Developing ASTM Standards for Measuring VOC and SVOC Emissions from Spray Polyurethane Foam Insulation Products

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CPI Emissions Task Force Objective

Develop methods for SPF standardization that can be used by manufacturers to evaluate potential emissions released from SPF insulation samples.

Participate in research to support the development of SPF product emissions standards through ASTM Subcommittee D22.05 on Indoor Air Quality.

ASTM Subcommittee D22.05 on Indoor Air

SPF Emissions Task Group Members

- Industry (CPI, III)
- Regulatory (US EPA, CPSC, etc.)
- Instrument Vendors
- Consultants
- Air Quality Testing Labs
- Certification Programs
- Other Stakeholders

ASTM Work Items

- ✓ **ASTM WK30960 (Recently published as D7859-13e1)**

  Standard Practice for Spraying, Sampling, Packaging, and Test Specimen Preparation of Spray Polyurethane Foam (SPF) Insulation Samples for Environmental Chamber Emissions Testing

- **ASTM WK40293**

  New Practice for Estimating Chemical Emissions from Spray Polyurethane Foam (SPF) Insulation using Micro-Scale Environmental Test Chambers

- **ASTM WK40292**

  New Test Method for Measuring Chemical Emissions from Spray Polyurethane Foam (SPF) Insulation Samples in Environmental Test Chambers with Thermal Desorption and Gas Chromatography / Mass Spectroscopy (TD-GC-MS)

Generic SPF Formulations

Three generic SPF formulations were developed by CPI to evaluate the test methods for measuring emissions.

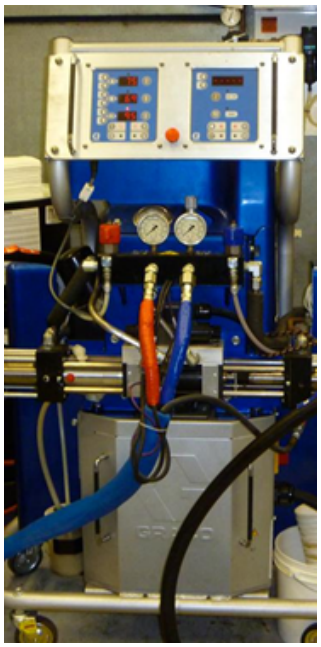
The formulations represent the following sample types:

Spray Polyurethane Foam
Open-cell 1/2 pound
High Pressure

Spray Polyurethane Foam
Closed-cell 2 pound
High Pressure

Spray Polyurethane Foam
Kit Formulation
2 Component, Low Pressure

ASTM Standard Practice D7859-13e1



Standardizes the procedures for spraying, sampling, packaging and test specimen preparation of SPF insulation for testing of emissions using environmental chambers.

Practice is applicable to both closed-cell and open-cell SPF insulation.

Spraying, Packaging and Transport

Spraying Parameters

- Specialized equipment with trained operators
- Samples are sprayed in spray booth under controlled conditions
- Samples sprayed onto sheets of HPDE as substrate
- Sample thickness specified for both open-cell and closed-cell SPF

Packaging and Transport

- Samples allowed to cure for 1-hour
- Samples are wrapped with aluminum foil then placed into layered polyethylene terephthalate (PET) bags with minimal headspace
- Samples shipped in insulated secondary container
- Electronic data loggers can be used to monitor temperature, pressure and relative humidity during transport
- Samples and data record shipped to laboratory via overnight delivery service

Laboratory Requirements

General Knowledge of SPF Formulations

- Define target compounds for emission monitoring
- Surface skin may need to be removed to simulate trimming of SPF insulation to wall studs or other structural elements

Sample Receipt and Storage

- Chain of custody
- Samples are stored in unopened bags at typical indoor office conditions (no refrigeration or freezing)

Holding Time

- Chamber testing must begin within 20 minutes of opening the PET bag and within 48-hours from spraying

