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Standard Practice for Measuring Chemical Emissions from Spray Polyurethane Foam (SPF) Insulation Samples in a Large-scale Ventilated Enclosure¹

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1. Scope

1.1 This practice describes procedures for measuring the chemical emissions of volatile and semi-volatile organic compounds (VOCs and SVOCs) from spray polyurethane foam (SPF) insulation samples in a large-scale ventilated enclosure.

1.2 This practice is used to identify emission rates and factors during SPF application and up to three days following application.

1.3 This practice can be used to generate emissions data for research activities or modeled for the purpose to inform potential reentry and reoccupancy times. Potential reentry and re-occupancy times only apply to the applications that meet manufacturer guidelines and are specific to the tested formulation.

1.4 This practice describes emission testing at ambient room and substrate temperature and relative humidity conditions recognizing chemical emissions may differ at different room and substrate temperatures and relative humidity.

1.5 This practice does not address all SPF chemical emissions. This practice addresses specific chemical compounds of potential health and regulatory concern including methylene diphenyl diisocyanate (MDI), polymeric MDI (MDI oligomeric polyisocyanates mixture), flame retardants, aldehydes, and VOCs including blowing agents, and catalysts. Although specific chemicals are discussed in this practice, other chemical compounds of interest can be quantified (see target compound and generic formulation list in [Appendix X1](#)). Other chemical compounds used in SPF such as polyols, emulsifiers, and surfactants are not addressed by this practice. Particulate sizing and distribution are also outside the scope of this practice.

1.6 Emission rates during application are determined from air phase concentration measurements that may include particle bound chemicals. SVOC deposition to floors and ceilings is

also quantified for post application modeling inputs. SVOC emission rates should only be used for modeling purposes for the duration of data collection.

1.7 Four quantification methods are described for isocyanates. The method chosen should consider safety issues such as flammability, the expected concentration, the presence of isocyanate aerosol during the phase of interest (during and post application), and if the tested SPF is high or low pressure.

1.8 This practice references similar standard practices for design, construction, performance evaluation, and use of full-scale chambers for chemical emission testing.

1.9 This practice references methods for the collection and analysis of air samples.

1.10 This practice applies to two-component open cell and closed cell SPF insulation system formulations that are processed using high-pressure or low-pressure installation processing practices and equipment.

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

1.12 *This standard does not purport to address all of the safety concerns, if any, associated with its use. The application of SPF in a ventilated enclosure has the potential to generate a hazardous condition putting the individual responsible for spraying inserts at risk. It is the responsibility of the user of this standard to establish appropriate health and safety procedures and require appropriate certified personal protective equipment (PPE) to minimize chemical exposure. Individuals entering the ventilated enclosure during and after SPF application, for any amount of time, are expected to wear appropriate PPE.*

1.13 *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.*

1.14 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

¹ This practice 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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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
- D1914** Practice for Conversion Units and Factors Relating to Sampling and Analysis of Atmospheres
- 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)
- D6670** Practice for Full-Scale Chamber Determination of Volatile Organic Emissions from Indoor Materials/Products
- D7859** Practice for Spraying, Sampling, Packaging, and Test Specimen Preparation of Spray Polyurethane Foam (SPF) Insulation for Testing of Emissions Using Environmental Chambers
- 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
- E741** Test Method for Determining Air Change in a Single Zone by Means of a Tracer Gas Dilution
- E779** Test Method for Determining Air Leakage Rate by Fan Pressurization

2.2 ISO Methods:³

- 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
- ISO 17734-1** Determination of Organonitrogen Compounds in Air Using Liquid Chromatography and Mass Spectrometry – Part 1: Isocyanates Using Dibutylamine Derivatives

2.3 Government Agency Methods:

- OSHA Method PV 2018** Diethanolamine
- OSHA Method PV 2111** Ethanolamine
- OSHA Method PV 2116** Aminoethanolamine
- OSHA Method Org 113** 1,1-Dichloro-1-fluoroethane (Freon 141b), 1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)
- NIOSH Manual of Analytical Methods (NMAM) Method 2540** Ethylenediamine

NIOSH Manual of Analytical Methods (NMAM) Method 5600 Organophosphorus Pesticides

ULC CAN-S705.2 Standard for Thermal Insulation – Spray Applied Rigid Polyurethane Foam, Medium Density – Application

2.4 Industry Methods:

American Chemistry Council (ACC) Diisocyanates Panel – Considerations for Modifications to NIOSH 5521 and OSHA 47 Air Sampling Methods for Diphenylmethane Diisocyanate (MDI)

3. Terminology

3.1 For terms commonly used in ASTM standards, including this practice, refer to Terminology **D1356**. For an explanation of units, symbols, and conversion factors, refer to Practice **D1914**. For terms specific to full-scale chamber determination of volatile organic emissions from indoor materials/products refer to Practice **D6670**. For additional SPF definitions refer to Practice **D7859** and Test Method **D8142**.

