Measurement of Ethylene Oxide from Ambient Air using SIFT-MS

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Overview

- Introduction to SIFT-MS
- ASTM test method
- Participants
- Method optimization
- Final method
- Timeline

Syft Tracer™: Industry-Scalable, Real-Time Trace Gas Analysis

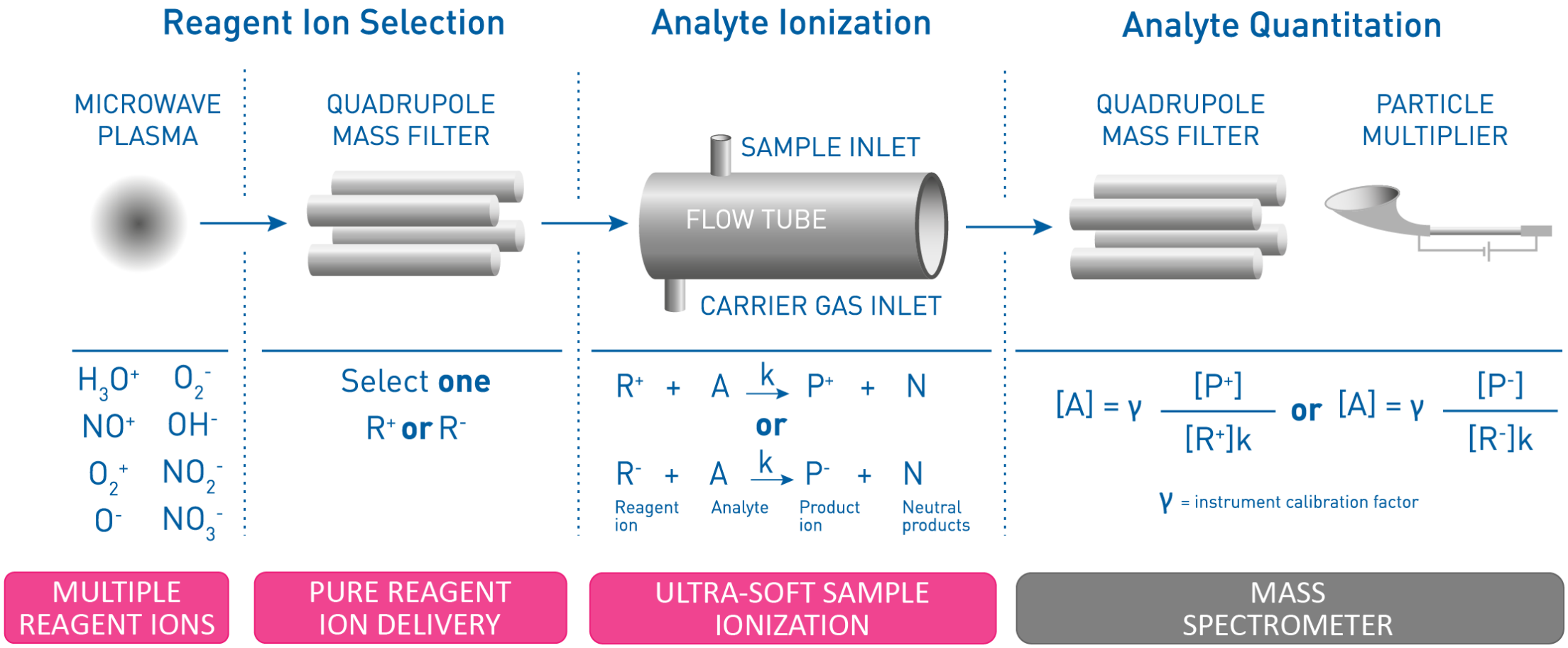
- Excellent multi-analyte sensitivity
- Highly selective and quantitative
- No chromatography or pre-concentration required
- *Fine Auto Retune* for optimal signal levels
- *Performance Authenticator* for superior analytical stability
- Hardware advancements to maximize system lifetime
- System optimized for 24/7 data collection
- Easy to operate and interpret data

Syft Tracer is the Solution for Diverse Markets and Applications

Relevant Markets and Applications:

- Pharma / CDMO
- Consumer goods
- **Environmental**
- Food, Flavors, & Fragrances
- **Petrochemical**
- Automotive
- Lab and Research
- Emerging Applications