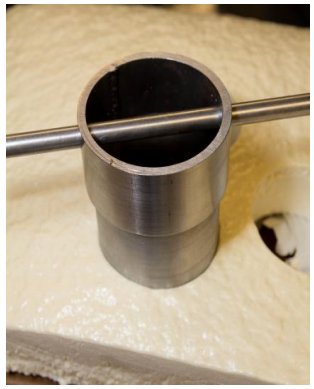
Preparation of Specimens

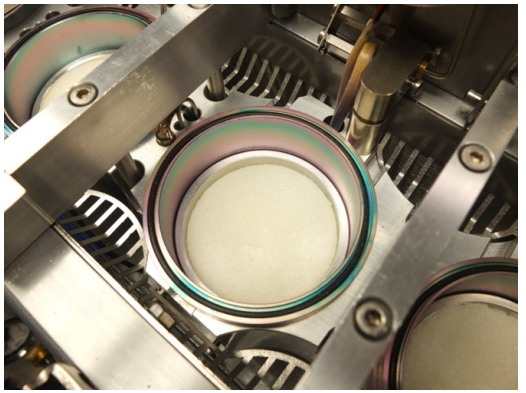
- Samples are cut to tightly fit into stainless steel sample holders or micro-scale chambers

ASTM Work Item WK40293

Describes the procedures to collect chemical emissions from SPF insulation samples using micro-scale environmental test chambers

Operating conditions minimize wall adhesion (sink effects) and maximize recovery of the SVOCs, which are captured onto sorbent tubes from the outlet of the micro-scale chamber





Micro-Scale Chamber Apparatus

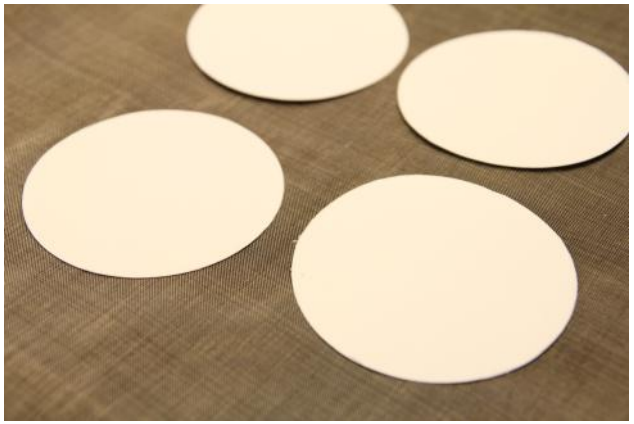
Apparatus shown below holds four micro-scale chambers, controls gas flow and temperature

After equilibration, samples are collected from exhaust ports at various times

Sampling from Micro-Scale Chamber

Internal 58mm internal glass-fiber filter with 1-(2-pyridyl)piperazine (PP)

Flow calibrator



90mm glass-fiber filters with PP to wipe chamber walls

Thermal desorption tubes

Silica gel with DNPH tube

ASTM Work Item WK40292

Analytical methodology utilizing thermal desorption GC/MS to measure the chemical emissions of semi-volatile and volatile organic compounds (SVOCs and VOCs) from SPF insulation samples

Thermal desorption tubes are used to collect air samples from:

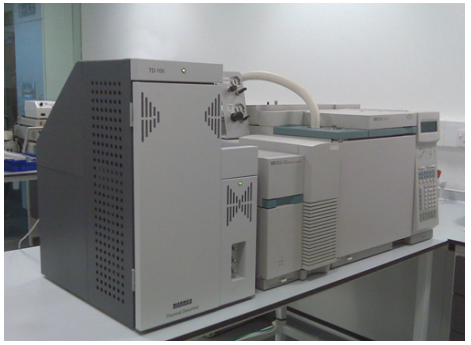
- Micro-scale or conventional test chambers
- Buildings to monitor indoor air concentrations

What is Thermal Desorption GC/MS?

VOCs and SVOCs are sampled onto thermal desorption tubes packed with a specific combination of sorbents containing glass wool, graphitized carbon and porous polymer adsorbent.