3.2 Definitions:

3.2.1 *A-side, n*—one part of a two-component SPF system, typically polymeric methylene diphenyl diisocyanate (MDI) consisting predominately of 4,4'-MDI and higher molecular weight oligomers of MDI. Low-pressure systems may include a blowing agent propellant other additives in addition to the MDI.

3.2.2 *B-side, n*—one part of a two-component SPF system, typically a formulated resin blend polyol or resin system, consisting of polyols, with smaller amounts of catalyst(s), flame retardant(s), blowing agent(s), and other additives.

3.2.3 *chamber loading ratio, n*—see definition in Practice **D6670** and Guide **D5116**.

3.2.4 *clean air, n*—see definition in Practice **D6670**.

3.2.5 *closed cell SPF, n*—SPF that contains cells or voids that are not interconnected; closed cell SPF insulation typically has a density between 24 Kg/m³ to 32 Kg/m³ when fully cured.

3.2.6 *emission factor, n*—see definition in Practice **D6670**.

3.2.7 *emission rate, n*—see definition in Terminology **D1356**.

3.2.8 *full-scale ventilated enclosure, n*—a full-scale chamber that can be used for the application of formulated SPF products and the measuring of chemical emissions from those products.

3.2.9 *high-pressure application, n*—installation practice where a SPF system is applied to a substrate at processing equipment pressures ranging from 68 atm to 102 atm (1000 psi to 1500 psi).

3.2.10 *low-pressure application, n*—installation practice where a SPF system is applied to a substrate at processing equipment pressures ranging from 10 atm to 20 atm (150 psi to 300 psi); low-pressure processing equipment may include mechanical systems or two-cylinder “kits” of the A-side and B-side that are either pre-pressurized or externally pressurized.

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, Chemin de Blandinnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <https://www.iso.org>.

3.2.11 *open cell SPF, n*—SPF that contains cells or voids that are largely interconnected; open cell SPF insulation typically has a density between 6.4 Kg/m³ and 9.6 Kg/m³ when fully cured.

3.2.12 *reentry times, n*—the time elapsed after installation of SPF in a building when applicators, helpers and other trade workers may enter the building and resume operations without the need for PPE.

3.2.13 *reoccupancy times, n*—the time elapsed after installation of SPF insulation in a building when building occupants may resume building operations and activities.

3.2.14 *semi-volatile organic compound (SVOC), n*—for an in-depth discussion, see Guide **D8141**.

3.2.15 *time zero, n*—see definition in Practice **D6670**.

3.2.16 *tracer gas, n*—see definition in Practice **D6670**.

4. Summary of Practice

4.1 This practice provides a standardized approach for spraying SPF insulation in a large-scale ventilated enclosure and measuring selected emissions for prediction of potential impact on concentrations in buildings.

4.2 This practice provides guidelines for using a large-scale ventilated enclosure for testing SPF-sprayed wall cavity inserts containing formulated SPF products.

4.3 This practice has been developed for measuring chemical emissions during and following SPF application. The enclosure and methods of evaluation presented in this practice are useful for a variety of purposes including: (1) assessing the impact of environmental factors such as air change rate, air speed, and turbulence on emissions, (2) generating emissions data that can be used in indoor air quality (IAQ) models to predict indoor environmental concentrations in buildings, (3) developing scale-up methods (for example, from small-scale microchambers to a full-scale chamber scenario), (4) evaluating performance of cleaning devices and surface cleaning practices.

4.4 The SPF application may take 5 min to 60 min depending on the type of SPF applied and the enclosure dimensions. SPF is sprayed onto substrates in the enclosure that is supplied with clean air at a rate of 10 h⁻¹. Air samples are collected at specified times on various sampling media for determination of concentrations of emissions. Two hours after the conclusion of the application phase the air change rate is decreased to 0.3 h⁻¹ and air sampling is continued at intervals specified in this practice. Emission rates of specific chemicals are then estimated from the concentration-time data, loading ratio and air change rate.

