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Standard Practice for Emission Cells for the Determination of Volatile Organic Emissions from Indoor Materials/Products¹

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INTRODUCTION

This practice complements Guide D5116 and Practice D6670.

1. Scope

- 1.1 This practice is intended for determining volatile organic compound (VOC) emissions from materials and products using emission cells. It can be applied in principle to most construction materials and many products used indoors. Objectives include:
- 1.1.1 To provide manufacturers, builders, and end users with emission data useful for evaluating the impact of building products, new or old, on indoor air concentrations in a model room.
- 1.1.2 To promote the development of products with lower VOC emissions.
- 1.2 This practice is for identifying emitted VOCs and for determining the area specific emission rate of VOCs from newly produced building products under defined climate conditions. The method can also be applied to aged products.
- 1.3 In accordance with the definition of an emission cell, it is also possible to perform nondestructive emission measurements on building products on-site in buildings. However, the procedure for such measurements is not described in this standard.
- 1.4 This practice describes the design, construction, performance evaluation and use of emission cells for VOC emission testing. Sampling, transport and storage of materials to be tested, and preparation of test specimens are also described.
- 1.5 Air sampling and analytical methods for the determination of VOCs are described in Practice D6196. Alternative sampling and analytical approaches for formaldehyde and other carbonyls are described in Test Method D5197.

Note 1—All volatile (vapor-phase) carbonyls except formaldehyde can be analyzed by either Practice D6196 or by Test Method D5197.

Note 2—Direct-reading instruments can also be applied for specific objectives.

Note 3—Some volatile inorganic compounds can, in principle, also be

analyzed (for example, ammonia).

1.6 An example of an emission cell is described in Appendix X2 of this practice.

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 Flow Rate Calibration of Personal (Sampling Pumps

D6196 Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air

D6330 Practice for Determination of Volatile Organic Compounds (Excluding Formaldehyde) Emissions from Wood-Based Panels Using Small Environmental Chambers Under Defined Test Conditions

D6670 Practice for Full-Scale Chamber Determination of Volatile Organic Emissions from Indoor Materials/ Products

2.2 Others Standards and Documents:

EN 196-1 Methods of Testing Cement—Part 1: Determination of Strength

EN 428 Resilient Floor Coverings—Determination of Overall Thickness

EN 430 Resilient Floor Coverings—Determination of Mass per Unit Area

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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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.

- EN 927-1 Paints and Varnishes—Coating Materials and Coating Systems for Exterior Wood
- EN 1937 Test Method for Hydraulic Setting Floor Smoothing and/or Leveling Compounds—Standard Mixing Procedures
- EN 13892-1 Methods of Test for Screed Materials—Part 2: Sampling, Making, and Curing Specimens for Test
- EN ISO 3251 Paints and Varnishes—Determination of Non-Volatile Matter of Paints, Varnishes, and Binders for Paints and Varnishes
- EN ISO 16017-1 Air Quality—Sampling and Analysis of Volatile Organic Compounds in Ambient Air, Indoor Air and Workplace Air by Sorbent Tube/Thermal Desorption/Capillary Gas Chromatography—Part 1: Pumped Sampling
- EN ISO DIS 13419-1 Building Products—Determination of the Emission of Volatile Organic Compounds—Part 1: Emission Test Chamber Method
- ISO 554 Standard Atmospheres for Conditioning and/or Testing
- ISO 1765 Machine-Made Textile Floor Coverings— Determination of Thickness
- ISO 2811 Paints and varnishes—Determination of density
 ISO 3233 Paints and Varnishes—Determination of Percentage Volume of Non-Volatile Matter by Measuring the Density of a Dried Coating
- ISO 8543 Textile Floor Coverings—Methods for Determination of Mass
- ISO 16000-6 Indoor air—Part 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
- EPA-600/4-89/017 US EPA Compendium of Methods for Determination of Toxic Organic Compounds in Ambient Air available through the National Technical Information Service, Springfield, VA 22161; PB90-116989. This report contains US EPA Method TO-17—Determination of volatile organic compounds in ambient air using active sampling onto sorbent tubes.
- Nordtest NT Build 438 (1995) Building Materials: Emission of Volatile Chemicals—Field and Laboratory Emission Cell

3. Terminology

- 3.1 *Definitions*—For definitions and terms commonly used in ASTM standards, including this practice, refer to Terminology D1356. For an explanation of general units, symbols and conversion factors, refer to Practice D1914
- 3.2 Definitions of Terms Specific to This Standard:For the purposes of this standard, the following terms and definitions apply.
- 3.2.1 air change rate—the flow rate of clean, conditioned air into the cell divided by the cell volume; usually expressed in units of h^{-1} .
- 3.2.2 *area specific air flow rate*—ratio between the supply air flow rate and the area of the test specimen.
- 3.2.3 *emission cell*—a portable device for the determination of volatile organic compounds emitted from indoor materials/ products.

- 3.2.3.1 *Discussion*—The emission cell is placed against the surface of the test specimen, such that the surface of the test specimen itself becomes part of the emission cell. This is the fundamental difference between emission cells and emission chambers. The air inlet of the emission cell is designed such that the flow of air is directed over the surface of the test specimen.
- 3.2.3.2 *Discussion*—An example emission cell is described in Appendix X2.
- 3.2.4 *product loading factor*—the ratio of the test specimen area and the emission cell volume.