Samples can be stored in sealed containers prior to analysis.

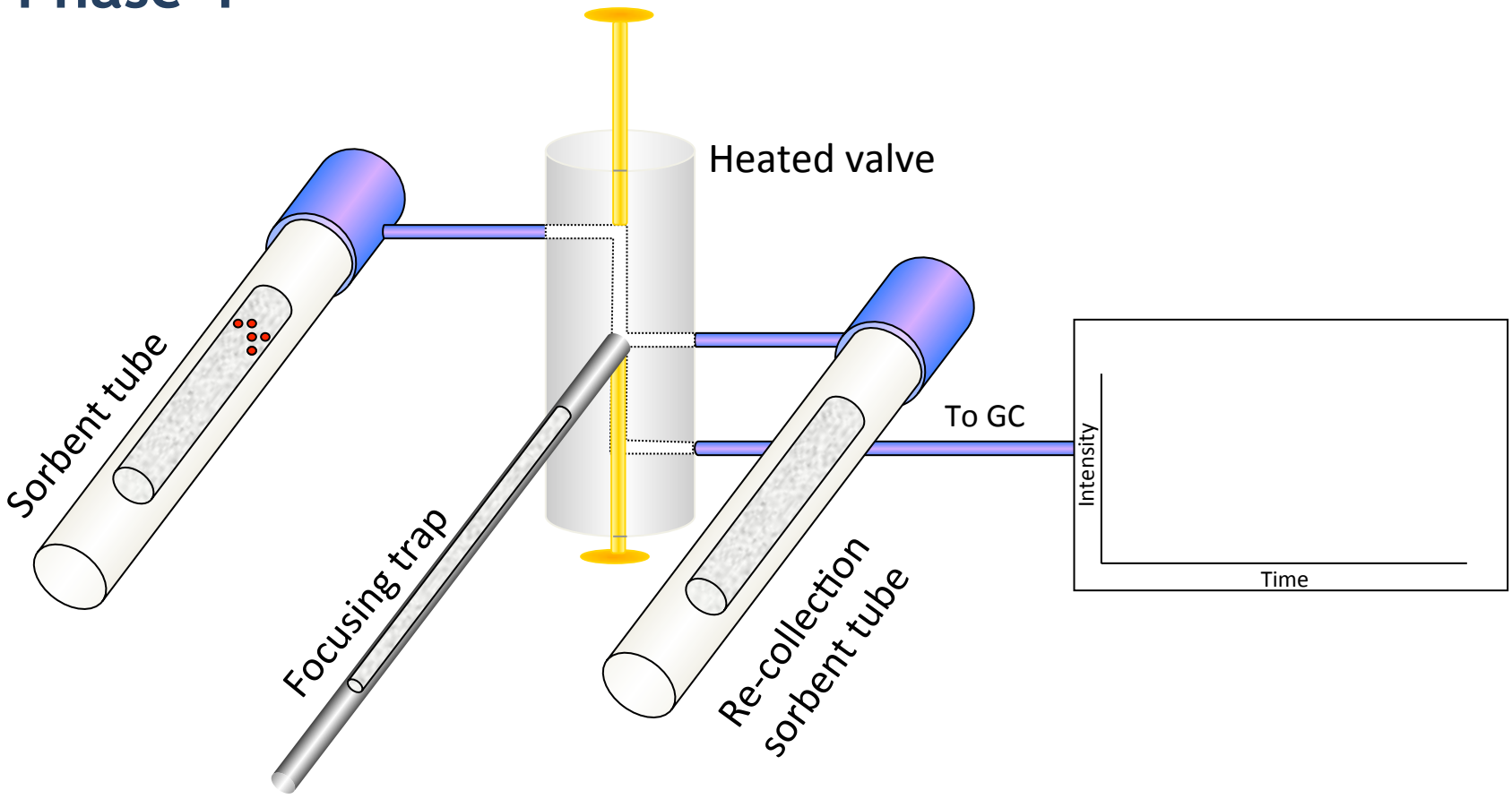
Captured organic compounds are released during a two-stage thermal desorption process and are identified and quantified by gas chromatography/ mass spectrometry.