5. Significance and Use

5.1 The demand for SPF insulation in homes and commercial buildings has increased as emphasis on energy efficiency increases. In an effort to protect the health and safety of both trade workers and building occupants due to the application of SPF, it is essential that reentry/reoccupancy-times into the structure where SPF has been applied, be established.

5.2 Concentrations of chemical emissions determined in large-scale ventilated enclosure studies conducted by this practice may be used to generate source emission terms for IAQ models.

5.3 The emission factors determined using this practice may be used to evaluate comparability and scalability of emission factors determined in other environments.

5.4 This practice was designed to determine emission factors for chemicals emitted by SPF insulation in a controlled room environment.

5.5 New or existing formulations may be sprayed, and emissions may be evaluated by this practice. The user of this practice is responsible for ensuring analytical methods are appropriate for novel compounds present in new formulations (see **Appendix X1** for target compounds and generic formulations).

5.6 This practice may be useful for testing variations in emissions from non-ideal applications. Examples of non-ideal applications include those that are off-ratio, applied outside of recommended range of temperature and relative humidity, or applied outside of manufacturer recommendations for thickness.

5.7 The determined emission factors are not directly applicable to all potential real-world applications of SPF. While this data can be used for VOCs to estimate indoor environmental concentrations beyond three days, the uncertainty in the predicted concentrations increases with increasing time. Estimating longer term chemical concentrations (beyond three days) for SVOCs is not recommended unless additional data (beyond this practice) is used, see **(1)**.⁴

5.8 During the application of SPF, chemicals deposited on the non-applied surfaces (for example, floors and ceilings) are the result of both gaseous phase emissions from the SPF and overspray. It is difficult to separate these two processes with current analytical methods. At present, the difference in how these two processes impact the long-term emissions is not known. This practice combines these two processes to generate data for modeling inputs.

6. Principles

6.1 *Tests Under Uniform Concentration Conditions* (Practice **D6670**)—VOCs—Assuming that the concentration of each emitted VOC tested in the enclosure air is uniform, the concentration is then governed by the mass balance equation:

$$\frac{VdC(t)}{dt} = R(t) - QC(t) - S(t) \quad (1)$$

where:

V = air volume of the enclosure excluding air volume taken by test specimens, m³,

t = time, h,

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

- $C(t)$ = concentration of the emitted VOC in the enclosure or air exhausted from the enclosure at time t (can be measured in the room or at the room return or exhaust air ducts), mg/m³,
- $R(t)$ = emission rate at time t , of the source in the room, mg/h,
- Q = exhaust airflow rate (measured at the exhaust air duct or determined by a tracer gas test), m³/h, and
- $S(t)$ = sink term representing loss (or re-emission if negative) of the VOC at time t due to adsorption/desorption effect on the interior surfaces of the room and ducts, mg/h. It also represents particle deposition and loss by chemical reactions as is the case with MDI, pMDI.

6.2 Based on Eq 1, the VOC emission rates of a test specimen as a function of time can be determined by measuring the concentrations of the enclosure air or air exhausted from the enclosure and the clean airflow rate. The concentrations and the clean airflow rate must be determined for the same temperature condition since the air volume changes with air temperature. Both closed cell and open cell SPF products generate significant heat as the chemical reaction occurs, therefore the exhaust air temperature can be 15 °C or greater than the supply air temperature. If the concentration is measured at the enclosure exhaust while airflow rate is measured at the enclosure supply, the supply airflow rate must be first adjusted to the equivalent airflow rate under the room/exhaust air temperature (that is, multiplied by the ratio of room/exhaust to supply air temperature in degrees Kelvin) before it is used for determining the emission rate.

6.3 In addition to the uniform VOC concentration assumption, Eq 1 assumes no air entry into the enclosure (infiltration) other than the supply air, and a negligible VOC concentration at the supply air, compared to that measured in the enclosure or at the room exhaust. The validity of using Eq 1 depends on how well the enclosure's actual operation meets these assumptions. Therefore, the performance of the enclosure must be evaluated against certain criteria in order to obtain reliable and reproducible test results (see Section 9).

