



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 Calculations<sup>1</sup>

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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 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 gas-phase mass transfer coefficient across the material surface,  $h_m$ , from the known dimethyl phthalate mass transfer coefficient in the chamber (1).<sup>2</sup>

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:*<sup>3</sup>

[D1193 Specification for Reagent Water](#)

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

<sup>1</sup> 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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<sup>2</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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:<sup>4</sup>

[Method 8270E Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry \(GC/MS\)](#)

#### 2.3 ISO Methods:<sup>5</sup>

[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:<sup>6</sup>

[Test Method CPSC-CH-C1001-09.4 Standard Operating Procedure for Determination of Phthalates](#)

### 3. Terminology

#### 3.1 Definitions:

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  $\text{m}^2/\text{m}^3$ .

### 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

<sup>4</sup> 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>.

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

<sup>6</sup> 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 ( $\mu\text{g h}^{-1} \text{m}^{-2}$ ). 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 ( $\mu\text{g h}^{-1} \text{m}^{-2}$ ) 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 semi-cylindrical (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  $16 \text{ m}^2/\text{m}^3$ . 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  $\pm 1$  °C and precision of  $\pm 1$  °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  $\pm 2$  % and precision of  $\pm 3$  % of the reading is needed.

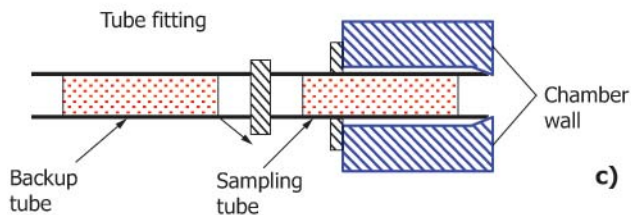
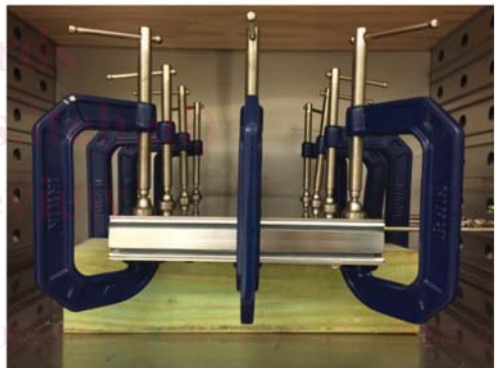
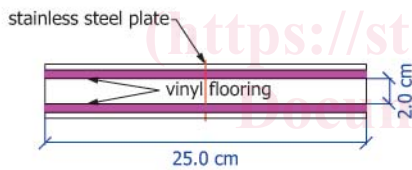
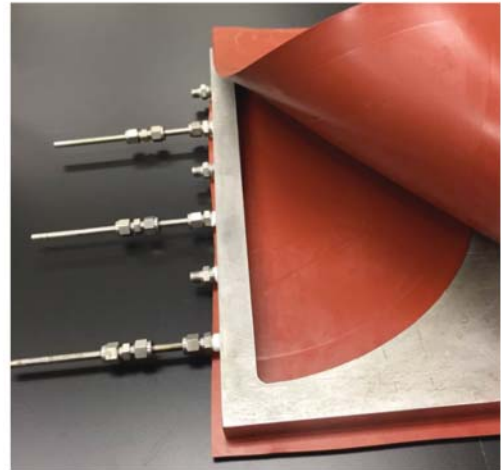
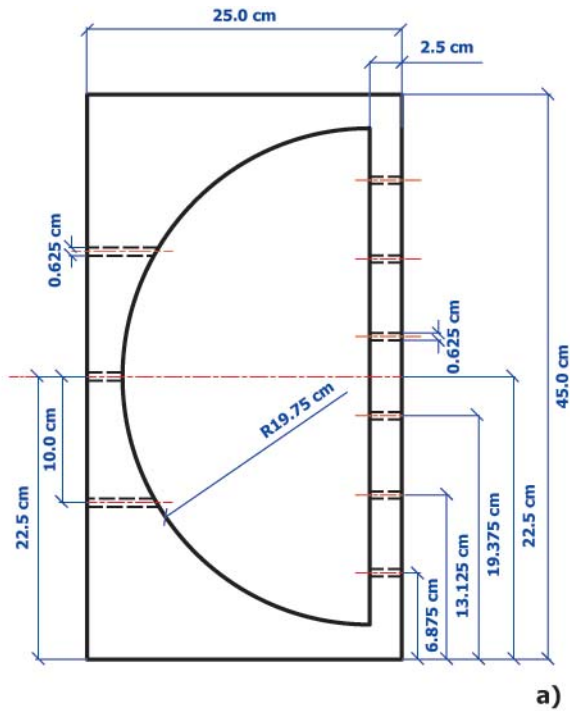


