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## Standard Test Method for Determining Chemical Emissions from Spray Polyurethane Foam (SPF) Insulation using Micro-Scale Environmental Test Chambers<sup>1</sup>

This standard is issued under the fixed designation D8142; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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<sup>e1</sup> NOTE—Editorially added research report information to Section 16 and Appendix X1 in February 2020.

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### 1. Scope

1.1 This test method is used to identify and to measure the emissions of volatile organic compounds (VOCs) emitted from samples of cured spray polyurethane foam (SPF) insulation using micro-scale environmental test chambers combined with specific air sampling and analytical methods for VOCs.

1.2 Specimens prepared from product samples are maintained at specified conditions of temperature, humidity, airflow rate, and elapsed time in micro-scale chambers that are described in Practice [D7706](#). Air samples are collected periodically at the chamber exhaust at the flow rate of the micro-scale chambers.

1.2.1 Samples for formaldehyde and other low-molecular weight carbonyl compounds are collected on treated silica gel cartridges and are analyzed by high performance liquid chromatography (HPLC) as described in Test Method [D5197](#) and ISO 16000-3.

1.2.2 Samples for other VOCs are collected on multi-sorbent samplers and are analyzed by thermal-desorption gas chromatography / mass spectrometry (TD-GC/MS) as described in U.S. EPA Compendium Method TO-17 and ISO 16000-6.

1.3 This test method is intended specifically for SPF insulation products. Compatible product types include two component, high pressure and two-component, low pressure formulations of open-cell and closed-cell SPF insulation.

1.4 VOCs that can be sampled and analyzed by this test method generally include organic blowing agents such as 1,1,1,3,3-pentafluoropropane, formaldehyde and other carbonyl compounds, residual solvents, and some amine catalysts. Emissions of some organic flame retardants can be measured after 24 h with this method, such as tris (chloroisopropyl) phosphate (TCPP).

1.5 This test method does not cover the sampling and analysis of methylene diphenyl diisocyanate (MDI) or other isocyanates.

1.6 Area-specific and mass-specific emission rates are quantified at the elapsed times and chamber conditions as specified in [13.2](#) and [13.3](#) of this test method.

1.7 This test method is used to identify emitted compounds and to estimate their emission factors at specific times. The emission

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factors are based on specified conditions, therefore, use of the data to predict emissions in other environments may not be appropriate and is beyond the scope of this test method. The results may not be representative of other test conditions or comparable with other test methods.

1.8 This test method is primarily intended for freshly applied, SPF insulation samples that are sprayed and packaged as described in Practice **D7859**. The measurement of emissions during spray application and within the first hour following application is outside of the scope of this test method.

1.9 This test method can also be used to measure the emissions from SPF insulation samples that are collected from building sites where the insulation has already been applied. Potential uses of such measurements include investigations of odor complaints after product application. However, the specific details of odor investigations and other indoor air quality (IAQ) investigations are outside of the scope of this test method.

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

1.11 *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.*

1.12 *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>2</sup>

- [D1356 Terminology Relating to Sampling and Analysis of Atmospheres](#)
- [D1622/D1622M Test Method for Apparent Density of Rigid Cellular Plastics](#)
- [D5116 Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products](#)
- [D5197 Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air \(Active Sampler Methodology\)](#)
- [D5337 Practice for Setting and Verifying the Flow Rate of Personal Sampling Pumps](#)
- [D6196 Practice for Choosing Sorbents, Sampling Parameters and Thermal Desorption Analytical Conditions for Monitoring Volatile Organic Chemicals in Air](#)
- [D7706 Practice for Rapid Screening of VOC Emissions from Products Using Micro-Scale Chambers](#)
- [D7859 Practice for Spraying, Sampling, Packaging, and Test Specimen Preparation of Spray Polyurethane Foam \(SPF\) Insulation for Testing of Emissions Using Environmental Chambers](#)

### 2.2 ISO Standards:<sup>3</sup>

- [ISO 16000-3 Indoor Air—Part 3: Determination of Formaldehyde and Other Carbonyl Compounds in Indoor Air and Test Chamber Air—Active Sampling Method](#)
- [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 or MS-FID](#)

