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Standard Practice for Spraying, Sampling, Packaging, and Test Specimen Preparation of Spray Polyurethane Foam (SPF) Insulation for Testing of Emissions Using Environmental Chambers[1]

This standard is issued under the fixed designation D7859; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Editorial changes were made in April 2013.

1. Scope

1.1 This practice describes standardized procedures for the preparation, spraying, packaging, and shipping of fresh spray polyurethane foam (SPF) insulation product samples to be tested for their emissions of volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs). These procedures are applicable to both closed-cell and open-cell SPF insulation products. Potential chemical emissions of interest include blowing agents, solvents, aldehydes, amine catalysts, diisocyanates, and flame retardants.

1.2 Typically, SPF insulation samples are prepared at one location, such as a chemical manufacturing facility or a field product installation site. The newly prepared samples are preserved in a sealed bag, placed in a secondary container, and then shipped to a laboratory for testing.

1.3 The spraying of SPF insulation products is only to be performed by trained individuals using professional spraying equipment under controlled conditions. The details of the spraying equipment and spraying procedures are based on industry practice and are outside of the scope of this practice.

1.4 This practice also describes procedures for the laboratory preparation of test specimens from open-cell and closed-cell SPF insulation product samples. These specimens are prepared for testing in small-scale chambers following Guide D5116 and in micro-scale chambers that are described in Practice D7706.

1.5 Procedures for VOC and SVOC emission testing, gas sample collection and chemical analysis are outside of the scope of this practice. Such procedures will need to address the potential for emissions of some SVOCs, for example, amine catalysts, flame retardant and isocyanates, to adhere to the chamber walls.

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

1.7 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D4840 Guide for Sample Chain-of-Custody Procedures

D5116 Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products

D7706 Practice for Rapid Screening of VOC Emissions from Products Using Micro-Scale Chambers

3. Terminology

3.1 For definitions of terms commonly used for sampling and analysis of atmospheres, refer to Terminology D1356. For definitions of terms commonly used when testing products and materials for VOC emissions, refer to Guide D5116.

3.2 *A-Side*—polymeric methylene diphenyl diisocyanate (MDI) consisting predominantly of 4,4'-MDI and higher molecular weight oligomers of MDI.

3.3 *B-side*—polyol system, or resin system, consisting mostly of polyol(s), with smaller amounts of catalyst(s), flame retardant(s), blowing agent(s), and other additives.

3.4 *Open-cell SPF*—SPF that contains cells or voids that are largely interconnected. Open-cell SPF insulation typically has a density between 6.4 to 9.6 kilograms per cubic metre when fully cured.

[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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[2] 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.

3.5 *Closed-cell SPF*—SPF that contains cells or voids that are not interconnected. Closed-cell SPF insulation typically has a density between 24 to 32 kilograms per cubic metre when fully cured.

4. Summary of Practice

4.1 This practice is applicable to open-cell and closed-cell spray polyurethane foam (SPF) insulation. Procedures are described for the preparation, spraying, packaging, shipping, and test specimen preparation of small, representative samples of these products. The samples are then sent to a laboratory and tested for emissions of volatile and semi-volatile organic compounds in environmental test chambers described in Guide D5116 and Practice D7706.

4.2 Samples are prepared by trained and competent operators using professional spraying equipment. Detailed instructions on the selection and operation of this equipment are outside of the scope of this practice. This practice specifies the information to be collected during the spraying operation. A test sample is sprayed to a defined thickness and size onto a defined substrate. The sample is wrapped with aluminum foil, packaged in a layered polyethylene terephthalate (PET) bag, placed in a secondary container, and sent to the laboratory on the same day it is sprayed and prepared, if possible.

4.3 Testing is to begin within 48 h of the time the sample is sprayed and prepared. When testing in a small-scale environmental chamber, the laboratory cuts the sample to create a test specimen of a defined thickness and size. The thickness varies with product type as specified in 8.3 and 8.4. The test specimen is placed into a tight-fitting stainless-steel holder with only the upper face of the product exposed. The specimen in its holder is transferred to the test chamber. Specialized procedures are described in 8.5 for preparing specimens for testing in micro-scale chambers.

5. Significance and Use

5.1 Manufacturers of SPF insulation may need to test their products for vapor-phase emissions of volatile and semi-volatile organic compounds in order to comply with voluntary standards, purchase specifications, or other requirements.

5.2 Since SPF insulation is formed by chemical reaction when combining a two-component mixture during spraying, specialized equipment and procedures are needed to reproducibly create representative samples suitable for emission testing.

5.3 SPF insulation product manufacturer's specifications and instructions must be followed carefully and detailed information regarding the spraying process must be recorded (see 7.3). Other precautions regarding handling and shipping are needed to ensure that the chemical integrity of the samples is preserved to the extent possible by practical means (see 7.5).