Target compounds compared with reference standards; unknown compounds are tentatively identified with NIST Mass Spectral Library.

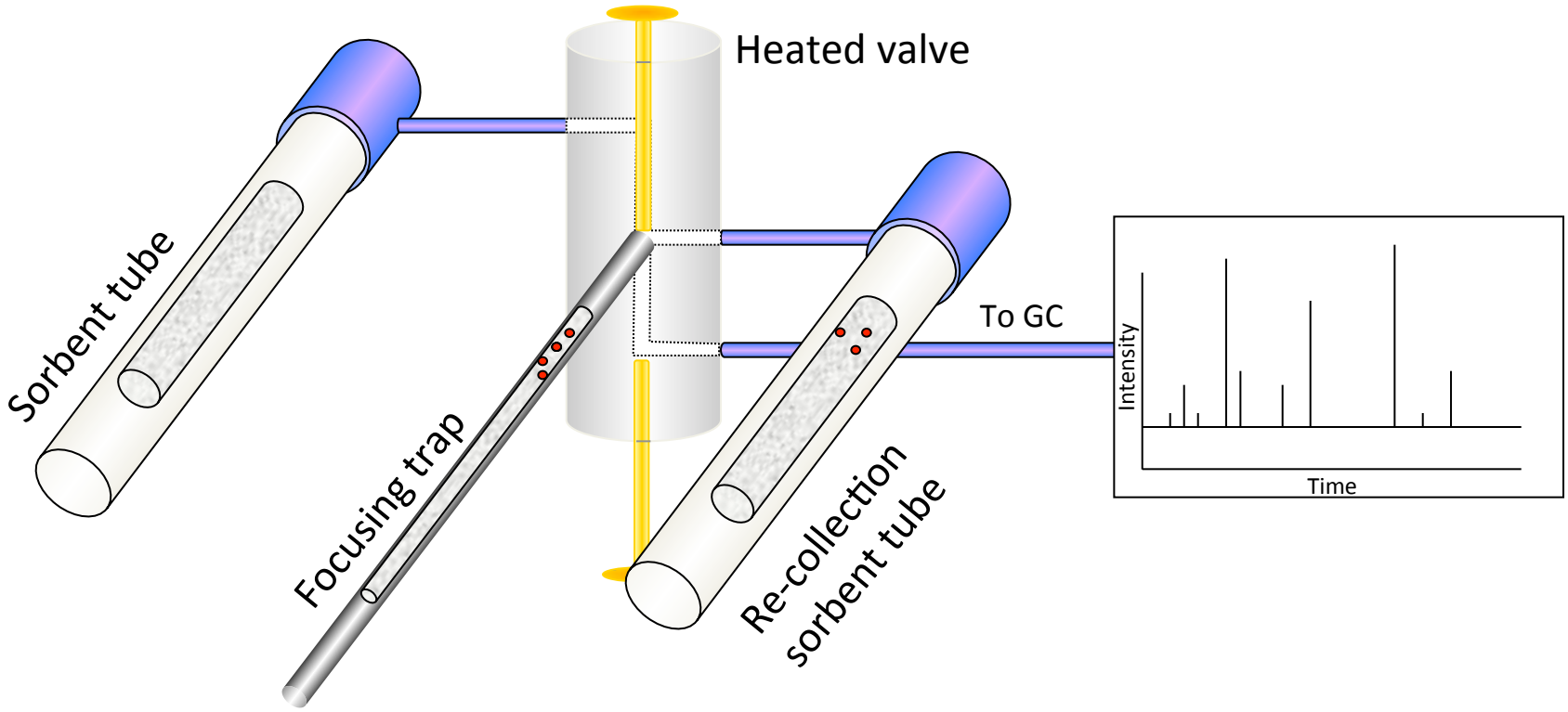
Two-Stage Thermal Desorption

Phase 1

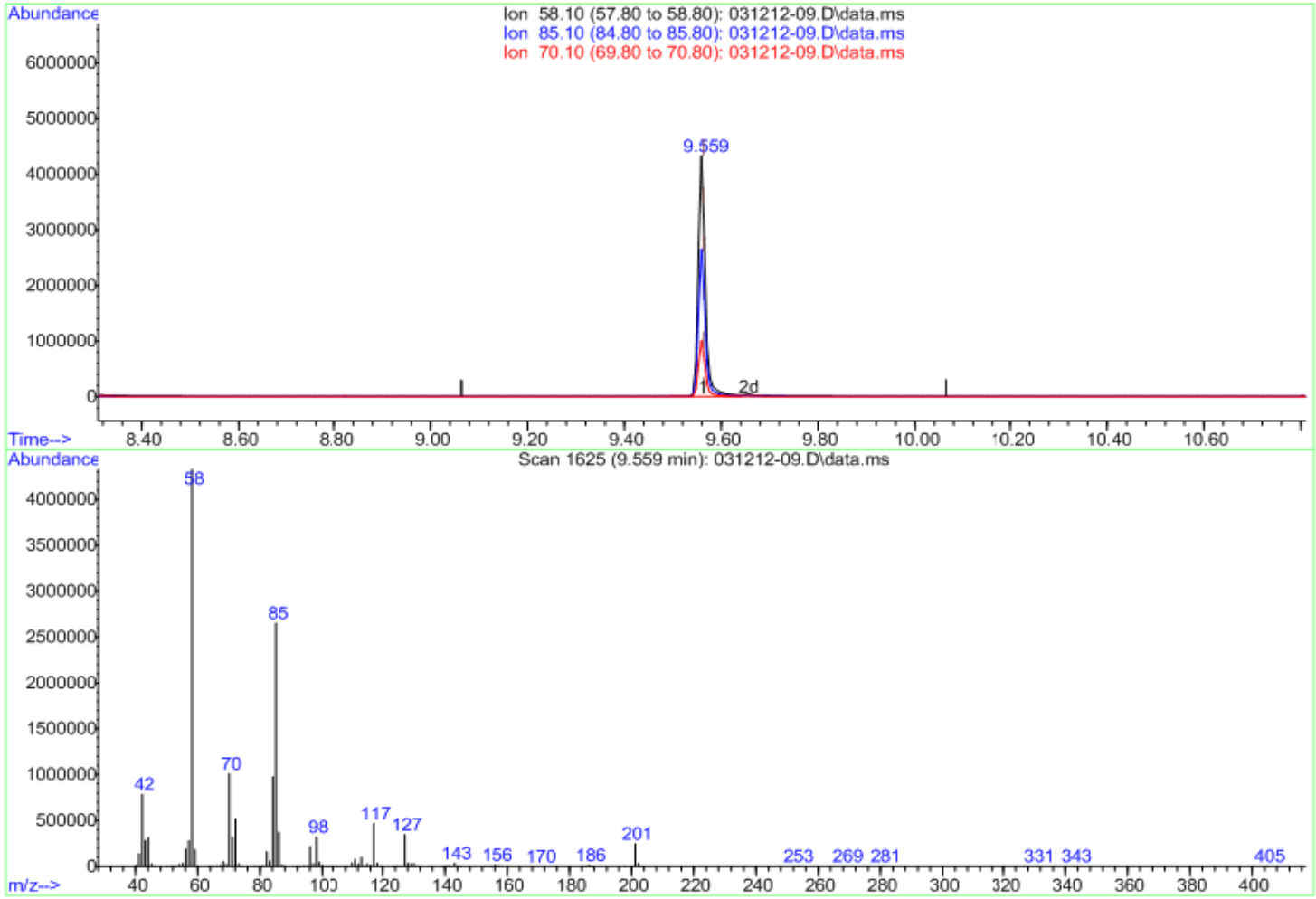


Two-Stage Thermal Desorption

Phase 2



Extracted Ion Chromatogram
Mass Spectrum



Generic SPF Target Compounds with Thermal Desorption GC/MS

Compound Name	Compound Type	Retention Time, minutes	Estimated Quantitation Limit, ng	Mean Recovery, %	Precision RSD, %
HFC-245fa	Blowing Agent	1.48	20	89.7	10.9
TMAEEA	Amine Catalyst	7.69	200	71.5	24.6
BDMAEE	Amine Catalyst	7.72	20	94.5	4.10
PMDTA	Amine Catalyst	8.26	50	92.9	3.52
DAPA	Amine Catalyst	9.56	100	96.2	3.38
TMIBPA	Amine Catalyst	9.65	200	68.1	13.3
TCPP	Flame Retardant	12.5	50	92.6	2.94

Evaluation of Data

Emissions factors

- Mass per surface area and time, µg / m^2 hour

