

Designation: D8345 – 21

Standard Test Method for Determination of an Emission Parameter for Phthalate Esters and Other Non-Phthalate Plasticizers from Planar Polyvinyl Chloride Indoor Materials for Use in Mass Transfer Modeling Calculations¹

This standard is issued under the fixed designation D8345; 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 Planar polyvinyl chloride (vinyl) indoor materials can contain semi-volatile organic compounds (SVOCs), such as phthalate esters and other non-phthalate plasticizers, that can emit into indoor air. Phthalate esters and other non-phthalate plasticizers that have been measured using this standard are listed in Table 1 and are referred to as SVOCs in the remainder of this document.

1.2 The SVOCs listed in Table 1 are present in a wide range of products and not limited to planar polyvinyl chloride (vinyl) indoor materials. This standard discusses specific planar polyvinyl chloride materials due to method development and associated quality control data produced from testing these materials. The materials inclusion in this standard does not indicate the SVOC source strength of specific polyvinyl chloride planar materials relative to other products.

1.3 This method describes the design of a 1 L environmental chamber with minimal exposed chamber walls.

1.4 This method measures the steady-state gas phase concentration of SVOCs in the chamber. Samples of products are tested at specified conditions of temperature, airflow rate, and elapsed time in a specially designed chamber with dry air. Air samples are collected periodically using sorbent sampling tubes at the chamber exhausts at controlled flow rates, and then analyzed by thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS).

1.5 This method determines the SVOC convective gasphase mass transfer coefficient across the material surface, h_m , from the known dimethyl phthalate mass transfer coefficient in the chamber (1).²

1.6 Using the steady-state gas phase concentration and mass transfer coefficient, the method estimates the gas-phase con-

centration of SVOC in equilibrium with the material phase (y_0) at a specified temperature. The obtained y_0 data can be used to predict emissions in real indoor environments. However, exposure modeling is beyond the scope of this method. For more information on mass transfer emission and exposure modeling see Little et al. (2), Liang and Xu (1, 3), and Guo (4).

1.7 The results for gas phase concentration change in the chamber with time, steady-state gas phase SVOC concentrations (y_{ss}) , and y_0 , only represent the conditions specified in the test method and are the result of assumptions built into the method such as instantaneous equilibrium at the source/air interface. The results may not be representative of those collected under other test conditions (that is, temperature or flow rate) or comparable with other SVOC test methods.

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

1.9 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.

1.10 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:³
- D1193 Specification for Reagent Water
- D1356 Terminology Relating to Sampling and Analysis of Atmospheres

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¹ This test method 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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 $^{^{2}}$ The boldface numbers in parentheses refer to a list of references at the end of this standard.

³ 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.

- D5116 Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/ Products
- D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps
- D6007 Test Method for Determining Formaldehyde Concentrations in Air from Wood Products Using a Small-Scale Chamber
- D6177 Practice for Determining Emission Profiles of Volatile Organic Chemicals Emitted from Bedding Sets
- D6196 Practice for Choosing Sorbents, Sampling Parameters and Thermal Desorption Analytical Conditions for Monitoring Volatile Organic Chemicals 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
- D6803 Practice for Testing and Sampling of Volatile Organic Compounds (Including Carbonyl Compounds) Emitted from Architectural Coatings Using Small-Scale Environmental Chambers
- D7143 Practice for Emission Cells for the Determination of Volatile Organic Emissions from Indoor Materials/ Products
- D8141 Guide for Selecting Volatile Organic Compounds (VOCs) and Semi-Volatile Organic Compounds (SVOCs) Emission Testing Methods to Determine Emission Parameters for Modeling of Indoor Environments
- D8142 Test Method for Determining Chemical Emissions from Spray Polyurethane Foam (SPF) Insulation using Micro-Scale Environmental Test Chambers
- 2.2 EPA Methods:⁴
- Method 8270E Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)
- 2.3 ISO Methods:⁵
- 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 or MS-FID
- 2.4 CPSC Methods:⁶
- Test Method CPSC-CH-C1001-09.4 Standard Operating Procedure for Determination of Phthalates

3. Terminology

3.1.1 For definitions of terms commonly used for sampling and analysis of atmospheres, refer to Terminology D1356. For definitions and terms commonly used in volatile organic compound (VOC) emission testing from materials and products refer to Guide D5116. For definitions and terms commonly used in SVOC emission testing using a mass transfer framework see Guide D8141.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *loading factor, n*—the ratio of the exposed test specimen area to the chamber's volume, usually expressed in m^2/m^3 .