5.4 Laboratories must prepare representative test specimens from samples of SPF insulation in a consistent manner so that emission tests can be reproduced and reliable comparisons can be made between test data for different samples.

6. Materials

6.1 *Spray gun and related spraying equipment* for application of the SPF insulation product shall be as specified by the SPF insulation product manufacturer. Contact the SPF equipment manufacturer for details concerning the spraying equipment. (**Warning—**DO NOT attempt to create SPF insulation samples without the proper spraying equipment or without proper training in the operation of this equipment including the use of appropriate personal protective equipment.[3]) The main spraying equipment components typically consist of:

6.1.1 Spray gun with impingement mixing technology for mixing the two part liquid product inside the gun, gun tip, and purge system;

6.1.2 Proportioning machine with pumps, pressure controls and heating capacity; and

6.1.3 Heated hoses.

6.2 *Substrate Material*—Clean high-density polyethylene (HDPE) sheets, cut to minimum dimensions of 30 by 30-cm, with a minimum thickness of 9-mm.

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

6.4 *Circular foam coring tool* constructed of steel to cut SPF insulation samples to fit tightly into sample holders or directly into micro-scale chambers as described in Practice D7706.

6.5 *Layered polyethylene terephthalate (PET) bags,* with a middle layer of aluminum foil and an inner layer of linear low density polyethylene (LLDPE), light resistant, preferably with zipper seal (zipper seal may not be available on larger sized bags), composite layer approximately 0.127-mm thick. Bags are available commercially for food storage and should be sized to minimize headspace when the sample is placed in the bag.

NOTE 1—For open-cell SPF products, a bag size of approximately 51 by 76 cm has been found to be suitable. For closed-cell SPF products, a bag size of approximately 46 by 71 cm has been found to be suitable.

6.6 *Packaging tape, clear,* approximately 5-cm wide.

6.7 *Stainless steel sample holder with open top;* dimensions are described in 8.3.3 and 8.4.3 depending on the type of material being tested.

6.8 *Stainless steel or polytetrafluoroethylene (PTFE) shims* for sample holder, as necessary.

6.9 *Aluminum foil,* clean, heavy-gauge roll, approximately 0.024-mm thick.

6.10 *Shipping container,* sturdy and insulated secondary container such as a recreational cooler or a molded insulated shipping container housed in a cardboard box. The insulated container should be as air tight as possible.

7. Sample Preparation

7.1 Prepare and spray the SPF insulation sample either in a controlled spray booth or room at a product manufacturing location or in the field at a building application site using the equipment and processing parameters that are specified by the SPF insulation product manufacturer for application of the product in buildings. The spray booth or room should be

[3] SPF applicator on-line health and safety training is available from the Center for the Polyurethanes Industry at www.spraypolyurethane.org

maintained at a constant temperature of 23 ± 2°C and relative humidity of ≤80%. However, the preparer may elect to utilize different environmental parameters in the spray booth or room to mimic a particular field condition (for example, cold weather application). Environmental conditions of the spray booth or room must be documented. For safety and to ensure that samples are prepared properly, preparation and spraying must be conducted by a trained and competent operator. Follow all applicable safety instructions.

NOTE 2—Industry programs that accredit spray foam contractors and operators are available.[4]

7.2 Prior to spraying the SPF insulation sample, the operator shall ensure that the container of the B-side material of the product is adequately mixed if specified by the manufacturer. Prior to spraying, the operator flushes the spray equipment using the product formulation to be tested so that potential residuals from previously-sprayed products do not contaminate the sample. Flush the A- and B-side hoses with the material to be sprayed using volume equal to at least three times the hose volume. If the spray gun incorporates a self-cleaning mechanism (that is, solvent), the solvent name and its Chemical Abstract Service (CAS) registry number are recorded and communicated to the laboratory.

7.3 At the time of spraying, record the following information:

7.3.1 Spray operator name, affiliation, and, if applicable, certifications/accreditations;

7.3.2 SPF insulation product manufacturer name, product name, whether open-cell or closed cell formulation, and lot numbers of the A- and B-side SPF materials;

7.3.3 Manufacturer, model, and serial numbers for the spray gun, gun tip, proportioner, and other related spraying equipment;

7.3.4 Spray booth or room temperature, relative humidity, and barometric pressure;

7.3.5 A-side and B-side preheater set-point temperatures, static (not spraying) and dynamic (spraying) pressures, and hose heater set-point temperature;

7.3.6 Date and time sample was sprayed, and the number of lifts (layers or passes) used to create the sample.

7.3.7 If known, whether or not the surface skin of the sample will be trimmed in the field to align the SPF surface with wall studs or other structural elements; and

7.3.8 Any other relevant information. Photographs of the key spraying operations are recommended to be included as part of the record.