FIG. 1 Configuration of the Chamber  
(a) Top View, (b) Side View, and (c) Sampling Sorbent Tubes and Fittings

6.3.3 The total inlet flow rate of the small-scale emission chamber is maintained at a constant 1000 mL/min. The total inlet flow is split evenly between the three inlets.

NOTE 2—Other flow rates can be used but would require re-measurement of the mass transfer coefficients following the procedure discussed in Liang and Xu (1). Hence, flow rates other than 1000 mL/min are beyond the scope of this method, and the precision statements

provided in Section 13 will not apply.

6.3.4 Air sampling tubes prepacked with a short (5 mm to 10 mm) plug of inert, non-friable quartz wool at the sampling end followed by at least 200 mg of poly (2,6-diphenyl-1,4-phenylene oxide) polymer resin are required to collect SVOC samples. The entire sorbent bed (quartz wool and porous

polymer sorbent) must fall within the heated zone of the thermal desorption unit. Sorbent tubes are commercially available and the most commonly used tubes are 8.9 cm long and 6.4 mm outside diameter stainless steel tubes. Other tube sizes and materials (glass) can be used if at least 180 mg of poly (2,6-diphenyl-1,4-phenylene oxide) polymer resin is present and breakthrough is checked on two tubes of the last set of samples (see 10.2). Because only gas phase chemicals are expected in chamber testing, a particle filter is not used. Connect backup tubes to one of the three primary sample tubes per sampling event to check for breakthrough (Fig. 1c).

6.3.5 Three air sampling pumps are calibrated to a nominal flow rate of 150 mL/min following Practice D5337, to ensure high sorbent collection efficiencies of SVOCs and maintain good flow distribution within the chamber. The flow rate of pumps must be checked before and after sampling. The pumps are placed outside the temperature-controlled environmental chamber with exhaust vents interfaced to a fume hood or other exhaust system.

#### 6.4 Chemical Analysis System:

6.4.1 A thermal desorption (TD) system is required to transfer SVOCs from the sorbent tube to a gas chromatograph (GC). In TD, the SVOCs are released from the sorbent by heating and are collected in a cold trap. Flash heating and purging of the cold trap carries the sample to the GC column in a focused plug. Under this method, quartz wool is used at the inlet/outlet end of the cold trap to optimize retention, desorption efficiency and analyte recovery across the SVOC volatility range. If a transfer line is used to transfer the sample from the cold trap to the column, it should be uniformly heated, narrow bore (to increase linear gas velocity and thus minimize risk of analyte condensation onto surfaces) and inert (to avoid analyte degradation). Analyte recovery through the entire thermal desorption process (desorption efficiency) –from the sample tube to transfer/injection into the GC column – should be tested

as part of routine quality control, as described in Practice D6196. Fig. 2 shows the schematic of a typical TD system operation.

NOTE 3—The transfer line temperature used to develop this method was 250 °C. Optimization of different TD systems can result in different transfer line temperature requirements.

6.4.2 A GC-MS system is used to analyze SVOCs.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available (5).<sup>7</sup>

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

7.3 *Standard Stock Solutions*—An example list of potential compounds for indoor planar polyvinyl chloride (vinyl) emissions determination is shown in Table 1. The compounds in Table 1 have been measured emitting from historical vinyl flooring and mattress covering samples. It is possible that the compounds listed in Table 1 will not be present in currently available vinyl flooring and mattress coverings. Standard stock solutions of typical SVOCs are commercially available. In cases where standard stock solutions of SVOC chemicals are unavailable for purchase, they can be prepared from pure reagent and dilution solvents.