### 2.3 Government Agency Methods:

- [40 CFR Appendix B to Part 136 Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11<sup>4</sup>](#)
- [California Department of Public Health Standard Method for the Testing and Evaluation of Volatile Organic Chemical Emissions from Indoor Sources Using Environmental Chambers, CDPH/EHLB/Standard Method V.1.2 \(January 2017\)](#)
- [U.S. EPA Compendium Method TO-17 Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition \(January 1999\)<sup>5</sup>](#)
- [U.S. EPA Method 325B Volatile Organic Compounds from Fugitive and Area Sources: Sampler Preparation and Analysis, Air Emission Measurement Center \(EMC\) \(September 29, 2015\)<sup>5</sup>](#)

<sup>2</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.

<sup>3</sup> Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

<sup>4</sup> Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.

<sup>5</sup> Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

### 3. Terminology

3.1 *Definitions*—For definitions and terms commonly used for sampling and analysis of atmospheres, refer to Terminology [D1356](#). For definitions and terms commonly used when testing materials and products for VOC emissions, refer to Guide [D5116](#). For definition of micro-scale test chamber, refer to Practice [D7706](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *area specific flow rate, n*—the ratio of the airflow rate into the chamber in m<sup>3</sup>/h and the projected surface area of the test specimen exposed to air in m<sup>2</sup> with units of m/h.

3.2.2 *closed-cell SPF insulation, n*—SPF insulation that contains cells or voids that are not interconnected.

3.2.2.1 *Discussion*—

Closed-cell SPF insulation typically has a density between ~~24~~24 kg/m<sup>3</sup> to ~~32–32~~ kg kg/m<sup>3</sup> when fully cured.

3.2.3 *open-cell SPF insulation, n*—SPF insulation that contains cells or voids that are largely interconnected.

3.2.3.1 *Discussion*—

Open-cell SPF insulation typically has a density between ~~6.4~~6.4 kg/m<sup>3</sup> to ~~9.6–9.6~~ kg kg/m<sup>3</sup> when fully cured.

### 4. Principles

4.1 Micro-scale test chambers are used for measuring emissions of VOCs including formaldehyde and other carbonyl compounds from materials and products.

4.2 Wall adsorption effects of reactive compounds and SVOCs are minimized by reducing the exposed inner surface of the chamber by fitting the SPF samples directly into the micro-scale chamber with minimal headspace. Passivated treatment of the interior surfaces also contributes to reduced sorption effects.

4.3 Micro-scale chambers are used for measuring area-specific emissions from the surface of SPF insulation or mass-specific emissions from the mass of the sample.

### 5. Summary of Test Method

5.1 A micro-scale chamber is used to measure VOC emissions from SPF insulation samples. A representative test specimen is prepared from the sample and is placed directly into a micro-scale chamber. Air samples are collected from the chamber exhaust at specified elapsed times.

5.2 Clean dry air is supplied to a micro-scale chamber and passes over the exposed surface of the test specimen before reaching the exhaust port. The airflow rate and the temperature within the micro-scale chamber are controlled. As the air passes over the test specimen, emitted compounds are swept away from the sample's surface.

5.3 The standard temperature for the test method is ~~35 ± 1 °C~~, 35 °C ± 1 °C. Emission rates of organic compounds from building materials and sorption and desorption of organic compounds from interior surfaces are sensitive to temperature. Operation of the micro-scale chamber at the specified temperature (above ambient) enhances the emission rates of some compounds and may improve the quantitative recovery of some less volatile compounds.

5.4 The area-specific flow rate specified for this test method is ~~0.95~~0.95 m/h ± ~~0.05~~0.05 m m/h-h. The maximum airflow rate for the micro-scale chamber shall not exceed 150 mL/min, and the airflow rate shall not exceed the maximum flow rate for the sampling media.

5.5 Gas samples are collected onto media cartridges or tubes at the exhaust port of the micro-scale chamber at various elapsed times throughout the test. The entire gas flow exiting the chamber passes through the cartridge or tube. The sampling flow rate is equivalent to the airflow rate of the micro-scale chamber.

5.5.1 Formaldehyde and other carbonyl compounds are sampled onto chemically treated cartridges (DNPH) and analyzed as described in Test Method [D5197](#) and ISO 16000-3. The sampling flow rate may be less than specified in the analytical methods.