7.4 *Spraying Operation:*

7.4.1 If spraying horizontally, elevate the HDPE substrate several centimetres above the spraying surface using small blocks of wood, disposable cups, or other support.

7.4.2 For open-cell SPF insulation, spray the product onto the substrate so that the substrate is covered to a foam thickness of greater than 10 cm (typically 10–11 cm), using one lift (layer or pass). An additional lift may be performed if it is necessary to achieve the proper thickness.

7.4.3 For closed-cell SPF insulation, spray the product onto the substrate so that the substrate is covered to a foam thickness of greater than 4 cm (typically 4–5 cm), using one lift (layer or pass). An additional lift may be performed if it is necessary to achieve the proper thickness.

7.5 *Packaging and Shipping:*

7.5.1 After spraying, wait one hour to allow curing of the SPF insulation material. Wrap the sample, including the HDPE substrate, with at least one layer of clean aluminum foil. Then, place the entire wrapped sample into a layered PET bag of a size that minimizes the headspace over the sample. To further minimize headspace, manually force as much air out of the bag as possible, but do not use any vacuum. Record the time the sample is packaged.

7.5.2 Label the outside of the layered PET storage bag with sample identification, but DO NOT write on the SPF insulation samples, aluminum foil or substrate. If the bag has a zipper seal, close the zipper. Seal again by creating a 5-cm fold, repeat the fold several times, and then apply packaging tape to the fold. Do not heat-seal the layered PET bag.

7.5.3 Place layered PET bag(s) in a sturdy and insulated secondary container such as a recreational cooler or a molded insulated shipping container housed in a cardboard box. The insulated container should be as air tight as possible. The sample is now ready for shipment to the laboratory. Do not add ice packs to the shipping container or expose the shipping container to excessive heat.

NOTE 3—This practice does not address the impact of environmental conditions during shipping on emissions tests. It is recommended to place electronic data loggers that monitor pressure, temperature and relative humidity inside the shipping container to record these parameters during transport. If data loggers are used during transport, record the data upon receipt in the laboratory as specified in 8.1. The data can be used to evaluate sources of variability during transport. Some carriers offer temperature controlled services for temperature sensitive products.[5]

7.5.4 Complete a chain of custody using Guide D4840. Send the prepared SPF insulation sample, the chain of custody, and the information collected regarding sample preparation and packaging to the testing laboratory. Specify to the laboratory if the surface skin of the sample needs to be removed to simulate trimming in the field to align the SPF surface with wall studs or other structural elements. Ship the sample on the same day it is prepared and utilize a next-day delivery shipping service. Schedule sample preparation and shipment so that the laboratory receives the package on a working day. Additionally, coordinate with the laboratory to be sure testing can start within the prescribed time schedule, which is within 48 hours of when the sample was sprayed.

7.5.5 The names and CAS numbers of the particular blowing agent(s), amine catalyst(s), flame retardant(s), and

[4] Accreditation programs are available through some manufacturers and the Spray Polyurethane Foam Alliance at www.sprayfoam.org.

[5] Shipping Temperature-Sensitive Products, Inbound Logistics, October 2003, http://www.inboundlogistics.com/cms/article/shippingtemperature-sensitive-products/

solvent(s), if any, in the SPF system to be tested should be provided to the selected laboratory in advance of testing.[6]

8. Laboratory Sample Receipt and Preparation

8.1 The laboratory logs the sample upon receipt. Note any apparent damage to the shipping container. If applicable, record parameters obtained during transport from the data logger. Remove the layered PET bag from the shipping container and note any damage to the bag. Prior to testing, the sample shall be stored in its original unopened layered PET bag at typical indoor conditions. During storage in the laboratory, the sample shall be protected from chemical contamination and exposure to temperatures in excess of 25°C and relative humidity in excess of 65 %. Sample shall not be refrigerated or placed in a freezer. The temperature and relative humidity of the storage location must be documented.

8.2 The sample is tested as soon as possible to reduce loss of VOCs from the sample. Unless otherwise specified, testing of the sample is to begin within 48 hours from the time the sample was sprayed. Document the date and time when the bag is opened. After the layered PET bag is opened, a specimen should be prepared for chamber testing within 20 minutes.

8.3 Use these steps to prepare the test specimen of open-cell SPF insulation for testing in small-scale chambers following Guide D5116:

8.3.1 Remove the sample from the bag. Remove the aluminum foil, and separate the sample from the HDPE substrate, maintaining the sample integrity. Note any observable defects, and if necessary, reject the sample due to such defects. Photograph the sample.

8.3.2 The specimen shall be cut between 8 and 9 cm in thickness to simulate the typical thickness of this product type when installed in buildings. Do not remove the surface skin of the SPF unless it is specified by the preparer in 7.5.4. If the surface skin is not to be removed, cut the bottom of the specimen to achieve the proper height. In either case, record whether or not the skin was removed. Use a clean knife or saw blade free of solvents and organic contaminants for cutting. Do not use a hot knife.