4. Summary of Test Method

4.1 A specially-designed stainless steel chamber is used to rapidly measure SVOC additive emissions from planar polyvinyl chloride materials. The chamber described in this method has a high area ratio of emission surface to sorption surfaces (that is, chamber walls), reduces mass loss of SVOCs onto sampling pathways, and improves air mixing inside the chamber. This chamber has a reduced time to reach steady-state of SVOC emissions compared to standard small chambers like those described in Guide D5116.

4.2 Specific planar polyvinyl chloride materials, vinyl flooring and mattress coverings, were used to develop this method and produce the precision data for this standard. As such, these materials are discussed in this standard. Identification of these materials does not indicate SVOC source strength relative to other planar materials.

4.3 Representative test materials are prepared and placed to form the top and bottom of the chamber. Clean dry air is supplied to the chamber and passes over the exposed surface of the test material before reaching the exhaust ports. Chamber conditions such as temperature and airflow rate are controlled.

4.4 Air samples are collected periodically using sorbent tubes at the chamber exhaust ports at controlled air sampling flow rates.

4.5 Sorbent tubes with air samples are analyzed by thermal desorption-gas chromatography/mass spectrometry (TD-GC-MS) to identify and quantify SVOC compounds. Information on TD-GC-MS analysis can be found in ISO 16000-6, Annex D. Information specific to SVOC analysis can be found in Liang and Xu (1).

4.6 Knowing the steady-state SVOC concentration in the chamber (y_{ss}) and the convective mass transfer coefficient (h_m) , the gas-phase concentration of SVOCs in equilibrium with the material phase (y_0) at specified temperature and flow rate can be quantified. The gas-phase concentration of SVOCs in equilibrium with the material phase (y_0) is needed to accurately model exposure to SVOCs in indoor environments.

5. Significance and Use

5.1 The conventional approach for characterizing VOC emissions from building materials and products (for example, Test Methods D6007 and D8142; Practices D6177, D6330, D6670, D6803, D7143; Guide D5116; and ISO 16000-6 standards) results in modeling VOC emissions in the indoor

^{3.1} Definitions:

⁴ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass-spectrometry.

⁵ Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

⁶ Available from United States Consumer Product Safety Commission (CPSC), 4330 East-West Highway Bethesda, MD 20814, https://www.cpsc.gov/Business--Manufacturing/Testing-Certification/Lab-Accreditation/Test-Methods.

environment using area-specific emission rates (μ g h⁻¹ m⁻²). These approaches work for most chemicals classified as VOCs, because most VOC emissions are controlled by the internal mass transfer processes (diffusion of the chemical through the material) and most VOCs sorb to minimal extent to chamber walls. Hence, chamber area-specific emission rates can be directly applied to models of indoor environments.

5.2 In contrast, chemicals classified as SVOCs will sorb strongly to chamber walls and are controlled by the external mass transfer process (migration through the air boundary layer on the material surface). When used for the equilibrium gas phase concentration of certain SVOCs above source materials, conventional chamber emission characterization approaches are typically time-consuming taking up to several months due to sorption of analytes to chamber walls. Due to SVOC's external mass transfer limitation, the SVOC area-specific emission rate (μ g h⁻¹ m⁻²) measured in a test chamber can be different from that for the same material in a real indoor environment. To accurately model SVOC concentrations in indoor environments, a mass transfer approach to determine gas phase concentrations in equilibrium with the material phase is needed.

5.3 Modeling emissions in a real environment using a mass transfer framework requires knowledge of the convective mass transfer coefficient (h_m) , the initial SVOC concentration in the material (C_{o}) , the diffusion coefficient in the material (D), and the concentration in the air immediately above the material surface (y_0) . Typically, the convective mass transfer coefficient, h_m , and diffusion coefficient, D, can be estimated. The initial concentration in the material (C_o) can be determined by means of extraction. EPA Method 8270E and Test Method CPSC-CH-C1001-09.4 can be used to determine bulk concentrations of phthalates in materials. The unknown mass transfer emission parameter required for exposure modeling in full-scale environments is the gas-phase concentration of SVOCs in equilibrium with the material phase (y_0) . This standard describes procedures for rapidly determining y_0 for phthalates from indoor planar polyvinyl chloride materials.