5.5.2 Emissions of other VOCs and SVOCs are collected onto sorbent tubes and analyzed by thermal desorption – gas chromatography (GC) with mass spectrometry (MS) to identify and quantify compounds as described in ISO 16000-6 (Annex D) and U.S. EPA Compendium Method TO-17.

## 6. Significance and Use

6.1 SPF insulation is applied and formed onsite, which creates unique challenges for measuring product emissions. This test method provides a way to measure post-application chemical emissions from SPF insulation.

6.2 This test method can be used to identify compounds that emit from SPF insulation products, and the emission factors may be used to compare emissions at the specified sampling times and test conditions.

6.3 Emission data may be used in product development, manufacturing quality control and comparison of field samples.

6.4 This test method is used to determine chemical emissions from freshly applied SPF insulation samples. The utility of this test method for investigation of odors in building scale environments has not been demonstrated at this time.

## 7. Apparatus

### 7.1 General Description:

7.1.1 Micro-scale chambers are described in Practice [D7706](#). The micro-scale chamber test apparatus comprises one or more micro-scale chambers, a means of controlling the micro-scale chamber(s) at  $35 \pm 1^\circ\text{C}$ ,  $35^\circ\text{C} \pm 1^\circ\text{C}$ , a regulated clean air supply system and gas sampling capabilities.

7.1.2 The micro-scale chamber suitable for SPF insulation emissions testing is cylindrical in shape to accommodate an O-ring or gasket seal with an internal diameter of ~~6 to 10 cm~~ 6 cm to 10 cm and a depth of at least ~~3.5 cm~~ 3.5 cm. The chamber shall be capable of achieving an area specific flow rate of  ~~$0.950.95 \text{ m}^3/\text{h} \pm 0.50.5 \text{ m}^3/\text{h}$~~  at a flow rate not exceeding 150 mL/min. All of the air exiting the chamber outlet passes onto the sampling media cartridge or tube during sampling events.

7.1.3 The prepared SPF insulation specimen must fit into the chamber body such that the back surface and edges of the specimen are not directly exposed to the stream of air. An example micro-scale chamber to measure emissions of SPF insulation is shown in [Fig. X1.1](#).

### 7.2 Construction:

7.2.1 The micro-scale chamber body and associated lid shall be constructed of polished stainless steel with an inert coating by passivation using a process (typically patented) that diffuses amorphous silicon material into the surface of the stainless steel.

7.2.2 The gasket or O-ring used to seal the lid to its body shall be low absorbing and low emitting at the operating temperature so it does not contribute significantly to background VOC concentrations. Gaskets and O-rings composed of fluoropolymer elastomer are suitable for this application. The apparatus shall facilitate disassembly for loading samples and for cleaning.

### 7.3 Temperature and Heating Requirements:

7.3.1 Verify the interior temperature of each micro-scale chamber with a traceable device, for example with a National Institute of Standards and Technology (NIST) traceable certificate. The device used to measure temperature shall have an accuracy of at least  $\pm 1^\circ\text{C}$  between  $30^\circ\text{C}$  and  $40^\circ\text{C}$ . Various micro-scale chamber designs may require different temperature measuring devices or techniques.

7.3.2 Measure the temperature of each individual micro-scale chamber prior to first use and within 90 days of subsequent use with the water-fill procedure below.

7.3.2.1 Remove each micro-scale chamber from the system and fill each micro-scale chamber with distilled or deionized water to between 50 % and 75 % of the chamber volume. Place the water containing micro-scale chambers in the system and set the system to the prescribed test temperature of  $35^\circ\text{C}$ . Insert the temperature device through the sampling port so that it is immersed

in the water and the sensor does not touch the chamber walls or bottom of the chamber. The airflow through the chamber shall be set as described in 7.4.3. Allow the micro-scale chambers to equilibrate for at least 90 min. Determine the average temperature from each micro-scale chamber from at least five data points evenly spaced over a time that is equal to or greater than period of the heating frequency for the chamber. Record the average micro-scale chamber temperature and standard deviation to the nearest  $0.1^{\circ}\text{C}$ – $0.1^{\circ}\text{C}$ . If the average temperature of any micro-scale chamber is not between  $34.0^{\circ}\text{C}$  and  $36.0^{\circ}\text{C}$ , the temperature control shall be rectified prior to conducting any tests.