<sup>7</sup> ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

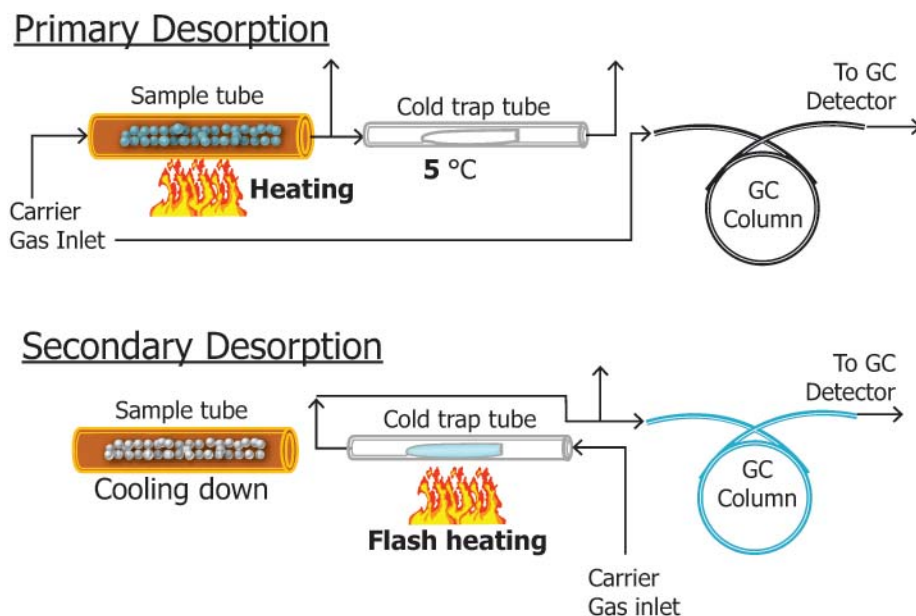


FIG. 2 Schematic Showing of a Typical TD System Operation

**TABLE 1 List of SVOCs Measured Emitting from Historical Vinyl Flooring and Mattress Covering Samples**

Compounds	Abbrev.	CAS no.	MW (g mol <sup>-1</sup> )
Di-n-butyl phthalate	DnBP	84-74-2	278.4
Butyl benzyl phthalate	BBP	85-68-7	312.4
Di(2-ethylhexyl) adipate	DEHA	103-23-1	370.6
Di(2-ethylhexyl) phthalate	DEHP	117-81-7	390.6
Di(2-ethylhexyl) isophthalate	Iso-DEHP	137-89-3	390.6
Diisononyl phthalate	DINP	28553-12-0	418.6
Diisononyl cyclohexane-1,2-dicarboxylate	DINCH	474919-59-0	424.7

7.4 *Hexane or Methanol*—Solvent used to dilute standard solutions and cleaning apparatus. The solvents need to be regularly analyzed to monitor potential SVOC contamination.

7.5 *Liquid Syringes*—1  $\mu\text{L}$ , 5  $\mu\text{L}$ , 10  $\mu\text{L}$ , 50  $\mu\text{L}$ , and 100  $\mu\text{L}$  and 0.5 mL and 1 mL are used to prepare calibration solutions.

7.6 *Nitrogen*, ultra high-purity grade (99.999 %).

7.7 *Air*, ultra-zero grade or better.

7.8 *Helium*, high-purity grade (best source).

7.9 *Tube cleaner*, instrument that heats sorbent tubes to a predetermined temperature while flowing clean helium or nitrogen through the tubes.

## 8. Preparation of Apparatus

### 8.1 Preparation of SVOC Standards:

8.1.1 If standard stock solutions of target SVOCs are not commercially available, prepare a standard stock solution of these SVOCs by dissolving weighted amounts of the target compounds in hexane or methanol as described in Practice D6196.

8.1.2 Prepare a working calibration standard mix from the standard stock solution. Following guidance given in Practice D6196, adjust the concentration of the target SVOCs in the standard mix solutions to reflect the range of mass expected in samples. Dilute the standard stock solutions for each target SVOC to a concentration range of 2.5 mg/L to 100 mg/L. This introduces a calibration mass range of 5 ng to 200 ng by means of a 2  $\mu\text{L}$  injection.

NOTE 4—Alternative mass ranges are also acceptable if more appropriate for the samples and compounds being tested.