4. Summary of Practice

- 4.1 Emission cells are suitable for relatively-homogeneous indoor materials/products, which present a planar surface to the emission cell.
- Note 4—Small emissions chambers are similarly limited with respect to sample inhomogeneity. To overcome this issue, with either emission cells or small emission chambers, multiple measurements should be made from different parts of the same sample in order to obtain an average emission measurement.
- 4.2 Indoor materials/products which have a planar surface (wood-based panel products, dried paints or coatings, flooring products, textiles, foams, polymer sheeting, dried adhesive layers, and so forth) or which can be made to present a planar surface to the emission cell (polymer beads, carpet, mold cultures in petri dishes, and so forth) are placed under the emission cell such that the test specimen itself forms one face of the emission cell. Pure, humidified air is passed into the cell through a baffle around the perimeter such that it passes over the whole surface of the test specimen. The temperature and humidity are closely controlled. As air passes over the test specimen, vapor-phase organics emitted from the surface are swept away from the test specimen in the flow of air. The air/vapor exit (exhaust) point is usually located centrally, immediately above the test specimen surface, to avoid unswept volumes and sink effects (see 7.6 and Appendix X2). The exhaust air is fully mixed such that air sampled at the exit point is representative of the air in the cell. Approximately 80 % of the flow of air into the cell is pumped onto two sample tubes. The excess air is allowed to exhaust through an overflow vent to ensure that a slight positive pressure is maintained inside the cell to prevent ingress of background air.
- 4.3 The air flow rate is set such that the air velocity over the surface of the test specimen has no effect on the area specific emission rate (see 6.4). The emissions tests are carried out at fixed times after preparation of the test specimen (for example, after 2 hours, 24 hours, 72 hours, 10 days, 28 days, 56 days, or 182 days (26 weeks)). Throughout the entire test period, test pieces shall either be kept under the emission cell under the flow of pure, humidified air, or stored in a clean, well-ventilated environment, under controlled conditions of temperature and humidity, with no risk of contamination from other samples or other emission sources.

Note 5—The air flow rate at the surface of the test specimen is particularly critical for wet indoor materials/products where the primary emission process is evaporation (external diffusion). In these cases, while it will remain possible to compare emission data from wet samples collected using similar emission cells under identical conditions, the

non-uniformity and relative slowness of the air velocity at the surface of the test specimen, will make it difficult to compare emission cell data with that obtained using an emission chamber (see Appendix X4).

Note 6—Similar limitations make it difficult to compare emission data from two different small chambers or from the same chamber under different operating conditions, if that data is obtained during the drying/curing stages of a wet product.

4.4 The sample tubes used for collecting VOCs are analyzed by thermal desorption: gas chromatography (GC); usually with mass spectrometry (MS) and flame ionization detection (FID) to identify and quantify the target volatile organic compounds as described in Practice D6196, ISO 16000-6 or ISO 16017-1. The measured masses of volatile organic compounds retained by the sorbent tubes are then used to determine the area specific emission rates of the material or product. Alternative sampling and analytical techniques are used for formaldehyde (and for other carbonyls) as described in Test Method D5197.

5. Significance and Use

- 5.1 Indoor materials/products are products or materials used for construction works or in the indoor environment. The area specific emission rates of volatile organic compounds from an indoor material/product may be used to estimate the expected contribution of emissions from that material/product to the atmosphere of a given indoor environment.
- 5.2 Emission data may also be used to compare and categorize different indoor materials/products of similar function.
- 5.3 Emission cell testing of area specific emissions may alternatively be used for studying secondary interactions (for example, sink effects (absorption and re-emission of volatile organics by the indoor material/product) or emissions generated by chemical degradation of the indoor material/product caused by specific atmospheric agents such as water, ozone or NO_x).

6. Principles lards iteh ai/catalog/standards/sist/a1bfe77

6.1 General Principles

- 6.1.1 Area specific emission rates at a given lapsed time (t) are calculated from the masses of target volatile organic compounds collected on the sample tubes, the flow of air pumped through each sample tube, the total flow of air entering the emission cell, the duration of the test and the exposed surface area of the test specimen. Area specific emission rates at a given lapsed time (t) can also be expressed as a function of the emission cell air concentrations for each VOC and the area specific air flow rate, q.
- 6.1.2 Air velocity at the surface of the test specimen (Appendix X3 and Appendix X4) is a critical parameter for the analysis of wet-applied indoor materials/products during the drying/curing stage when the dominant emission mechanism is evaporation (external diffusion) (see 6.4).
- 6.2 Using Emission Data to Estimate Contribution to Atmospheric VOC Concentration Indoors
- 6.2.1 Provided the area specific air flow rate over the surface of the test specimen is similar to that found in the built environment, and provided the surface of the indoor material/product is sufficiently homogeneous to ensure that the area of the test specimen exposed in the emission cell is representative of the whole; area specific emission rates determined by these

tests can be used to estimate the likely contribution to atmospheric VOC concentrations from that indoor material/product in real use, at time (t) after installation/application (assuming similar nominal conditions of temperature and humidity).

6.3 Intercomparison of Emission Data

6.3.1 Provided the test conditions are duplicated, area specific emission rate data generated from these tests may be used for comparison with area specific emission rate data produced for the same or similar products by other laboratories using similar emission cells.

NOTE 7—The principles described in 6.2 and 6.3 are true for all applicable product types, whatever the dominant process of emission.

6.4 Effect of the Emission Mechanism on Test Data

- 6.4.1 Provided the dominant emission mechanism is (internal) diffusion, not evaporation (external diffusion), area specific emission rate data will be broadly independent of air velocity over the surface of the indoor material/product. This will remain true provided the surface air velocity exceeds the minimum velocity required to prevent build up of vapor-phase contaminants at the surface of the indoor material/product (see Appendix X4).
- 6.4.2 Provided the dominant emission mechanism from a material/product is internal diffusion, it is therefore possible to compare area specific emission rates generated from emission cells under different air flow conditions or to compare area specific emission rate data generated by emission cells with that obtained using test chambers (D5116 or EN/ISO DIS 13419-1.) (see Appendix X4).

Note 8—Evaporative emissions predominate during the drying/curing stages of wet-applied products and are significantly effected by the following factors: sample conditioning procedures; timing of the test; loading factor (and related vapor concentration within the cell); and air velocity over the test specimen surface. Comparative tests on wet-applied products during the drying/curing stage should therefore be carried out using identical equipment, conditions and procedures and using an air velocity which approximates to that seen in real-world use (1-3)³. These restrictions apply in principle to both cells and small chambers.

Note 9—Emissions testing of wet-applied materials/products is typically carried out after the drying/curing stage, when internal diffusion-controlled emission processes predominate. This is more representative of real-world use. People or animals are unlikely to occupy a room or building until wet-applied coatings have dried or cured.