6.4 When using post-application phase chamber concentration data to calculate emission rates and emission factors, it is important to recognize that the emission rate of a chemical from the material to the air may be controlled by the rate of diffusion of the chemical to the surface of the material (source limited) or controlled by the rate of mass transfer from the surface of the material to the bulk air of the room (gas-phase limited). Section 6.6 of Guide D8141 provides guidance on determining if a chemical should be treated as source limited or gas-phase limited. If the chemical is source limited (typical of VOCs) the emission rate can then be modelled as an area-specific emission approach. If the chemical is gas-phase limited (typical of SVOCs), then the limiting factor is the rate of mass transfer of the chemical from the surface to the air and the emission rate should be modeled using a gas-phase limited mass transfer model.

6.5 Flame retardants such as tris(1-chloro-2-chloropropyl) phosphate (TCPP) may be source limited or gas-phase limited

depending on the SPF type, enclosure turbulence and the temperature (Guide D8141). The volatility of a given chemical, and therefore the mass transfer limitation, may be temperature dependent (approximately a doubling of vapor pressure/concentration for every 10 °C increase in temperature).

6.6 During SPF application, emissions may be non-uniform due to air turbulence and the high rate of aerosol/vapor emissions in the proximity of the spray gun. For these reasons, it may be necessary to sample two or more locations in the enclosure, and/or at the exhaust duct during SPF application. The relative percent difference of concentrations between samples collected at two locations over the same time period should be 15 % or less to indicate the enclosure was well mixed. If the RPD is greater than 15 % for a given chemical, it should be sampled in locations specified in 7.2.10.

$$RPD = \frac{100|C_1(t) - C_2(t)|}{(C_1(t) + C_2(t))/2} \quad (2)$$

where:

RPD = relative percent difference, %, and
 $C_i(t)$ = concentration at sample location i .

7. Facilities and Equipment

7.1 Enclosure Location:

7.1.1 The enclosure should be located in a clean and air-conditioned laboratory with sufficient space for the enclosure and related equipment. The laboratory must be sufficiently large to house the enclosure, and if deemed necessary, a ventilated egress chamber. The egress chamber is also a ventilated enclosure between the enclosure and laboratory for the spray applicator to doff safety equipment following SPF application. The laboratory must be sufficiently large to accommodate associated ventilation, required health and safety equipment, and control equipment, and provide space around the enclosure for access to air sampling ports, if air sampling is conducted by personnel located outside the enclosure. The laboratory must also have a ventilated spray facility (spray booth) for the control of chemical emissions prior to and after evaluations (for example, preparing chemical pumps and flushing spray equipment).

7.2 Enclosure Design:

7.2.1 The make-up air supplied to the enclosure must be conditioned to have a standard air temperature of 25 °C ± 1.0 °C and a relative humidity of 50 % ± 5 %.

7.2.2 The enclosure itself will not be temperature and humidity controlled, however, room temperature and the temperature of the substrate to be sprayed must be recorded prior to SPF application. Room temperature and relative humidity must be monitored and recorded during each air sampling session (SPF application and post application).

7.2.3 The enclosure should be at least 2.4 m × 2.4 m × 2.4 m (8 ft × 8 ft × 8 ft) to accommodate the individual applying SPF onto wall cavity inserts that may be mounted in wood frames or otherwise attached to the walls of the enclosure. If wood frames are selected, they should be 5 cm × 15 cm × 213 cm (2 in. × 4 in. × 7 ft) with a space between the studs of 41 cm (16 in.). The insert placed between each stud should be 15 cm × 41 cm × 213 cm (6 in. × 16 in. × 7 ft). The face of the studs

can be covered with either folded cardboard from the inserts or an extra cardboard strip may be placed on exposed stud surfaces. The total number of inserts sprayed should be sufficient to achieve the enclosure loading ratio of 0.7 m²/m³ to 0.8 m²/m³.

7.2.4 Larger enclosures of varying dimensions are acceptable provided the SPF surface area is adjusted to attain a loading ratio of 0.7 m²/m³ to 0.8 m²/m³.

7.2.5 The enclosure and air distribution components must be constructed of materials such as stainless steel or aluminum that have been selected to minimize absorption of SPF chemicals and can be cleaned between uses.

7.2.6 The enclosure door may be connected to an egress chamber. The door and gaskets around the door must be constructed of non-absorbing or non-emitting materials. If an egress chamber is used, the ventilation must remain off when the spray applicator enters or exits the enclosure.

7.2.7 All openings to the enclosure must be sealed to minimize leakage. Openings should be sufficient to allow air sampling devices to pass in and out of chamber.