5.4 This method may be used to provide manufacturers, builders, and end users with some of the input data (y_0) required for models used to evaluating the impact of indoor planar, polyvinyl chloride materials on concentrations of indoor SVOCs as well as for mass transfer exposure models.

5.5 This method assumes that an instantaneous equilibrium exists between gas phase and material surface. This assumption has been made for a variety of SVOC mass transfer emission and exposure models (see Little et al. (2), Liang and Xu (1, 3), and Guo (4)). However, this assumption may be invalid under some environmental conditions.

6. Apparatus

6.1 Small-scale Emission Chamber:

6.1.1 The specially-designed emission chamber is made of 316 stainless steel (SS) with an electropolished internal chamber surface. Chambers can also be used to measure sorption of SVOCs to stainless steel surfaces and other materials/surfaces, but this is not within the scope of this method.

6.1.2 As shown in Fig. 1, the thin chamber is positioned between two test sample sheets. The chamber is short semicylindrical (40 cm in diameter \times 2 cm in height) in shape. Exact dimensions of the chamber are shown in Fig. 1. The chamber walls are stainless steel. The top and bottom of the chamber consist of two sheets of the tested sample. Air is injected into the circular side of the chamber and flows out the ports on the flat side. The design of the chamber maximizes the material emission area and minimizes the stainless steel sorption area. Stainless steel plates (at least 2 mm thick) are placed at the top and bottom of the chamber. Ten clamps or screws are used to seal the chamber. The chamber has a volume of 1 L and a loading factor of 16 m^2/m^3 . Users should ensure that test material samples have an area that extends beyond the perimeter of the chamber so that the edges of the material are not exposed to the stream of air and the chamber can be sealed properly.

6.1.3 Three inlets and six outlets enhance air flow mixing inside the chamber and increase SVOC emissions (Fig. 1). Each inlet shall have an electronic mass flow controller capable of providing 333 mL/min of air (one third of the total airflow). Outlet sampling flows shall also be controlled using electronic mass flow controllers.

6.1.4 Construct the outlet ports to fit the used sorbent tubes, so that tubes can be directly inserted into the chamber without any fittings. This avoids the loss of SVOCs to tubing and fittings along the sampling pathway.

Note 1—The outlet ports can also be used to measure sorption of SVOCs to stainless steel surfaces and other surfaces as described by Liang and Xu (1), but it is not within the scope of this test method.

6.1.5 Relatively thick (greater than 2 mm) planar polyvinyl chloride materials typically serve as an effective gasket that can pass leakage tests. Polytetrafluoroethylene (PTFE) sheets (3 mm thick) are required for adequate sealing in tests of thinner materials (1 mm thick, white layers in photo in Fig. 1b). Prior to testing determine if the chamber is leak free. The chamber can be considered sufficiently leak free if the outlet gas flow rate is greater than 95 % of the inlet gas flow rate. Measure in the flow rate in accordance with Practice D5337.

6.2 Temperature-controlled Environmental Chamber:

6.2.1 A temperature-controlled environmental chamber or incubator must be used to ensure the chambers are operated at a constant temperature during the emission test. The small-scale emission chambers are placed in a 25 °C environmental chamber that can evenly heat the chamber body and maintain it at controlled temperatures with an accuracy of ± 1 °C and precision of ± 1 °C at the set point. Interior chamber temperature is verified using a NIST traceable device.

6.3 Air Supply and Sampling System:

6.3.1 Ultra-zero grade dry air either from a gas cylinder or from a zero air generator is used to supply clean air to the small-scale emission chambers. All tubing between the cylinder and the chambers should be 316 stainless steel or PTFE where pressure appropriate.

6.3.2 Mass flow meters or mass flow controllers are required for metering/setting the air flow rate through the emission chamber and air sampling tubes. An accuracy of ± 2 % and precision of ± 3 % of the reading is needed.