



Designation: D8142 – 17^{ε1}

Standard Test Method for Determining Chemical Emissions from Spray Polyurethane Foam (SPF) Insulation using Micro-Scale Environmental Test Chambers¹

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^{ε1} NOTE—Editorially added research report information to Section 16 and Appendix X1 in February 2020.

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 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 measure are used.

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*

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Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

- D1356** Terminology Relating to Sampling and Analysis of Atmospheres
- 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 Flow Rate Adjustment 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:³

- 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 1.11⁴
- 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

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.

³ 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>.

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

for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (January 1999)⁵

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)⁵

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 m³/h and the projected surface area of the test specimen exposed to air in m² 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 to 32 kg/m³ 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 to 9.6 kg/m³ 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

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

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. 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 ± 0.05 m/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 ± 1 °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 and a depth of at least 3.5 cm. The chamber shall be capable of achieving an area specific flow rate of 0.95 ± 0.5 m/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 ± 1 °C between 30 °C and 40 °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 °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 °C. If the average temperature of any micro-scale chamber is not between 34.0 °C and 36.0 °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 ± 1 °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 °C to 2 °C cycles (at 35 °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 °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 °C.

7.3.4 The apparatus may provide for heating of chamber bodies in some cases as high as 250 °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 ± 2 % and a precision of ± 3 % of the reading.

7.4.3 A constant inlet airflow rate is maintained to achieve an area specific flow rate between 0.9 and 1 m/hr. Micro-scale chambers with a 6.4 cm diameter are operated at 50 mL/min and those with a 9.2 cm diameter are operated at 100 mL/min. For other chamber sizes, do not exceed a flow rate of 150 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, m/h,
 Q = flow rate of micro-scale chamber, mL/min,
 A = specimen's horizontally projected surface area (13.1), m^2 , and
 0.00006 = a constant to convert mL/min to m^3/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 cm long and 6.4 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 (VVOCs) such as 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 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 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 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.

8.3.4 Spacers, clean, constructed of PTFE, stainless steel, or other inert material to elevate the specimen to the specified height in the micro-scale chamber.

8.4 *Thermal Desorption GC/MS:*

8.4.1 The instrumentation for thermal desorption GC/MS is described in Practice D6196 (thermal desorption), U.S. EPA Compendium Method TO-17 and U.S. EPA Method 325B, ISO 16000-6 or use equivalent GC/MS technology. Operate the MS

in full scan mode. The analytical column and transfer line shall be amine-compatible and optimized for the analysis of the target compounds specified in Table 1. See Table X1.1 for an example of instrument conditions for measuring SPF emissions.

8.4.2 *Calibration Solution Loading Rig*—Consists of an unheated injector port with a controlled carrier gas (nitrogen) supply and a sorbent tube connection point. Heated tube injectors shall not be used to avoid potential losses of flame retardants and reactive amine compounds. The sampling end of a packed sorbent tube is connected to the unit and the carrier gas flow is set to between 50 and 100 mL/min. Carrier gas sweeps through the injection port and passes through the sorbent tube to vent. The calibration standard (liquid or gas) is introduced through the injector septum using a standard GC syringe. Liquid standard solutions vaporize in the flow of gas allowing analytes to reach the sorbent bed in the gas phase.

8.5 *Reference Standards for Thermal Desorption GC/MS:*

8.5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.5.2 *Reference Target Compounds*—High purity compounds of interest can be sourced from commercial vendors. Alternatively, components from raw materials of the SPF insulation formulation are sourced directly from the SPF insulation manufacturer or raw material supplier if the primary chemical content is known. When compound purity is assayed to be 96 % or greater, the weight may be used without

TABLE 1 Example List of Target Compounds

NOTE 1—TD = Thermal desorption tube (8.1.2),
DNPH = 2,4-dinitrophenylhydrazine tube (8.2), and
The primary quantitation ion for each compound is in parentheses.

Compound	CAS #	Sample Media	GC/MS Primary and Secondary Ions
1,1,1,3,3-Pentafluoropropane (HFC-245fa)	460-73-1	TD	(64), 69, 134
Trimethylamine	75-50-3	TD	(58), 59, 42
1,1-Dichloroethene	75-35-4	TD	(61), 96, 98
3-Chloro-1-propene (Allyl Chloride)	107-05-1	TD	(41), 76, 78
trans-1,2-Dichloroethene (TDCE)	156-60-5	TD	(61), 96, 98
1,2-Dichloropropane	78-87-5	TD	(63), 62, 76
1,4-Dioxane	123-91-1	TD	(88), 58, 57
2-Ethyl-4-methyl-1,3-dioxolane	4359-46-0	TD	(87), 59, 72
Chlorobenzene	108-90-7	TD	(112), 77, 114
2-Butoxyethanol	111-76-2	TD	(57), 87, 45
1,4-Dichlorobenzene	106-46-7	TD	(146), 148, 111
Triethylenediamine (TEDA)	280-57-9	TD	(58), 55, 112
1,2-Dichlorobenzene	95-50-1	TD	(146), 148, 111
Bis(2-chloroisopropyl) ether	108-60-1	TD	(45), 121, 77
Bis[2-(N,N-dimethylamino)ethyl] ether (BDMAEE)	3033-62-3	TD	(58), 71, 42
Triethyl phosphate (TEP)	78-40-0	TD	(99), 127, 155
Pentamethyldiethylenetriamine (PMDTA)	3030-47-5	TD	(72), 58, 115
4-(1,1-Dimethylpropyl) phenol	80-46-6	TD	(135), 107, 164
Tris(1-chloro-2-propyl) phosphate (TCPP)	13674-84-5	TD	(99), 125, 157
Propionaldehyde	123-38-6	DNPH	—
Formaldehyde	50-00-0	DNPH	—
Acetaldehyde	75-07-0	DNPH	—

correction to calculate the concentration of the stock standard. The target compounds of interest are determined from specific VOCs and SVOCs that are known to be present or emit from the SPF formulation. An example list of target compounds derived from a variety of SPF insulation samples is shown in [Table 1](#).