### 8.2 Preparation of Sorbent Tubes:

8.2.1 Prepacked and pre-conditioned sorbent tubes must be cleaned again prior to use by heating to 300 °C for 1 h at a flow of 100 mL/min pure nitrogen or helium. Use a stainless steel manifold inserted in an oven and mount the sorbent tubes with graphite or graphite (15 %)/polyimide (85 %) ferrules, check the flows through each tube. Alternatively, a commercial tube conditioning unit or thermal desorption unit can be used for the cleaning. Desorb tubes at 300 °C for 20 min in cleaning mode or as described in the TD manual. Freshly-packed but unconditioned sorbent tubes require extensive preconditioning before use, typically for several hours. Follow manufacturer's instructions prior to use.

8.2.2 The cleaned sorbent tubes are fitted with end caps with PTFE ferrules before storage. Store tubes in a clean container or desiccator at ambient temperature. Equip the container with a bed containing activated charcoal to remove potential organic

contaminants from the container air. Check the tightness of the end caps weekly using hand tightening and spanners.

8.2.3 Before sorbent tube sampling usage, one tenth of the cleaned tubes should be randomly selected and analyzed to verify that they do not contain target SVOCs. If any of the tubes tested contain target SVOCs, all tubes must be tested with only those tubes that are below detection limit values for target SVOCs used for sampling.

### 8.3 Preparation of the Small-scale Emission Chamber:

8.3.1 Disconnect the chamber assembly and clean the chamber components including the stainless steel plates and fittings. Use an alkali detergent followed by three separate water rinses and three separate methanol rinses. Rinse all tubing with methanol. Dry the components thoroughly at an elevated temperature of 80 °C for one hour.

8.3.2 Measure background SVOC concentrations for each emission chamber prior to using them for sample testing. Background measurements are performed under normal operating conditions but without a material specimen in the chamber. Instead, PTFE sheets are used in the blank chambers as gaskets. Sample the chamber for 24 h in duplicate after 48 h of steady gas flow and constant temperature. The chamber can be considered sufficiently clean if the concentrations in the blank chamber are at least one tenth those found in samples. Preliminary tests may be needed to determine these levels, illustrative SVOC chamber concentrations can be found in (1, 6).

### 8.4 Preparation of Test Materials:

8.4.1 Cut new or used planar polyvinyl chloride materials into two 45 cm  $\times$  25 cm sheets with a clean knife. Record the age and condition of the sample. Wrap the sample sheets separately in clean aluminum foil and seal them in large polyethylene terephthalate (PET) storage bags.

8.4.2 During storage, protect samples from chemical contamination and from exposure to temperatures in excess of 25 °C.

### 8.5 Preparation of Other Lab Equipment:

8.5.1 Because laboratory equipment such as glassware and solvents may be contaminated with SVOCs, pre-wash all glassware. Use an alkali detergent followed by three separate rinses with water and three separate rinses with methanol. Dry the glassware thoroughly at an elevated temperature of 80 °C for one hour. Collect and analyze solvents, experimental blanks and spiked samples.

## 9. Calibration and Standardization

9.1 Standard stock solutions (8.1) are used to prepare calibration standards at concentrations spanning the applicable range of interest. Prepare standards covering at least five concentrations encompassing the range of interest. Typically, a range starting at the lowest range up to at least 20 times that value is used. Prepare standards no greater than five times the concentration of the next lower standard.

9.2 Inject 2  $\mu\text{L}$  of a standard solution into the sampling end of conditioned sorbent tubes using a microlitre syringe. The solvent in the solution must be purged using a flow of 100 mL/min of helium or nitrogen for 5 minutes.

NOTE 5—Longer purge times or higher flows may lead to breakthrough of standard SVOCs. If hexane is used as the solvent, a smaller spiking volume should be used due to insufficient solvent purge from the sorbent at 100 mL/min for 5 minutes. For more information on preparing standard solutions see Practice D6196.

9.3 Analyze each calibration standard and tabulate area response against mass injected. If internal standard (for example, tetradeuterium ring labeled DEHP, D4-DEHP) is used, the area ratio between the target SVOC and the internal standard compound is used. From the results, prepare a calibration curve, as illustrated in Fig. 3 (for example target SVOCs). Linear response is indicated when the correlation coefficient for a least-squares fit of the data is not less than 0.99. Use of a linear regression does not eliminate the possibility of large systematic errors if the intercept value is large relative to the quantity being measured (for example, over 25 % of the method detection limit). Users need to be aware of unusually large intercepts, which may bias the results and report these along with analytic results.