6.5 Precautions for Inhomogeneous Materials

6.5.1 If the indoor material/product under test is not homogeneous (for example, natural wood), the test will have to be repeated for multiple test specimens of the same material in order to establish a mean area specific emission rate. This is also true in principle for emissions testing using small chambers.

6.6 Controlling Key Test Parameters

6.6.1 In order to produce meaningful area specific emission rate data from emission cells or chambers (large or small), the following key parameters must meet minimum performance criteria:

³ The boldface numbers in parentheses refer to the list of references at the end of this standard.

- 6.6.1.1 Background interferences (see 8.2).
- 6.6.1.2 Control of humidity and temperature (see 8.1).
- 6.6.1.3 Control of the air velocity at the surface of the test specimen (see 7.3 and Appendix X3) and throughout the emission cell such that there are no unswept volumes (volumes of still air) and ideally such that it closely matches that expected under real use conditions. The latter is particularly relevant during evaporative (external) diffusion.

Note 10—The relationship between air flow rate into the emission cell and air velocity at the surface of the test specimen, for the type of emission cell described in Appendix X2, is discussed in Appendix X2 and Appendix X3.

- 6.6.2 Examples are presented in Table X2.1. Typical inlet air flow rates are in the order of 200 to 1400 mL/min, for the type of emission cell described in Appendix X2 (see 8.3).
- 6.6.2.1 Thorough mixing of the air such that the concentration at the sampling (exit/exhaust) point is representative of the air within the emission cell (see 7.3).
 - 6.6.2.2 Air leaks (see 7.4).
- 6.6.2.3 Recovery and sink effects (see 7.6 and Appendix Appendix X5).

7. Apparatus

NOTE 11—General specifications and requirements, which apply to all types of emission cells, are given in 7.1 to 7.7 below. An example emission cell is described in Appendix X2.

Note 12—Quality assurance/quality control activities shall be carried out as described in Annex A1.

- 7.1 Introduction
- 7.1.1 A complete emission cell system, designed and operated to determine area specific emission rates of VOCs from building products, shall comprise the following key components: emission cell, clean air generation and humidification system and monitoring and control systems (to ensure that the test is carried out in accordance with specified conditions). Appropriate sample tubes are also required.

Note 13—Analysis of VOC or carbonyl samples collected from emissions cells can be carried out remotely.

- 7.1.2 For rigid (non-compressible) materials/products with a smooth planar surface, the emission test cell is placed directly onto the surface of the test specimen. Other compressible or textured products shall be placed in specially constructed test specimen holders (see 7.7).
 - 7.2 Emission Cell Construction Materials
- 7.2.1 The emission cell itself and all parts of the sampling system that come into contact with emitted VOCs (all tubing and couplings) are normally made of surface treated (polished) stainless steel or glass. However, in all cases the requirements specified in 7.3 and 7.5 shall be fulfilled.
- 7.2.2 The sealing material (gasket or o-ring) which links together the emission cell and the test specimen (or test specimen holder) shall be low emitting and low absorbing and shall not contribute to the emission cell background concentration. It shall be easy to remove and replace the o-ring or gasket to facilitate cleaning of the emission cell.
 - 7.3 Air Supply and Mixing Facilities
- 7.3.1 The emission cell shall be supplied with pure and humidified air and have a device for controlling the air flow

rate with an accuracy of ± 3 %. The air velocity in the emission cell shall be distributed over the entire test specimen surface (see Appendix X3). There should be no volumes of still air within the cell. Inlet air flow rates between 200 and 1400 mL/min are typical for the type of emission cell described in Appendix X2 (see 8.3).

7.4 Air Leaks

- 7.4.1 The emission cell shall be operated above atmospheric pressure to avoid any influence from the laboratory atmosphere. This is achieved by ensuring that the sum of the pump sampling flows is <90 % (typically 75-80 %) of the inlet flow rate. The excess air shall exit through an exhaust vent.
- 7.4.2 The emission cell is considered sufficiently leak-free if the inlet air flow differs from the sum of the outlet air flows (pump flow rates plus exhaust flow) by less than 10 %. This should be checked at the start of every recovery test (see 7.6), background test (see 12.1) and emissions test (see 12.6).
- 7.4.3 Indoor materials/products which are permeable to air, shall be placed in air tight test specimen holders or sealed on to air tight, inert base plates to avoid permeation through the back of the test specimen.
 - 7.5 Air Sampling
- 7.5.1 The air at the emission cell outlet shall be used for sampling. Sampling of the outlet air (for example, with a sampling pump) is achieved by connecting sample tubes to the outlet couplings of the emission cell. The sum of sampling air flows shall be less than 90 % (typically 75-80 %) of the inlet air flow to the emission cell. The excess air shall exit through an exhaust vent.
- 7.5.2 A multiport sampling manifold can provide the flexibility for duplicate air sampling. The sampling manifold shall enter directly into the outlet air stream of the emission cell. If a duct is used, it shall be as short as possible and maintained at the same temperature as the emission cell.

Note 14—The exhaust from the emission cell should be ducted into a fume hood, ensuring that any chemicals emitted from the test specimen are isolated from the laboratory environment.

- 7.6 Recovery and Sink Effects
- 7.6.1 The recovery of a target VOC can be determined using a VOC source of known specific emission rate in the emission cell. This test can be carried out, for example, using permeation, or diffusion-controlled, vials of pure liquid VOC_x, sunk into a polished steel or glass plate and placed centrally under the emission cell (see Appendix X5). The concentrations generated shall be of similar magnitude to those expected during the emission tests of indoor materials/products.

Note 15—If a compound more volatile than toluene is being tested for recovery, it may be necessary to cap the vial, for example with a ground glass plug with a capillary hole through it, to reduce the rate of emission.

