

Introduction

Chemical Safety and Human Health's research studies use the most robust and qualified analytical procedures. For the air collection and analysis of volatile organic compounds (VOCs) and aldehydes, our studies use UL's ISO 17025 accredited indoor air quality (IAQ) laboratory.

VOCs

For VOCs, air is collected on Tenax® TA (60/80 mesh) sorbent tubes that are thermally desorbed and analysed by thermal desorptiongas chromatography/mass spectrometry (TD-GC/MS). This technique scans for VOCs in the volatility range of n-hexane (C₆) to n-hexadecane (C₁₆) following methods presented in ASTM D6196 (ASTM, 2015), EPA Methods TO-17 (US EPA, 1999a) and TO-1 (US EPA, 1999b)). A laboratory-specific spectral database of approximately 700 VOCs is used to identify individual VOCs collected by the sampling media. The VOCs in this database have been previously found in indoor air and product emissions studies and validated by the laboratory for analysis using the laboratory-specific systems. By matching spectral characteristics and retention times based on the laboratory method, the identification is accurate with little uncertainty down to a quantitation level of approximately 0.5 μ g/m³ for most common VOCs.

For those VOCs detected but not identified by the laboratory spectral database, a general mass spectral library, available from the National Institute of Standards and Technology (NIST), which includes characteristics of more than 75,000 compounds, is used. Mass spectral characteristics are identified, and compounds identified if they showed an 80% match. All VOCs are quantified from multi-point calibration curves prepared using authentic



Environmental exposure sampling media for air sampling: Tenax[®] tube (left) and 2,4-dinitrophenylhydrazine (DNPH) cartridge (right) for volatile organic compounds (VOCs) and aldehydes respectively.

standards if available; otherwise, they were calibrated relative to toluene. Authentic standard calibrations are available for 73 specific VOCs that are the most commonly measured VOCs listed by various IAQ or regulatory programs including California's Proposition 65 (OEHHA, 2012), California's Chronic Reference Exposure Levels (OEHHA, 2019), EPA's Integrated Risk Information System (US EPA, 2018), Agency for Toxic Substances and Disease Registry's minimal risk levels (ATSDR, 2019), and the Occupational Safety and Health Administration's permissible exposure limits (OSHA, 2018). For compounds that did not have an authentic standard, the measurement is reported as a toluene equivalent. Calculations for total volatile organic compound (TVOC) levels are made by taking the total GC/MS scan response between $\rm C_6$ and $\rm C_{16}$ and converting to a concentration based on a toluene equivalent.

Aldehydes

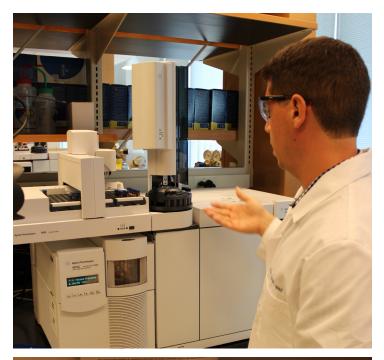
Formaldehyde or other aldehydes in the air are collected on 2,4-dinitrophenylhydrazine (DNPH) cartridges. The cartridges are extracted with acetonitrile and analysed using high-performance liquid chromatography (HPLC) following method ASTM D5197 (ASTM, 2016). Specific target species are quantified using a multi-point internal calibration method prepared from hydrazone derivatives of the pure compounds. Target aldehydes include: formaldehyde, acetaldehyde, 2-propenal, acrolein, propanal, 2-butenal, butanal, benzaldehyde, 3-methylbutanal, pentanal, 2-methylbenzaldehyde, 3-and 4-methylbenzaldehyde, hexanal, and 2,5-dimethylbenzaldehyde. Aldehydes are also reported down to a quantitation level of approximately 0.5 µg/m³.

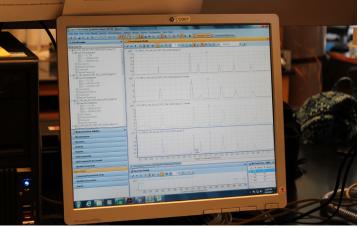
Quality measures

The overall accuracy of identification and quantification of VOCs and aldehydes is expected to be within 20%. Sample collection flow rates and analysis processes are optimized around expected VOCs levels. Duplicate VOC and aldehyde measurements show less than 10% as tracked by the laboratory's quality program. Detection limits for most VOCs and aldehydes typically fall from 0.01 to 0.1 μ g/m³, but quantitation levels of 0.5 μ g/m³ are reported for field studies. Compounds in the laboratory's IAQ database are analytically validated as part of the quality assurance program for a breakthrough, linearity, toluene equivalency, and percent recovery. Quality measurements are performed at levels typically found in the indoor air. The IAQ laboratory as part of its quality program participates in various proficiency programs for the analysis of formaldehyde and representative VOCs.

References

- ASTM, 2016. ASTM D5197-16 Standard Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air (Active Sampler Methodology). ASTM International, West Conshohocken, PA. https://doi.org/10.1520/D5197-16.2
- ASTM, 2015. ASTM D6196-15 Standard Practice for Choosing Sorbents, Sampling Parameters and Thermal Desorption Analytical Conditions for Monitoring Volatile Organic Chemicals in Air ASTM International, West Conshohocken, PA. https://doi. org/10.1520/D6196-15.2
- ATSDR, 2019. Minimal Risk Levels (MRLs) for Hazardous Substances.
- OEHHA, 2019. OEHHA Acute, 8-hour and Chronic Reference Exposure Level (REL) Summary. California Office of Environmental Health Hazard Assessment.
- OEHHA, 2012. PROPOSITION 65 SAFE HARBOR LEVELS : No Significant Risk Levels for Carcinogens and Maximum Allowable Dose Levels for Chemicals Causing Reproductive Toxicity. Office of Environmental Health Hazard Assessment, Sacramento, CA.





Instrumental analysis of flame retardants at Emory University, GC/ MS (top) and a gas chromatogram (bottom).

OSHA, 2018. OSHA Annotated Table Z-1.

US EPA, 2018. Integrated Risk Information System.

- US EPA, 1999a. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition Compendium Method TO-17 Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes. U.S. Environmental Protection Agency, Cincinnati, OH.
- US EPA, 1999b. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air - Second Edition Compendium of Method TO-01 Method for the Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Tenax[®] Adsorption and Gas Chromatography/Mass Spectrometry (GC/MS). U.S. Environmental Protection Agency, Cincinnati, OH.

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