the outlet port of the chamber. This initiates gas sampling.

11.1.7 Re-check the gas flow rate at the exhaust of the sampler to determine if any leakage has been introduced.

11.1.8 The time from t_0 to the mid-point of the period of sample collection is considered the sampling time, t .

11.1.9 At the end of the sampling period, disconnect the sampling device.

11.1.10 Seal the sampling device using appropriate fittings. If analysis is not to be performed immediately, store the sampler as described in the relevant method.

11.1.11 Analyze the sample within the prescribed time following the applicable method.

11.2 *Replication*—For materials and products that are heterogeneous on the scale of the micro-scale chamber, it is necessary to test multiple replicate specimens in order to obtain representative VOC emission factors. The scheme for the collection of replicate specimens and the number of required specimens must be determined on a case-by-case basis for each material/product. If sample heterogeneity is such that the uncertainty of a value determined from a practical number of replicates is unacceptable (for example, if average emission rates from multiple replicate sets vary by more than ± 25 %; see 8.7.2), then a different measurement procedure likely is required.

12. Control Emission Levels for Factory Production

12.1 Establishing a Control Level:

12.1.1 If data are to be used for quality control of raw materials or products in a manufacturing environment, appropriate reference levels need to be established. Control levels can be set for all relevant target compounds or key 'indicative'

target compounds and/or measures of total VOCs, depending on user requirements. These control levels are set by comparing micro-scale chamber data with reference emission test data for five or more different batches of the material/product of interest. To be effective, the batches selected for this purpose should include examples of the material/product that fail or come close to failing the established emission limits for the compound(s) of interest in reference tests.

12.1.2 Samples for conventional and micro-scale chamber tests should be simultaneously collected from the same material/product batches. Collect six or more replicate samples from each batch and place each sample in impermeable, non-contaminating packaging. Test (either in house or by contract) half the samples from each batch by the reference method and test the other half using the optimized micro-scale chamber procedure.

NOTE 4—The optimized micro-scale procedure may require testing of replicate specimens for each sample.

12.1.3 Compare the micro-scale and conventional emission test results for each compound of interest. This may require calculating specific emission rates (see Section 13).

12.1.4 Average the results by batch and procedure and compute RSDs. These RSDs should be within ± 25 %.

12.1.5 Plot the average micro-scale chamber results versus the average conventional chamber results. Apply a linear least-squares fit to the data. Calculate and plot the 95 % and 99 % confidence intervals for the relationship.

12.1.6 At the regulated or desired conventional chamber emission level, determine the corresponding lowest micro-scale chamber emission level at the 95 % or 99 % confidence level as appropriate. This can be used directly as the control level if it provides the necessary level of confidence.

12.1.7 If there is substantial variation due to a heterogeneous source or if the adverse consequence of a false positive result is substantial, it may be necessary to introduce an additional uncertainty factor, that is, set a lower control level.

12.2 Procedures for Mixtures:

12.2.1 For a mixture of VOCs over a defined volatility range, adjust the test parameters of the micro-scale chamber test so that the overall chromatographic 'fingerprint' of emissions resembles that from the reference method assuming the same analytical method is used. This will ensure that the same compounds over the same volatility range of interest are represented in the micro-scale test.

12.2.2 The closeness of this fit will dictate the data analysis procedures. For example, if 90 % or more of the combined mass of the VOCs of interest from the reference method, is represented in the micro-scale results, data analysis can proceed as described above. If not, it may be necessary to quantify and create correlations for individual compounds to establish a valid control levels.

12.3 Continuous Maintenance of Control Level:

12.3.1 Document the procedures used to establish a control level including production details for the samples selected for the test. If any of the production parameters change (for example, a change in formulation or in the supplier of a component material), it will be necessary to evaluate the