8.3.3 Trim the sides of the sample following the same procedure so the final specimen fits as tightly as possible into a rectangular holder with only the top surface exposed. The holder is a box constructed of stainless steel with an open top and 9 (±0.5) cm high sides. The side dimensions are selected to provide the desired product loading ratio in the chamber (m^2/m^3) based on the exposed face of the specimen and the chamber volume. Prior to use, the holder is cleaned with alkaline detergent and water, rinsed with deionized water, rinsed with methanol, and then dried. Alternative cleaning methods may be used provided they meet the laboratory quality control background/blank chamber concentration requirements. Transfer the specimen to the holder so the surface is nearly flush with the top of the holder and there are no gaps larger than 1 mm on the sides between the specimen and the holder. If the gaps are larger than 1 mm, the testing laboratory shall use clean stainless steel or PTFE shims between the specimen and holder walls to reduce the gaps to no larger than 1 mm. The top of the SPF specimen should be within 1 cm of the top of the holder, but the specimen should not protrude above the holder. Quickly place the specimen into the chamber testing environment. Record the elapsed time from removal of the sample from the bag and the insertion of the specimen into the chamber. After chamber testing has been completed, photograph the prepared specimen.

8.4 Use these steps to prepare the test specimen of closed-cell SPF insulation for testing in small-scale chambers following Guide D5116:

8.4.1 Remove the sample from the bag and separate the sample from the substrate, maintaining the sample integrity. Note any observable defects, and if necessary, reject the sample due to such defects. Photograph the sample.

8.4.2 The specimen shall be between 4 and 5 cm in thickness to simulate the typical thickness of this product type when installed in buildings. The height of the sample will not usually require cutting; however, minor trimming may be necessary. Do not remove the surface skin of the SPF unless it is specified by the preparer in 7.5.4. In either case, record whether or not the skin was removed. If the specimen needs to be cut, use the same cutting procedures as described for open-cell SPF insulation.

8.4.3 Trim the sides of the sample following the same procedure so the final specimen fits as tightly as possible into a rectangular holder as described for open-cell samples. The stainless steel holder has an open top and 5 (±0.5) cm high sides. Transfer the specimen to the holder so the bottom of the specimen rests on the bottom of the holder and there no gaps larger than 1 mm on the sides between the specimen and the holder. The top of the SPF specimen should be within 1 cm of the top of the holder, but the specimen should not protrude above the holder. Quickly place the specimen into the chamber testing environment. Record the elapsed time from removal of the sample from the bag and the insertion of the specimen into the chamber. After chamber testing has been completed, photograph the prepared specimen.

8.5 Follow these steps to prepare the test specimen of open-cell and closed-cell SPF insulation for testing in micro-scale chambers described in Practice D7706:

8.5.1 Micro-scale chambers typically are small cylinders that range in volume from a few mL to 150 mL. When used for surface emission testing, specimens must be cut to fit snugly into the chamber body. Alternately, specimens are cut to fit into cylindrical cups or rings that are placed in the chamber body.

8.5.2 Do not remove the surface skin of the SPF unless it is specified by the preparer in 7.5.4. In either case, record whether or not the skin was removed. Using a fabricated, circular cutting tool, cut the specimen to the correct diameter to fit into the chamber body or circular holder, and cut the specimen to the height needed to achieve the desired headspace in the chamber. If the surface skin is not to be removed, trim the bottom of the specimen to achieve the proper height. Quickly insert it into the chamber and close the chamber lid to initiate testing. Record the elapsed time from removal of the sample

[6] Due to the typically proprietary nature of SPF formulations, the SPF insulation product manufacturer may need to enter into a non-disclosure agreement with the testing laboratory.

from the bag and the insertion of the specimen into the chamber to begin testing. After chamber testing has been completed, photograph the prepared specimen.

9. Quality Control

9.1 The SPF insulation product manufacturer or the organization preparing the sample of SPF insulation is required to keep a detailed record of the spray and sample parameters as described in 7.3. This record is transmitted to the test laboratory and becomes part of the record for the test.

9.2 In order to preserve the chemical integrity of the sample, which will be shipped to a remote laboratory, the sample is packaged as described in 7.5, and the time between spraying of the sample and initiation of testing is minimized. The overall precision associated with sample preparation and testing can be assessed by preparing and testing replicate samples from the same product lot.

9.3 Construct an empty sample holder (with shims, if used) to serve as a laboratory blank for subsequent emissions testing.

10. Keywords

10.1 aldehydes; amine catalysts; blowing agents; chamber testing; diisocyanates; emissions; semi-volatile organic compounds; spray polyurethane foam insulation; SVOC; VOC; volatile organic compounds