9.4 Once a linear response has been documented, daily calibration is performed using an intermediate concentration standard near the anticipated levels of target SVOCs, but at least ten times the detection limit. Acceptable variation in the day-to-day response for various SVOCs is 15 %. If greater variability is observed, recalibration is required, or a new calibration curve must be developed from fresh standards.

9.5 Care must be taken to ensure that the calibration standards do not exceed the capacity of the sorbent material, column, or the detector. The process of thermally desorbing sorbent tubes can be carried out in split or splitless GC mode. The analyst may need to adjust the split flow to accommodate the needed sensitivity, the column capacity or the detection limits.

## 10. Procedure

### 10.1 Chamber Setup and Operation:

10.1.1 Prepare cleaned emission chamber following instructions in 8.3.1.

10.1.2 Place the cleaned emission chamber in a temperature-controlled environmental chamber. Set a constant temperature of  $25 \pm 1$  °C for the duration of the test. The test is run slightly above ambient temperatures ( $\sim 23$  °C) to ensure the experiment is not influenced by changes in laboratory room temperature. Continuously record the environmental chamber temperature either by using the automatic recording function of

the environmental chamber or by placing a temperature data logger inside the environmental chamber.

10.1.3 Use 3 mm PTFE sheets as gaskets for the blank chamber and ensure the leakage rate is less than 5 % of the total inlet flow. Run dry, clean air through the blank chamber for two days. Measure background concentrations of blank chamber target compounds. The chamber is sufficiently clean if the concentrations are one tenth or less than the steady-state gas phase SVOC concentrations measured in emission chambers containing samples.

10.1.4 Carefully insert the material sheets on the bottom and top of the chamber ring while wearing polyethylene or nitrile gloves. Cover the top of the chamber with 2 mm stainless steel plates and seal the chamber using at least eight clamps (Fig. 1) or screws as applicable. For rigid materials, 3 mm PTFE sheets are recommended for use of gaskets to provide better sealing between test specimens and metal sheets.

NOTE 6—Holes may be drilled in the top and bottom metal plates to allow chamber sealing using screws.

10.1.5 Turn on the air supply and adjust the inlet flow rates using three mass flow controllers (333 mL/min each). Maintain the air supply flow rate to the chamber continuously for the entire duration of test. Place the mass flow controller outside of the temperature-controlled environmental chamber.

10.1.6 Record the start date and time of test.

10.1.7 Connect the six exhaust ports together and measure the total flow rates using a bubble meter or electronic flow meter. Ensure the leakage flow rate, defined as the difference between total inlet and the sum of six exhaust flows, is less than 5 % of the total inlet flow rate.

10.1.8 Wait for two hours prior to collecting air samples.

### 10.2 Sample Collection:

10.2.1 Collect air samples every 24 h starting at 48 h. For samples analyzed at 48 h and 72 h use a single tube without a backup tube. Steady-state is assumed when the concentration change is less than 10 % over 24 h (that is, 48 h and 72 h). If the change is greater than 10 %, the next sample (that is, 96 h) should be a single tube until the less than 10 % change criterion is met.

10.2.2 Once the less than 10 % change criterion is met between two previous samples (earliest is at 96 h) insert three primary sorbent tubes to the exhaust ports for triplicate air sampling (Fig. 1). Connect backup tubes to one of the three primary sample tubes per sampling event to check for breakthrough. The backup tube is connected to the outlet of the primary tube with a stainless-steel fitting with PTFE ferrules (Fig. 1). If the change between the average of triplicate sample and the previous sample (24 h earlier) increases more than 10 % the triplicate sample should be re-run 24 h later.

10.2.3 Assemble the sampling system and ensure that the pump is keeping constant flowrate throughout the sampling period.

10.2.4 Set the flowrate of the mass flow controllers at 150 mL/min following procedures described in Practice D5337. The sampling flow at exhaust ports influences the flow distribution inside the chamber according to this method. The sampling mass flow controllers can be placed inside or outside