7.2.8 The enclosure's supply air and exhaust ventilation must be controlled over air change rates ranging from 0.3 h⁻¹ to 10 h⁻¹.

7.2.9 A mixing fan(s), separate from the enclosure air supply and exhaust ventilation, must be installed and operated in a manner to ensure mixing throughout the enclosure. The fan(s) should be constructed from stainless steel or other non-porous material so that it can be cleaned between experiments.

7.2.10 For reactive (isocyanates) or non-well mixed compounds (SVOCs) sampling media should be placed at a height of 1.0 m to 1.50 m and a distance inside the room to be a minimum of 0.75 m from the spray foam surface or enclosure walls. Air sampling pumps and flow controllers can be located outside of the chamber.

7.2.11 For all other chemicals, the sample location can be at the same location as 7.2.10, at the exhaust duct, or other location that provides a representative sample. If sample is collected in the exhaust duct, any temperature changes must be accounted for when determining the chamber concentration as described in 6.2.

7.2.12 Sampling ports may be used to connect the sampling media to the sampling pumps using tubing. Sampling media and connecting tubing should be inserted and withdrawn into the chamber through the sample ports. Sample ports must be designed to be sealed around sample tubing during experiments and sealed when not in use.

7.2.13 High-pressure formulations should be sprayed using commercially available spray equipment. Low-pressure formulations should be sprayed using equipment provided by the manufacturer or commercially available spray equipment recommended by the manufacturer.

7.2.14 High-pressure spray equipment as well as the SPF chemicals must be stationed outside the enclosure. After equipment preparation work, such as flushing the equipment with the SPF to be tested, the spray gun and hose are passed through an opening in the wall of the enclosure. When in place, the opening is sealed around the hose.

7.2.15 Low-pressure internally pressurized or externally pressurized cylinders must be placed outside the enclosure, if possible. The spray gun and hose are passed through an opening in the wall of the enclosure in a manner similar to high-pressure systems. When in place, the opening is sealed.

7.2.16 The enclosure must have on-line monitoring of test conditions such as airflow rates (make-up and exhaust fan speed), air temperature, relative humidity, and differential pressure inside the enclosure (indicator of air infiltration).

8. Air Sampling Methods

8.1 Air sampling and analytical methods described in this section represent validated methods that may be used to evaluate atmospheric conditions during SPF application and post application. Alternative air sampling and/or analytical methods equivalent to those listed in this section may be substituted, as appropriate.

8.2 Air sampling should be conducted throughout the application phase. Air sample volumes should be set in accordance with individual air sampling method requirement and adjusted to avoid saturation of sorbent materials. Air sampling procedures, including the establishment of air sample flowrates and air sample times should be determined by the individual(s) collecting the air samples and the laboratory performing the air sample analyses prior to initiating the SPF chemical emissions testing.

8.3 SPF VOC and SVOC emissions generated during SPF application are generally significantly greater than those generated during post application, therefore consideration must be given to the selection of the appropriate air sampling method (Table 1). For certain formulations VOC/SVOC sorbent materials described in Test Method D8142 may be suitable for both SPF application and post application segments provided air sample volumes are adjusted to avoid sorbent saturation. In the event appropriate sample volumes cannot be achieved, the air sampling methods described in 8.4 should be followed during the application phase and methods described in 8.5 should be followed for post application emission testing.

8.4 Air sampling methods that may be used *during* SPF application:

8.4.1 *Carbonyls*—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.

8.4.2 *Amine Catalysts*—VOCs emitted during application, such as amine catalysts, may be collected on XAD-2 treated with 10 % 1-naphthylisothiocyanate (NITC) and analyzed by high-pressure liquid chromatography (HPLC) with ultraviolet (UV) detector according to OSHA Methods PV 2116, PV 2018, PV 2111, and NMAM Method 2540.

8.4.3 *Blowing Agents*—Chlorofluorocarbon and hydrofluoro-olefin blowing agents may be collected using by drawing air through tubes containing Anasorb sorbent (150 mg and 75 mg). Samples are desorbed with carbon disulfide analyzed by GC-FID using OSHA Org 113.