Predicted building concentrations

- Loading factor in building, area /volume, m^2/m^3
- Air exchange rate (ventilation rate), fresh air exchanges / hour
- Predicted concentration values are typically calculated in µg/m^3

Evaluation of Data

Re-entry time

- Compare predicted concentrations at a given time with Occupational Exposure Limits (OELs)

Re-occupancy time

- Compare predicted concentration at a given time with Chronic Reference Exposure Limits (CRELs) or 1/100 OEL

Evaluate ventilation rates

- Evaluate ventilation rates to meet exposure limits or odor threshold limits

Research and Collaboration

Current Research Projects

- **Center for Polyurethane Industry (CPI)**
  - Further support development of analytical test methods
  - Optimize recovery of flame retardant and amine catalysts using micro-scale test chambers
- **International Isocyanate Institute (III)**
  - Evaluate prototype micro-scale test chambers optimized for MDI emissions

US EPA, Office of Research and Development

- Ongoing CPI and US EPA meetings to discuss current activities in each organization

ASTM D22.05 on Indoor Air

Subcommittee Ballot

- WK40292 Standard Test Method for Measuring SPF Chemical Emissions with Thermal Desorption Tubes and GC/MS
- WK40293 Standard Practice for using Micro Chamber to Measure SPF Emissions

Scheduled Meetings

- October 22, 2013, Jacksonville, Florida
- April 8, 2014, Toronto, Ontario, Canada

Acknowledgments

ASTM International Committee D22 on Air Quality

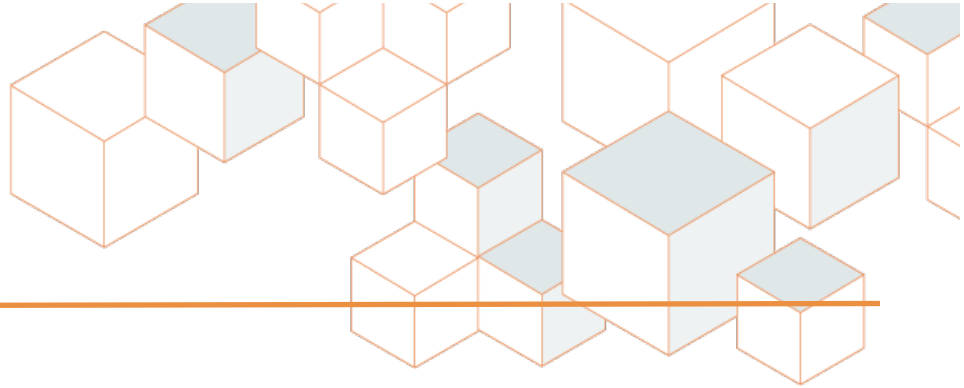
- Subcommittee D22.05 on Indoor Air

Bayer MaterialScience LLC

- Environmental Analytics Laboratory
- Product Safety and Regulatory Affairs

Center for Polyurethane Industry (CPI)

- SPF Emissions Task Force
- SPF Ventilation Task Force



Questions?