7.6.2 Recovery tests shall be performed in the emission cell using at least toluene and n-dodecane (see Note 17). An example polar compound such as ethylhexanol should also be used if any of the target VOCs are polar. Emission cell air concentrations shall be determined several times during the recovery test, typically 24-72 h after start of the test (see Note 19). Emission cell air concentrations shall be determined using the procedure outlined in 12.6 and an average air concentration established. At the end of the recovery test, the actual weight

loss of the permeation, or diffusion-controlled, emission source, shall be measured and used to generate an expected average vapor concentration in the emission cell during the recovery test. The experimentally determined mean emission cell concentration of vapor must be at least 90 % of that expected from the actual weight loss of the emission source.

7.6.3 The results of this recovery test shall be reported in the test report as concentration expected versus concentration measured.

Note 16—Dehumidified air should be present in the case of determination of the recovery of hygroscopic VOCs such that weight measurements are not affected by the sorption of water. (This may not be necessary for permeation tubes, depending on the material of construction of the permeation tube).

Note 17—Failure to meet the minimum recovery requirements can be the result of leaks, poor calibration of the analytical system, and, possibly, sink/wall effects, especially for polar high boiling VOCs. It is a test of the performance of the whole procedure. Sink and adsorption characteristics are very much dependent on the type of compound emitted. Additional recovery tests using target VOCs with different molecular weight and polarity can be used to increase understanding of these effects.

Note 18—Emission cells as described in Appendix X2 have an exit centrally located, immediately above the surface of the test specimen and have polished, inner walls formed into a shallow trumpet shape which narrows towards the vapor exit. This reduces turbulence and helps eliminate volumes of still air thus minimizing risk of condensation and sink effects

Note 19—Recovery tests can be carried out over shorter periods of time (for example, 1-24 h) for more volatile analytes.

7.7 Test Specimen Holders

7.7.1 Test specimen holders shall be designed such that the weight of the emission cell is carried by the rim of the test specimen holder, not the material itself. Test specimen holders shall have a flat rim with dimensions such that the emission cell o-ring or gasket seals effectively onto the rim of the holder. The internal dimensions of the holder shall match that of the exposed area of the test specimen within the cell. The depth of the test specimen holder shall be adjustable such that the test specimen surface can be raised or lowered to an appropriate height so that the internal volume of the emission cell is unaffected (relative to its volume when placed directly onto the surface of rigid products.)

7.7.2 Preparation of test specimens for insertion into test specimen holders, in such a way as to minimize edge effects, is described in 10.4.

Note 20—Typical test specimen holders for the type of emission cell described in Appendix X2 comprise cylinders with an internal diameter of 15 cm and with adjustable depth. They also have a flat rim, >1 cm wide, with an appropriate diameter to match that of the sealing o-ring of the emission cell.

7.8 Equipment

- 7.8.1 The apparatus necessary for carrying out tests with an emission cell are listed below:
- 7.8.1.1 clean air supply (for example, pressurized purified air or synthetic air in gas cylinders),
 - 7.8.1.2 emission cell system,
 - 7.8.1.3 humidification system,
 - 7.8.1.4 humidity and temperature monitoring systems,
- 7.8.1.5 capped, conditioned sample tubes containing one or more sorbents. [Tenax TA sorbent is most commonly used and

is suitable for most compounds ranging in volatility from n-hexane to n-hexadecane. Further advice on sorbent selection (for example, for more or less volatile compounds) and on the preparation and use of sorbent tubes for thermal desorption is given in Practice D6196, ISO/EN 16017-1 and US EPA Method TO-17.] Each measurement typically requires four or five identical sample tubes: one or two for collecting a sample from the blank emission cell as a background check (see 12.1), two for air sample collection during the emissions test (see 12.5), and one to be used as a blank, and

Note 21—A suitable internal standard (for example deuterated toluene) can be added to blank tubes prior to air sample collection if desired. Recovery of the internal standard, determined during subsequent TD-GC-MS analysis, can then be used as a check on the entire process of sample tube storage, air sample collection and analysis (see A1.4)

7.8.1.6 two sampling pumps capable of pumping flows of 80-500 mL/min through sorbent tubes and calibrated in accordance with Practice D5337. Constant flow type pumps, offering electronic mass flow control, are recommended.

7.8.1.7 one or more calibrated air flow meters.

7.8.1.8 suitable test specimen holders (if required, see 7.1).

7.8.1.9 facilities for recovery testing (see Appendix X5).

7.8.1.10 Either a cleaning agent for the emission cell or a vacuum oven for heating and cleaning the emission cell.

Note 22—The apparatus listed here is for sampling general VOCs and is described in more detail in Practice D6196. Alternative sampling apparatus for formaldehyde and other carbonyls is described in Test Method D5197.

8. Test Conditions

- 8.1 Temperature and Relative Humidity
- 8.1.1 Temperature and relative humidity inside the emission cell shall be $23 \pm 2^{\circ}\text{C}$ and 50 ± 5) % RH during the emission test (see ISO 554) unless there is some specific alternative testing requirement (for example, if the indoor material/product is used at elevated temperatures in real world applications, or if it is only used in dry (for example, desert) environments).
- 8.2 Quality of Supplied Air and Background Concentrations of VOCs
- 8.2.1 Supplied air shall not contain any VOCs at levels greater than the emission cell background requirements.
- 8.2.2 The sum of VOC (TVOC) (see Practice D6330) background concentration shall be lower than 20 μg^{m-3} . The background concentration of any single target VOC shall be lower than 2 μg^{m-3} .
- 8.2.3 Emissions tests for specific target VOCs can be carried out at lower concentrations, but background levels of target compounds shall be shown to be below 10 % of measured emission cell concentrations in all cases.
- 8.2.4 Water used for humidification shall not contain interfering VOCs.
 - 8.3 Air Velocity
- 8.3.1 The air velocity over the surface of the test specimen shall be in the range of 0.003 m/s⁻¹ to 0.1 m/s⁻¹(see Appendix X2 and Appendix X3) which, for the type of emission cell described in Appendix X2, will require an inlet air flow rate in the range 100 to 2800 mL/min (see Table X2.1). Typical inlet

air flow rates are in the range 200 to 1400 mL/min with associated tube sampling rates of 80 to 500 mL/min, respectively.

Note 23—Air velocity is generally significant for evaporative controlled (external diffusion) emissions, for example, from liquid products before they have dried/cured (see Section 6). This depends on the substrate.