7.3.3 Additionally, prior to each use of 24 or more hours, measure the temperature of each micro-scale chamber with either the water-fill procedure (7.3.2.1) or by placing a measuring device in the air space of the chamber (7.3.3.1) or taping the device to the bottom of the chamber (7.3.3.2). The average of the chosen temperature measurement technique shall be demonstrated to agree with  $\pm 1^{\circ}\text{C}$  of the average of the most recent water-fill measurement. The heating and cooling cycle in some micro-scale chamber designs can result in  $1^{\circ}\text{C}$  to  $2^{\circ}\text{C}$  cycles (at  $35^{\circ}\text{C}$ ). The averaging time should be three times the period of the heating frequency for the chamber. Allow the chamber to equilibrate prior to measurement (up to 20 min).

7.3.3.1 Devices in air should be placed at a consistent depth in the chamber to get reliable values. Micro-scale chamber's air temperature can be vertically stratified in air up to  $2^{\circ}\text{C}$ .

7.3.3.2 The ability of temperature devices taped to or touching the bottom of the chamber to represent the water determined values is dependent upon the chamber design. Micro-scale chamber's wall temperature can be horizontally stratified up to  $0.6^{\circ}\text{C}$ .

7.3.4 The apparatus may provide for heating of chamber bodies in some cases as high as  $250^{\circ}\text{C}$  for facilitating chamber cleaning.

#### 7.4 Air Supply Requirements:

7.4.1 Ultra-zero grade air is used as the carrier gas through the micro-scale chamber. The air is dry and not humidified.

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

7.4.3 A constant inlet airflow rate is maintained to achieve an area specific flow rate between  $0.9$ – $0.9\text{ m/h}$  and  $1\text{ m}$ – $1\text{ m/h}$ . Micro-scale chambers with a  $6.4\text{ cm}$  diameter are operated at  $50$ – $50\text{ mL/min}$  and those with a  $9.2\text{ cm}$  diameter are operated at  $100$ – $100\text{ mL/min}$ . For other chamber sizes, do not exceed a flow rate of  $150\text{ mL/min}$ , and calculate the area specific flow rate as follows:

$$R = (Q \cdot 0.00006)/A \quad (1)$$

where:

- $R$  = area specific flow rate,  $\text{m/h}$ ,
- $Q$  = flow rate of micro-scale chamber,  $\text{mL/min}$ ,
- $A$  = specimen's horizontally projected surface area (13.1),  $\text{m}^2$ , and
- $0.00006$  = a constant to convert  $\text{mL/min}$  to  $\text{m}^3/\text{h}$ .

7.4.4 During operation, the micro-scale chamber should be leak free. The procedure for determining this differs for micro-scale chambers arranged to measure multiple samples in parallel to that of a serially operated micro-scale chamber which analyzes one sample at a time. For a parallel-operated micro-scale chamber with a common gas source for all chambers, the airflow rate of each chamber is measured and if all the flows are all within  $5\%$  of each other this indicates a leak free system. If one or more of the chambers shows anomalously low flow result this indicates a leak. For a serially operated micro-scale chamber the cell is considered sufficiently leak-free if the outlet airflow rate is within  $95\%$  of the inlet airflow rate. The airflow rate can be measured as described in Practice D5337.

## 8. Reagents and Materials

### 8.1 Gas Sampling Devices:

8.1.1 *Sorbent Tubes for Active Sampling*—Sorbent tubes are commercially available either pre-packed by the manufacturer or empty. Different suppliers provide different size tubes and packing lengths. The most commonly used tubes for thermal desorption are  $8.9\text{ cm}$  long and  $6.4\text{ mm}$  O.D. stainless steel. Appropriate sorbents for analysis by thermal desorption GC/MS are described in Practice D6196, U.S. EPA Compendium Method TO-17, ISO 16000-6 (Annex D).