8.4.4 *Flame Retardants*—Flame retardants such as TCPP emitted during application may be collected on OSHA Versatile Sampler (OVS) with 13mm quartz filter and XAD-2

TABLE 1 Summary of Air Sampling Methods

Target Chemical	During Application	Method	Post Application	Method
Carbonyls	Silica gel treated with 2,4-dinitrophenylhydrazine (DNPH)	ASTM D5197	Silica gel treated with 2,4-dinitrophenylhydrazine (DNPH)	ASTM D5197
Amine Catalysts	Multi Sorbent Tube and/or XAD-2 treated with 10 % 1-naphthylisothiocyanate (NITC)	ASTM D8142 and/or OSHA Methods PV 2116, PV2018, PV2111, NMAM 2540 (Amine catalysts)	Multi Sorbent Tube	ASTM D8142
Flame retardants	Multi Sorbent Tube and/or OSHA Versatile Sampler (OVS) with 13 mm quartz filter and XAD-2 sorbent (270/140)	ASTM D8142 and/or NMAM 5600	Multi Sorbent Tube	ASTM D8142
Blowing agents	Multi Sorbent Tube and/or Anasorb sorbent (150 mg and 75 mg)	ASTM D8142 and/or OSHA Org 113	Multi Sorbent Tube	ASTM D8142
MDI – High-Pressure Systems	Impinger solution containing toluene and either dibutylamine or 1,2 PP with back-up 13 mm glass fiber filter with 1,2-PP and/or CIP10 device with a sampling head that makes use of a centrifuged liquid medium composed of DMPS + MOPIP	ASTM D5932 (2,4 -TDI), D6561 (HDI), D6562 (HDI) and/or ISO 17734 – 1 and/or IRSST and/or ACC Diisocyanates Panel	Impinger solution containing toluene and either dibutylamine or 1,2 PP with back-up 13 mm glass fiber filter with 1,2-PP and/or ASSET (trademarked) EZ4-NCO Dry Sampler and/or ISO-CHEK . Dual filter cassette; 5- μ m PTFE membrane traps aerosol phase, glass fiber filter impregnated with MAMA collects vapor phase	ASTM D5932 (2,4 -TDI), D6561 (HDI), D6562 (HDI) and/or ISO 17734 – 1 and/or ACC Diisocyanates Panel
MDI – Low-Pressure Systems	Impinger solution containing toluene and either dibutylamine or 1,2 PP with back-up 13 mm glass fiber filter with 1,2-PP and/or ASSET EZ4-NCO Dry Sampler and/or ISO-CHEK . Dual filter cassette; 5- μ m PTFE membrane traps aerosol phase, glass fiber filter impregnated with MAMA collects vapor phase	ASTM D5932 (2,4 -TDI), D6561 (HDI), D6562 (HDI) and/or ISO 17734 – 1 and/or ACC Diisocyanates Panel	Impinger solution containing toluene and either dibutylamine or 1,2 PP with back-up 13 mm glass fiber filter with 1,2-PP and/or ASSET EZ4-NCO Dry Sampler and/or ISO-CHEK . Dual filter cassette; 5- μ m PTFE membrane traps aerosol phase, glass fiber filter impregnated with MAMA collects vapor phase	ASTM D5932 (2,4 -TDI), D6561 (HDI), D6562 (HDI) and/or ISO 17734 – 1 and/or ACC Diisocyanates Panel

sorbent (270/140). Samples are analyzed by GC, flame photometric detection (FPD) according to NMAM 5600.

8.4.5 *Isocyanates*—Several methods are available to collect 2,4-MDI, 4,4-MDI, and pMDI during and post SPF application (Table 2). Isocyanates are highly reactive and contact with a derivatizing reagent is needed to quantify isocyanates. Air samplers can use filters that are impregnated with a derivatizing agent, an impinger that contains a derivatizing agent dissolved in a solvent, and/or a derivatizing agent packed into a denuder tube (2, 3). Each sampling method has advantages and disadvantages related to collection efficiency and ease of sample collection. The selected method should be based upon the degree of aerosol generation and sampling location.