Note 24—For certain types of material/product, secondary source emissions can occur at high air velocities.

Note 25—Examples of air velocity calculations are given in Appendix X2 and Appendix X3.

- 8.4 Relationship Between Air Flow Rate, Air Change Rate, and Vapor Concentrations
- 8.4.1 Vapor concentrations inside the emission cell depend on air flow rate, the exposed surface area of the test specimen and the area specific emission rate.
- 8.4.2 The air change rate is simply a function of the volume of the cell and the air flow rate and has no independent impact on VOC emissions or vapor concentrations inside the emission cell.

Note 26—Example air change rates for one type of emission cell are shown in Table X2.1 of Appendix X2.

9. Verification of Test Conditions

- 9.1 General
- 9.1.1 All control measures shall be traceable to a certified standard in accordance with the quality assurance and quality control schemes (see Annex A1).
 - 9.2 Temperature and Relative Humidity Control Systems
- 9.2.1 Control of temperature can be made by placing the emission cell within a location controlled to the required temperature.
- 9.2.2 Control of relative humidity can be made by various systems (for example, using integrated humidification of the air supply).
- 9.2.3 Temperature and relative humidity shall be measured independently of the systems for controlling the temperature and relative humidity.
 - 9.3 Test Conditions in the Emission Cell
- 9.3.1 Temperature, relative humidity, and air flow rate shall be measured with the following accuracy:
 - 9.3.1.1 temperature ± 0.1 °C,
 - 9.3.1.2 relative humidity ± 3 % RH, and
 - 9.3.1.3 air flow rate $\pm 3\%$.
- 9.3.2 The relative humidity shall be measured at the outlet from the emission cell. The temperature sensors shall be placed either in the emission cell or in the air outlet.
 - 9.4 Air Flow Rate and Air Velocity in the Emission Cell-
- 9.4.1 The inlet and outlet air flow rates shall be checked and readjusted prior to air sampling using a non-restricting calibrated gas flow meter. The air flow rates shall not vary by more than ± 3 % of the set value. For the type of emission cell described in Appendix X2, typical (inlet) air flow rates are in the range of 200-1400 cm³/min⁻¹ and typical pumped sample collection flow rates are in the order of 80-500 cm³/min⁻¹. The air velocity in the emission cell can be calculated from the air flow rate (see Appendix X2 and Appendix X3).

Note 27-If the test is carried out with a gas volume meter/flow meter,

which is not permanently installed, be aware that the back pressure, introduced by the meter, can lower the flow rate through the emission cell.

- 9.5 Air Leaks Into or Out of the Emission Cell
- 9.5.1 Air leaks into or out of the emission cell shall be checked at the beginning of every recovery, background and emissions test (see 7.4).

10. Test Specimens

- 10.1 Studies of the emission of volatile organic compounds from unused indoor materials/products in emission cells require proper handling of the test specimen prior to testing, and during the testing period.
- 10.2 This practice is generic and can be applied to many different indoor materials/products. Types of indoor materials/products are defined as solid, liquid and combined. For each type of product the sampling procedure, transport conditions, storage, and substrate used (which can all affect emissions) shall be specified in the test report. For individual materials/products within each product class, the preparation of the test specimen is prescribed in Annex A2 and Annex A3.

NOTE 28—Depending on the inhomogeneity of the material/product, it can be necessary to make measurements on multiple test specimens from the same sample to determine the mean specific emission rate (see 6.5).

- 10.3 Product Sampling and Sample Transport/Storage
- 10.3.1 Sampling of Indoor Materials/Products to be Tested
- 10.3.1.1 Samples of materials/products collected at the point of manufacture shall be taken from the normal manufacturing process. Product samples can also be collected from retail stores.
 - 10.3.2 Sample Packaging and Transport
- 10.3.2.1 Samples shall be thoroughly protected from chemical contamination or any physical exposure (for example, heat, light, humidity, and contaminated atmospheres). For solid products, this can usually be achieved by shipping the product inside the manufacturers packaging or, if this is too large for practical purposes, by selecting a sample (Annex A2), wrapping it separately in aluminum foil and then placing it in a polyethylene bag. Alternatively, each sample can be wrapped in aluminized packaging lined with polyethylene or clear polyvinyl fluoride film. Samples should be wrapped within one hour of selecting them at the production line. Liquid products shall be shipped in unopened cans, tubes, and so forth. (see Annex A3).

NOTE 29—Transportation of collected samples can affect the emission characteristics of the product. The possible effects of temperature, humidity and high VOC levels are of particular concern.

10.3.3 Sample Description

- 10.3.3.1 The sample shall be labeled with the details of the source (store or manufacturer), source location, type of product, date of manufacture (if known), any identification numbers, (for example, batch numbers), and details of the complete chain of custody between source and receipt at the laboratory.
 - 10.3.4 Sample Storage Prior to Testing
- 10.3.4.1 In many cases it is necessary to store the sample in the laboratory before starting the test. The sample shall be kept in its packaging (see 10.3.2), and stored under normal indoor conditions (23 \pm 5°C, 50 \pm 5 % RH) before testing begins.

Note 30—Storage may affect the emission properties due to ageing of the sample even if it is well wrapped. It is recommended to minimize the storage time of the sample before it is prepared for emissions testing.

10.4 Test Specimen Preparation

10.4.1 The procedures to be used for preparing different types of sample/test specimen for emissions testing are prescribed in Annex A2 and Annex A3 and described in Appendix X6. The period of time between unpacking the sample and preparation of the test specimen shall be as short as possible and shall be recorded. After preparation of the test specimen, it shall immediately be put in conditioned storage in accordance with Sections 8 and 12.5 or under the emission cell itself. This time shall be regarded as the starting time of the emission test (that is, $t = t_0$).

10.4.2 If the indoor material/product is compressible or textured and requires a test specimen holder, the size of the test specimen taken should be such that it is a tight fit inside the test specimen holder to eliminate edge effects. Once a test specimen has been placed inside a holder at time t_0 it should be stored in that specimen holder (either under the emission cell or in the conditioned storage area) throughout the duration of the emissions test.