8.1.2 Thermal desorption tubes are packed with more than one sorbent to cover the wide volatility range for this method. Multi-sorbent tubes are packed in order of increasing sorbent strength to facilitate quantitative retention and desorption of VOCs over a wide volatility range. The higher molecular weight compounds are captured on the front, weaker sorbent; the more volatile compounds are retained farther into the packing on a stronger sorbent. The sample tube is marked to designate the proper flow path during sampling. Due to the wide range of volatility of compounds two multi-sorbent tubes are specified:

8.1.2.1 *Multi-sorbent Tube A, for VOCs and SVOCs*—Quartz Wool backed up a weak porous polymer further backed up by a strong strength graphitized carbon black. See Practice [D6196](#), Table X1.1, Tabulated Summary of Sorbent Types, Examples, Features, and Applications for further detail on these sorbents.

8.1.2.2 *Multi-sorbent Tube B, for very-volatile organic compounds* *Very Volatile Organic Compounds (VVOCs) such as blowing agents—Blowing Agents*—Weak porous polymer backed up a medium strength graphitized carbon black further backed up by a carbon molecular sieve. See Practice [D6196](#), Table X1.1, Tabulated Summary of Sorbent Types, Examples, Features, and Applications for further detail on these sorbents.

NOTE 1—Not all blowing agents, such as 1,1,1,2-tetrafluoroethane, used in low-pressure SPF insulation systems, can be determined with this test method.

8.1.3 Follow Practice [D6196](#) for selection of sorbent tubes if alternative tubes are required to recover specific compounds not listed in this standard.

8.1.4 Tubes are conditioned before initial use as specified by the tube supplier or as described below.

8.1.4.1 For multi-sorbent Tube A, under a flow of ~~50–100~~ 50 mL/min to 100 mL/min of pure inert gas (helium or nitrogen) condition the sorbent tubes as follows: ~~2 hours~~ at 320 °C, followed by a further ~~30 minutes~~ at 335 °C. Recondition the tubes after each use for ~~30 minutes~~ at 335 °C.

8.1.4.2 For multi-sorbent Tube B, under a flow of ~~50–100~~ 50 mL/min to 100 mL/min of pure inert gas (helium or nitrogen) condition the sorbent tubes as follows: ~~1 hour~~ at 100 °C, followed by ~~1 hour~~ at 200 °C, followed by ~~1 hour~~ at 300 °C, followed by ~~30 minutes~~ at 335 °C. Recondition the tubes after each use for ~~15 minutes~~ at 100 °C, followed by ~~15 minutes~~ at 200 °C, followed by ~~15 minutes~~ at 300 °C, followed by ~~15 minutes~~ at 335 °C.

8.1.5 Each tube is indelibly etched with a unique identification number and optional bar code. The numbers of the tubes selected are recorded and batched according to date of packing and number of thermal cycles. Sorbent tubes are sealed with long-term storage caps until the start of sample collection and are resealed with the same caps immediately after sample collection unless they are to be analyzed immediately.

8.1.6 *Sorbent Tube End Caps for Storage*—Blank and sampled tubes are sealed with metal screw-cap compression fittings with combined (one-piece) PTFE ferrules for storage and transportation. If alternate fittings/storage systems are used, the laboratory shall determine that they meet storage and transportation stability requirements. To check that storage caps have been fitted correctly, check the length of the capped tube to make sure the seals are seated as far down the tube as possible and check that the caps cannot be pulled off the tubes by hand using reasonable force.

8.2 For formaldehyde and other carbonyl compounds, use cartridges or tubes containing silica gel treated with 2,4-dinitrophenylhydrazine (DNPH) as described in Test Method [D5197](#). Use adapters if necessary to obtain a seal on the micro-scale chamber exhaust.

### 8.3 *Cutting Tools and Accessories:*

8.3.1 Circular foam coring tool, described in Practice [D7859](#). The tool shall be clean, constructed of steel to cut SPF insulation samples to fit tightly into micro-scale chambers.

8.3.2 Knife or saw, clean, and free of cutting oils and other organic contaminants.

8.3.3 Shim rings, clean, constructed of PTFE, stainless steel, or other inert material, which may be required to seal off edges of open-cell SPF specimens so they fit tightly into the micro-scale chambers.