8.4.5.1 Particle size plays an important role on the efficacy of the collection method. Data gathered on particle-size distribution is limited, and available data suggests that high-pressure systems generate relatively larger particles compared to low-pressure systems (4-6). For high-pressure systems, solid phase samplers (such as the ASSET sampler) appear to result in lower MDI air concentrations than impingers (7). It is unknown whether a similar bias between impinger and solid-phase samplers exists for low-pressure systems. Liquid devices (CIP10 and impinger) may not capture smaller particles. However, for impingers, a filter treated with a derivatizing agent can be placed at the impinger outlet to capture smaller particles (American Chemistry Council (ACC) Diisocyanates

TABLE 2 2,4-MDI, 4,4-MDI, and pMDI Air Sampling Methods

Method	Advantages	Disadvantages	Reference
Impinger	Collection efficiency for monomer and oligomers approaches 100 %	Cannot be operated through a sampling port Flammable absorbing solution Post device derivatizing filters needed to capture smaller particles	(9, 10)
CIP 10	Collects MDI monomers in same range as impinger method. Oligomer captured more efficiently than impingers	The air sampling pump and sampling head come as a single unit, therefore the device cannot be operated through a sampling port outside the enclosure Only samples larger particles sizes No post device filters can be attached to capture smaller particles	(4)
ISO-CHEK	Dual filter separates monomer and oligomers Dry sampler Small size allows sampling through a sampling port	Heavy filter loading with aerosol may prevent contact with derivatizing agent Requires field filter desorption	(6)
ASSET EZ4-NCO	Dry sampler Small size allows sampling through a sampling port Does not require field filter desorption.	Monomer and oligomer concentrations underestimated during SPF application for high-pressure systems when compared to the impinger method	(7)

Panel, ISO 17734-1). Solid devices, such as (ISO-CHEK, ASSET EZ4-NCO) derivatizing agent utilization issues may lead to lower readings compared to liquid methods (8).

8.4.5.2 Dry filter samplers or nonflammable liquid samplers minimize the exposure to sampling personnel and keep flammable chemicals out of the chamber. Dry isocyanate samplers can be inserted into the chamber from external sampling ports rather than having personnel entering the chamber to change impingers.

8.4.5.3 Impingers are commonly used, but like the CIP 10, cannot easily be inserted into the chamber. Use of these methods may require two people to be in the chamber during application phase: one to spray and one to operate air sampling equipment.

8.4.5.4 The impinger method requires the use of 1 (2-pyridyl piperazine (1,2-PP) or dibutylamine as derivatizing agents suspended in toluene. A 13 mm filter cassette equipped with a 13 mm glass fiber filter is attached to the impinger outlet for the purpose of capturing particles less than 2 micrometers in size. Although the impinger method has close to 100 % collection efficiency, there are flammability issues related to the toluene absorbing solution and that glass impingers are susceptible to breakage during air sampling. Like the CIP 10 method, air sampling from outside the enclosure can be difficult.

8.4.5.5 The CIP10 device is equipped with a sampling head that makes use of a centrifuged liquid medium composed of derivatizing agents dimethylpolysiloxane (DMPS) + 1-(2-methoxyphenyl) piperazine MOPIP (0.5 mg mL⁻¹). The MDI–MOPIP monomer and oligomer derivatives are analyzed by reverse phase liquid chromatography with UV detection (HPLC/UV). The air sampling pump and sampling head come as a single unit, therefore the device cannot be operated

through a sampling port outside the enclosure. For high-pressure systems, the CIP10 collects monomer concentrations equal to the impinger method and oligomer concentrations better than the impinger method as demonstrated by the positive bias between 76 % and 113 % (4).

8.4.5.6 The ISO-CHECK (trademarked) filter method that simultaneously traps and separates both monomers and oligomers at the point of collection for highly sensitive physical and chemical characterizations of diisocyanates. The ISO-CHEK uses a two-stage filter arrangement, the first stage being a 5 micrometer PTFE filter for aerosols followed by a second stage glass fiber filter impregnated with 9-(N-methylaminoethyl) anthracene (MAMA) for the vapor phase. The primary disadvantage to the ISO-CHECK method as well as most filter methods is, MDI concentrations may be underestimated due to heavy filter loading where the aerosol may not contact the MAMA derivatizing agent. A second disadvantage is the PTFE filter must be placed in a MOPIP in toluene derivatizing solution prior to shipping to the laboratory for analysis (6).

8.4.5.7 The ASSET EZ4-NCO Dry Sampler (trademarked) is composed of two sections: a filter located at the base of the sampler and a denuder section that runs the length of the sampler. Both components are treated with a dibutylamine (DBA) derivatizing agent. Samples are analyzed in accordance with ISO method 17734-1. The samplers have the advantage that they are sufficiently small to fit through a sampling port along with other sorbent tube air sampling media. In addition, the method does not require a derivatization step prior to shipment. The major disadvantage to using the dry sampler during SPF application is, concentrations of monomers and oligomers may be underestimated when compared to the impinger method (7).