10.4.3 If the material/product is permeable, secure the underside of the test specimen to a sheet of clean glass or stainless steel using non-contaminating aluminum tape (see A2.3.1).

11. Emission Cell Preparation

11.1 The emission cell shall be cleaned in accordance with either 11.2 or 11.3.

Note 31—It will be necessary to remove the sealing material (gasket or o-ring) before cleaning the emission cell by either method.

11.2 Cleaning Using a Detergent

11.2.1 The emission cell can be cleaned by washing the inner surface with a diluted alkaline detergent, followed by two separate rinsings with freshly distilled water. The inner surface is then washed with non-denatured ethanol or another appropriate solvent.

11.3 Cleaning by Thermal Desorption

11.3.1 The emission cell can also be cleaned by heating in a vacuum oven at elevated temperature (70°C to 100°C) for approximately two hours.

12. Test Method

12.1 Measuring Background Concentrations

12.1.1 An air sample of the emission cell background is taken before the start of an emission test to quantify any background contribution of volatile organic compounds from the air supply or from the emission cell apparatus itself.

12.1.2 Place the emission cell, complete with its sealing material (o-ring or gasket) on a clean and planar surface (for example, a glass or stainless steel plate). Set the air flow to a similar rate to that to be used for emissions testing and flush the emission cell with clean, humidified air, to vent, for approximately 15 minutes. Check for leakage in accordance with 7.4. Connect one or two conditioned sample tube and pump assemblies to the outlet ports of the emission cell, setting similar sample flows to those to be used for materials emissions

testing (see 12.6). Check the inlet and sampling air flows quickly at the beginning of the background test (see 7.4).

12.1.3 Background concentrations shall meet the requirements in 8.2.

12.2 Test Specimen Location in the Emission Cell

12.2.1 If the indoor material/product to be tested is not to be stored under the emission cell, it must be placed (or replaced) under the emission cell at least 15 minutes prior to air sample collection (in the case of most dry materials/products) or at least two hours prior to air sample collection whenever the test specimen is a dried/cured coating or target compounds include highly polar species. The supply of clean humidified air must be turned on as soon as the emission cell is placed over the test specimen. The positioning of the emission cell shall ensure that the air flow is distributed over the entire emitting surface of the test specimen.

Note 32—The action of placing the emission cell on the surface of the product or material must not distort the test specimen surface. If the indoor material/product is compressible or textured, it must be placed in a suitable test specimen holder such that the weight of the cell seals onto the rim of the test specimen holder not onto the product itself (see 7.1).

12.3 Preparation for Air Sample Collection

12.3.1 The inlet air flow rate should be selected based on the target air velocity or area specific air flow rate. For the type of emission cell described in Appendix X2, example inlet air flow rates and respective air velocities and area specific air flow rates are given in Table X2.1. Two conditioned and calibrated sample tube and pump assemblies should be connected to the outlet ports of the emission cell. When the pumps are switched on, this marks the beginning of air sample collection. Quickly check for leaks (see 7.4).

Note 33—In order to ensure that the air inside the test cell is maintained at slight positive pressure, the sum of the sampled air flows must not exceed 90 % of the inlet air flow. This means that if the inlet air flow rate is required to be set at 200 mL/min, the two air samples are typically collected at 80 mL/min. Similarly if the inlet air flow rate is required to be set at 500 mL/min, the two air samples are usually collected at 200 mL/min.

12.4 Time for Measurements of Emission Cell Air Concentration

12.4.1 The concentration measurements shall be carried out at predefined sampling times, depending on the objective of the test. Emission test duration is determined by the objective of the test. (More information on test duration is given in 12.6.)

Note 34—Typical sample collection times are 2 hours, 24 hours, 72 hours, 10 days, 28 days, 56 days, and 182 days (26 weeks) after preparation of the test specimen at time t_0 .

Note 35—The mid-point of the period of air sample collection is considered the sampling time.

12.5 Storage of Test Specimen between Emission Tests

12.5.1 Throughout the entire test period, test specimens shall either be kept under the test cell under the flow of pure humidified air, or stored in clean, well-ventilated environments, under controlled conditions of temperature and humidity, with minimal risk of contamination from other samples or other emission sources (see 8).

12.6 Air Sample Collection

- 12.6.1 Two air samples shall be collected at each sampling time whenever practicable. The duration of each air sample collection period depends on the analytical methods to be used and shall be documented. Typical air sample collection periods for general VOCs range from 15 minutes to 2 hours and typical pumped sample flow rates used for air sample collection range from 80 to 500 cm³/min⁻¹. Different pump flow rates within this range may be used to sample onto each of the two sample tubes attached to the emission cell, provided that the actual period of sample collection is the same.
 - 12.7 Sealing the Sample Tubes after Air Sample Collection
- 12.7.1 Sampling pumps shall be switched off at the end of air sample collection and the duration of air sampling recorded. Sample tubes shall be disconnected from the sample pumps and emission cell immediately after air sample collection and quickly resealed using appropriate caps and fittings (see Practice D6196 and Test Method D5197). Store the sample tubes carefully before analysis following guidance given in Practice D6196 or Test Method D5197, appropriately.
 - 12.8 Cleaning the Emission Cell after Use
- 12.8.1 At the end of air sample collection, the emission cell shall be cleaned in accordance with Section 11.