NOTE 2—Toluene equivalents can be used to estimate non-target compounds, as described in 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.

8.5.3 Dilution Solvent—Purge-and-trap grade methanol is used as the solvent to prepare calibration standards. It shall be of chromatographic quality and free from compounds co-eluting with the compound or compounds of interest. Alternative dilution solvents, for example, ethyl acetate or cyclohexane can be used, particularly if there is a possibility of reaction or chromatographic co-elution with methanol.

8.5.4 Stock Standard Solutions—Prepare stock standard solutions in dilution solvent using assayed liquids or gases, as appropriate at 2000 ng/μL or appropriate concentration. Place about 9 mL of methanol in a 10-mL tared ground-glass-stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all the dilution solvent wetted surfaces have dried. Weigh the flask to the nearest 0.0001 g. Add the assayed reference material, as described below.

8.5.4.1 Liquids—Using a syringe or pipette, immediately add one or more drops (0.02 g for 2000 ng/μL) of the reference material to the flask; then reweigh. The liquid shall fall directly into the dilution solvent without contacting the neck of the flask. Very volatile organic liquids (for example 1,1,1,3,3-pentafluoropropane) are placed in a freezer at -10°C to -20°C prior to preparation.

8.5.4.2 Gases—Solution Containing Approximately 1 mg/mL of Gas Components—For gases, prepare a low level calibration solution as follows. Obtain pure gas at atmospheric pressure by filling a small plastic gas bag from a gas cylinder. Fill a 1-mL gas-tight syringe with 1 mL of the pure gas and close the valve of the syringe. Using a 2-mL septum vial, add 2-mL solvent and close with the septum cap. Insert the tip of the syringe needle through the septum cap into the solvent. Open the valve and withdraw the plunger slightly to allow the solvent to enter the syringe. The action of the gas dissolving creates a vacuum, and the syringe fills with solvent. Return the solution to the flask. Flush the syringe twice with the solution and return the washings to the flask. Calculate the mass of gas added using the gas laws, that is, 1 mol of gas at STP occupies 22.4 L.

8.5.5 Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in mg/L from the net gain in weight. The concentration in mg/L is equivalent to ng/μL.

8.5.6 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10 to -20°C . Return standards to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent evaporation of the solution.

8.5.7 Calibration Solutions—Following guidance given in Practice [D6196](#), dilute the stock standards such that a 0.5 to 2-μL injection, by means of the calibration solution loading rig introduces 20 to 2000 ng of each compound, or an alternative mass range if more appropriate to the samples being analyzed.

8.5.8 Internal Standards—Internal standards shall be used as described in U.S. EPA Compendium Method TO-17 or U.S. EPA Method 325B. Gas-phase standards must be obtained in pressurized cylinders containing vendor certified gas concentrations accurate to $\pm 5\%$. The concentration shall be such that the masses of internal standard components introduced are similar to those of the target compounds of this method. Alternatively, liquid solutions containing the internal standards can be loaded onto TD tubes prior to analysis as described in [9.1.2](#). See [Table X1.1](#) for an example of internal standards used for measuring SPF emissions.

8.6 Reagents and materials for the analysis of formaldehyde and other carbonyl compounds are described in Test Method [D5197](#).

8.7 Analytical Balance and Containers:

8.7.1 Class A volumetric flasks for preparing standard solutions, as needed.

8.7.2 Analytical balance, readable and accurate to the nearest 0.1 mg.

9. Calibration and Standardization

9.1 Thermal desorption GC/MS standard calibration curves are required for each compound of interest comprising at least five points covering an applicable range and with a factor of at least 20 between the lowest and the highest level standard. At least six points are used for quadratic fit calibration curves. The standard concentrations distribution shall not be greater than five times the concentration of the next lower standard (for example; 20, 50, 100, 200, 500, 1000, 2000 ng). Prepare loaded tubes by injecting aliquots of standard solutions onto clean sorbent tubes as follows:

9.1.1 Fit the sampling end of the clean sorbent tube into the calibration solution loading rig (see [8.4.2](#)) with a 50–100 mL/min flow rate of inert purge gas and inject 0.5 to 2-μL aliquot of an appropriate standard solution through the septum for 1 to 5 minutes. Reproducible and quantitative transfer of higher boiling compounds in liquid standards is facilitated if the injection unit allows the tip of the syringe to just touch the sorbent retaining gauze or quartz wool inside the tube. If calibration tubes are to be prepared using multiple standards, introduce those containing the least volatile compounds of interest first and the most volatile compounds of interest (typically the gas phase standards) last. See Practice [D6196](#), [8.5](#), for further information on loading TD sorbent tubes.

9.1.2 If a liquid solution is used to load internal standards, with 50–100 mL/min flow rate of inert purge gas, inject 1 μL of the internal standard solution through the septum of the calibration loading rig for 1 to 5 minutes, and then disconnect the tube and seal it using long-term storage caps.

9.1.3 Use the GC/MS data acquisition software to plot relative response factors for each compound. Use the average of response factors for each compound if the relative standard