13. Calculation of Area Specific Emission Rates (SER_a) and Expression of Results

- 13.1 Calculation of SER_a from experimental parameters and data—assuming negligible background
- 13.2 Parameters known or determined during the emission test are as follows:
- 13.2.1 A = Surface area of test specimen exposed to the clean humidified air (cm² = (m² × 10^4))
- 13.2.2 M_1 and M_2 = Mass of each target VOC_x retained by the first and second sample tubes respectively ($\mu g = ng \times 10^{-3}$)—Determined using a calibrated thermal desorption-GC-MS/FID analytical system and following Practice D6196, ISO 16000-6, ISO 16017-1, or equivalent. Alternatively, use Test Method D5197 for formaldehyde
- 13.2.3 F = Flow rate of pure, humidified air into the cell $(\text{cm}^3.\text{min}^{-1} = (\text{m}^3/\text{h}^{-1} \times 10^5/6))$
- 13.2.4 F_1 and F_2 = Pump air flow rates through the first and second sample tubes respectively (cm³.min⁻¹ = m³/h⁻¹ × 10⁵/6)
 - 13.2.5 T = Duration of test (min = $h \times 60$)
- 13.2.6 V = Volume of air (cm³ = (m³ × 10⁶)) passed into the cell during the emission test = F × T
- 13.2.7 V₁ and V₂ = Volumes of air (cm³ = (m³ × 10⁶)) pumped through the first and second sample tube respectively = $F_1 \times T$ and $F_2 \times T$, respectively
- 13.2.8 C_x = Concentration of x vapor within the emission cell (ng/cm⁻³ = μ g/m⁻³ \times 10⁻³) = mean of that determined from sample Tube 1 (M₁/V₁) and that determined from sample Tube 2 (M₂/V₂)

Note 36—This practice requires the use of two parallel sample tubes monitoring the exhaust gases from the emission cell, whenever practicable. $C_{\rm x}$ derived from sample Tube 1 shall agree within 10 % to that derived from sample Tube 2 or the test must be repeated.

- 13.2.9 Assuming negligible background of each target VOC, the area specific emission rate SER_a for VOC_x can be simply derived from these parameters as follows:
- 13.2.10 Mass of x (μ g) emitted by the exposed area of sample during the emissions test (M_x) is derived as follows:

$$M_x = C_x$$
 (that is, mean of M_1/V_1 and M_2/V_2) $\times V$ (1)

13.2.11 To derive the mass of x (μ g) emitted by the exposed area of sample per hour, multiply M_x by 60 and divide by the actual duration of the test in minutes:

$$= M_{\rm v} \times (60/T) \tag{2}$$

13.2.12 To derive the area specific emission rate, at time (t), in $\mu g/m^{-2}/h^{-1}$, multiply the result from Eq 2 by 10 000 (cm²) and divide by the actual test specimen area exposed (cm²)

$$SER_a = M_x \times (60/T) \times 10\ 000/A$$
 (3)

Note 37—The actual test specimen surface area exposed in the type of emission cell described in Appendix X2 is 177 cm².

- 13.3 Background Correction of SER_aData
- 13.3.1 To correct for background concentrations of VOC_x , derive the effective area specific emission rate ($\mu g/m^2/h^{-1}$) for the blank emission test (see 12.1) following the steps described in 13.1 and subtract this from the experimentally derived area specific emission rate for the test specimen.

Note 38—High background concentrations of target VOCs will invalidate the emissions test (see 8.2).

13.4 Deriving Units for Comparison with Small Chamber Emission Tests

- $L = \text{surface area } (m^2) \text{ of test specimen exposed in cell/cell } volume <math>(m^3)$
- n = air change rate (changes per hour) = air flow rate entering cell (cm³/h⁻¹)/volume of cell (cm³)
- q = area specific air flow rate = n/L
- 13.5 Deriving SER_a Using Analogous Methods to Those Used for Small Chamber Emissions Tests
- 13.5.1 At a given test condition, C_x depends on the area specific emission rate of the test specimen and the air flow rate through the emission test cell. For individual VOCs, the compounds found both in the material and in the background shall be subtracted compound by compound. For TVOC the measured background shall be subtracted. The relation between C_x , the area specific emission rate (SER_a) and the area specific air flow rate (q) of the emission test cell can be expressed as:

$$C_x = SER_a \cdot (L/n) = SER_a/q$$
 (4)

13.5.2 Eq 4 shows that the area specific air flow rate equals the n/L ratio. For a given product tested under given emission test cell conditions, the concentration of VOC_x depends on the area specific air flow rate. C_x is the mean concentration of VOC_x calculated from a duplicate air sample as described in 13.1.

$$SER_a \times = C_x \cdot q \text{ at time (t)}.$$
 (5)

13.6 Relating SER_a to the Time of the Emission Measurement

13.6.1 The result shall be related to the time of the emission measurement after t_0 and may be reported quantitatively as the area specific emission rate, of individual VOCs, or TVOC, or both, at time (t).

Note 39—The sum of emitted compounds, TVOC (see Practice D6330), should be regarded only as a factor specific to the product studied and to the monitoring method used. It is therefore only to be used for comparison of products with similar target VOC profiles if exactly the same test method has been used for each comparative measurement

14. Test Report

- 14.1 The test report shall include the following information:
- 14.1.1 Test Laboratory:
- 14.1.1.1 name and address of the laboratory,
- 14.1.1.2 name of the responsible person,
- 14.1.1.3 reference to this practice, and
- 14.1.1.4 reference to the appropriate in-house protocol or detailed description of the equipment and methods used (that is, test cell, clean air system, environmental control, sample collection, analytical instrumentation, standard generation and calibration);
 - 14.1.2 Sample/Test Specimen Description:
 - 14.1.2.1 type of product (and brand name if appropriate),
 - 14.1.2.2 sample selection process (for example, random),
- 14.1.2.3 product history (that is, date of production, date of arrival to the test laboratory), and
 - 14.1.2.4 description of packaging.
 - 14.1.3 Test Specimen Preparation:
- 14.1.3.1 date and time of unpacking and test specimens preparation [hour, day, month and year]), and
- 14.1.3.2 method of preparation, including thickness and substrate.
 - 14.1.4 Experimental Conditions and Procedures:
- 14.1.4.1 test cell conditions (that is, temperature, relative humidity, air change rate, air velocity),

- 14.1.4.2 test specimen area and loading ratio (for liquid products, for example, paint, describe sample substrate and coating procedure, paint thickness and coating density [net mg/cm²]),
- 14.1.4.3 sampling of emitted compounds (that is, adsorbent used, volume sampled, sampling duration and times after introduction into the cell), and
- 14.1.4.4 analytical conditions used (that is, thermal desorption parameters, GC column selected, GC-MS conditions, and so forth).
 - 14.1.5 Data Analysis:
- 14.1.5.1 describe the method used to derive specific emission rates from measured cell concentrations (specify mathematical models or equations used).
 - 14.1.6 Results:
- 14.1.6.1 specific emission rates shall be reported for each test specimen, for individual VOCs, or TVOC, or both, at the times of air sampling.
 - 14.1.7 Quality Assurance/Quality Control:
- 14.1.7.1 background test cell concentrations of target compounds,
- 14.1.7.2 recovery data of toluene and n-dodecane (to evaluate the overall performance),
- 14.1.7.3 recovery data for the three most significant target VOCs.
 - 14.1.7.4 results of duplicate sampling/analysis, and
- 14.1.7.5 quality of the environmental variables (that is, temperature, relative humidity, air change rate, air velocity).

15. Keywords

15.1 emissions cells; indoor air quality; indoor materials; indoor products; materials emissions testing; organic emissions; volatile organic compounds

ANNEXES

(Mandatory Information)

A1. SYSTEM FOR QUALITY ASSURANCE/QUALITY CONTROL

A1.1 General

A1.1.1 Emission cell testing of organic emissions from indoor materials/products shall be conducted within the framework of a Quality Assurance Project Plan (QAPP). The QAPP shall contain a project description, data quality objectives/acceptance criteria, QA/QC approaches/activities, and QA/QC audits.

A1.2 Project Description

A1.2.1 A brief description shall include what materials are to be tested; how the testing is to be conducted; and who is responsible for various project activities. The project experimental design shall contain the necessary information for this portion of the QAPP.

A1.3 Data Quality Objectives/Acceptance Criteria

A1.3.1 This section of the QAPP defines the precision, accuracy, and completeness desired for each parameter being measured.

A1.4 QA/QC Approaches/Activities

- A1.4.1 The types of QA/QC activities that can be specified in the QAPP include establishment of a system of records/notebooks to ensure proper operation of equipment and recording of data, such as:
- A1.4.1.1 sample log to record receipt, storage, and disposition of materials,
- A1.4.1.2 GC standards preparation log to document preparation of all organic compound substances,

A1.4.1.3 Permeation/diffusion tube log to record weight loss data for all permeation/diffusion tubes used during recovery measurements,

A1.4.1.4 calibration logs to contain environmental systems calibration data.

A1.4.1.5 instrument maintenance logs to document maintenance and repairs of all equipment,

A1.4.1.6 materials testing logs in which to record all pertinent information for each test, including sample details, sample ID number, and GC run ID number,

A1.4.1.7 sorbent cartridge cleanup/desorption log detailing thermal cleanup and QC validation of sorbent cartridges,

A1.4.1.8 floppy disk storage log to document location and content of electronically stored data, and

A1.4.1.9 manuals governing operation of all equipment used by the project.

A1.4.2 QC activities are carried out by project staff in a routine, consistent manner to provide necessary feedback in operation of all measurement systems. Such activities are

described in this and associated standards such as Practice D6196 or Test Method D5197 and can include:

A1.4.2.1 routine maintenance and calibration of equipment and analytical systems used,

A1.4.2.2 daily recording of GC calibration accuracy and precision (that is, control charting),

A1.4.2.3 monitoring of percent recovery of internal standard, if added, to blank or sampled tubes,

A1.4.2.4 correlation of data from duplicate samples,

A1.4.2.5 QC checking of the blank profile of organic collection sorbent tubes, and

A1.4.2.6 periodic analysis of audit gases or certified standard reference tubes (pre-loaded with traceable masses of target VOCs) supplied by an independent source.

A1.5 QA/QC Audits

A1.5.1 Finally, the QA/QC program shall include periodic audits by QA personnel to evaluate compliance with QAPP protocols.

A2. SOLID PRODUCTS—PROCEDURE FOR SAMPLING AND TEST SPECIMEN PREPARATION

A2.1 General

A2.1.1 This method covers only unused products.

A2.2 Sampling

A2.2.1 Selection of Samples from Rolls

A2.2.1.1 Discard one metre or at least the outer layer of the roll to take the sample (Fig. A2.1). Cut out a large sample (typically >1.2 m²)

A2.2.1.2 Roll the sample up in the same direction as the original production roll and make sure that the roll is several layers thick. Secure with staples, wrap it in aluminum foil, and seal it in an unprinted, air tight (sealable) polyethylene bag. Each bag shall contain only one sample. Minimize the air space within the package. An alternative approach is to cut several 60 by 60 cm (or larger) squares and stack at them at least four high, before wrapping the set in aluminum foil. This approach helps minimize the volume of air inside the package. Not more than 1 h shall elapse from the time of selecting the test piece to placing it in the polyethylene bag. Pack the samples carefully and send them straight to the testing laboratory.

A2.2.2 Selection of Samples of Rigid Products

A2.2.2.1 Send an unopened standard package of the material/product to the testing laboratory. Boards are transported in the normal manufacturer's delivery package unless the delivery package is large and impractical to handle. In this case a sample may be taken from the middle of a board and enclosed in air tight wrapping (see A2.2.1) for more convenient transportation. Tiles, laminated parquets and other products assembled from pieces are transported to the laboratory for emission testing in their original packaging.

A2.3 Preparation of Test Specimens _____7143_05

A2.3.1 Preparing Test Specimens from Samples from Rolls A2.3.1.1 Unpack the sample and select an appropriate area of the material/product at least 50 cm from any edge and cut out a test specimen (see Fig. A2.2). If a stack of multiple samples of the same product has been packaged together as described in A2.2.1, use one of the sheets from the center of the stack and cut the test specimen at least 10 cm from any edge. The area of the test specimen must be large enough for the

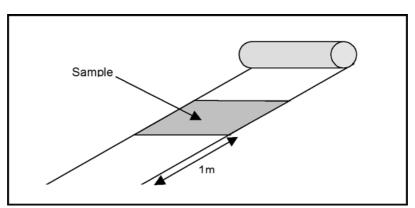


FIG. A2.1 Selection of Samples from Rolled Materials/Products