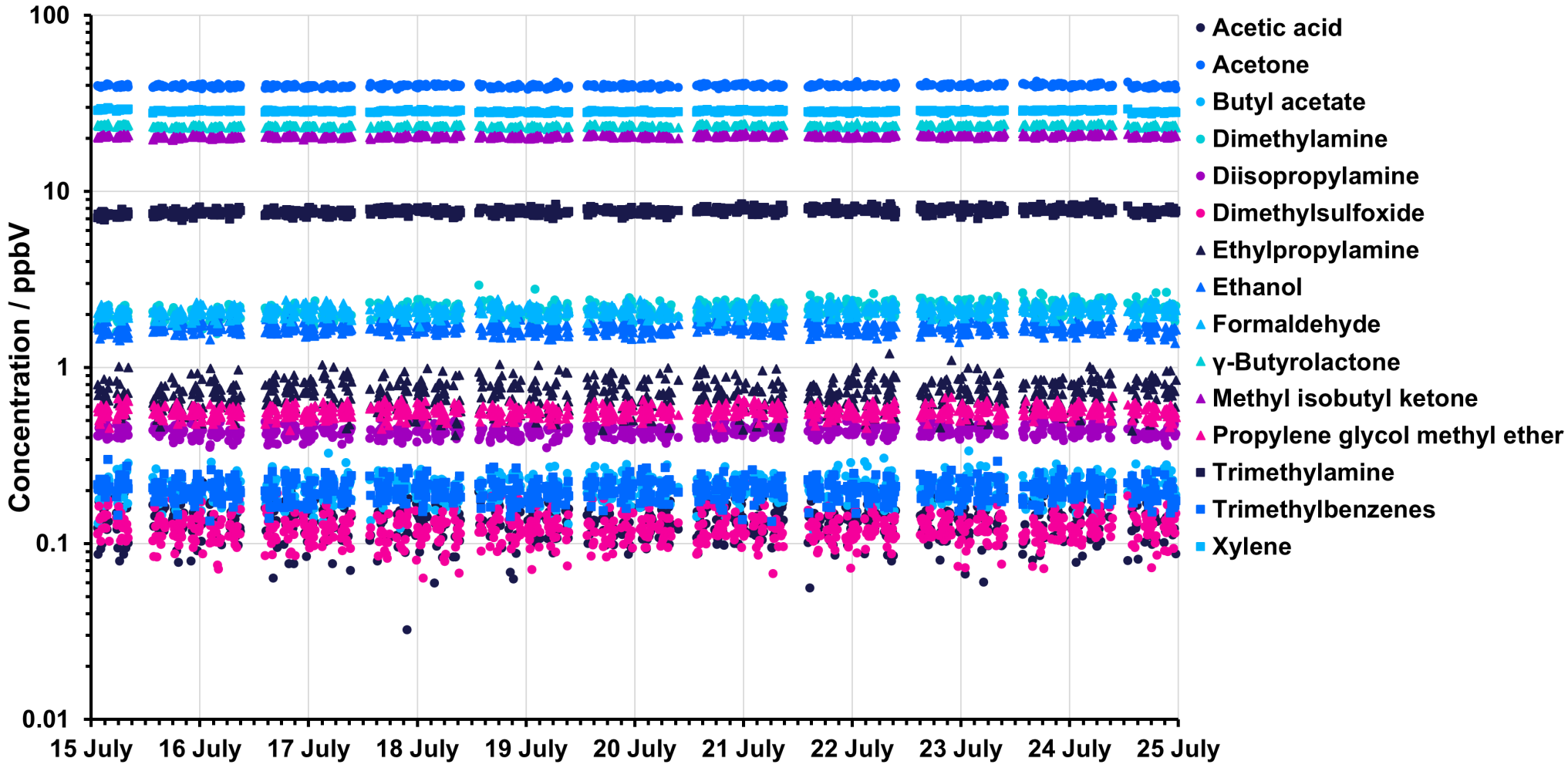
SIFT-MS: How it works



SIFT-MS: Comprehensive, real-time analysis

Class	Examples
hydrocarbons	alkanes, alkenes, aromatics, monoterpenes
oxygenates	alcohols, aldehydes (including formaldehyde), ketones, esters, ethers, carboxylic acids
nitrogen compounds	amines, amides, nitriles, nitrated organics, nitrosamines
sulfur compounds	mercaptans, thioethers, carbonyl sulfide
halogenated compounds	aliphatic and aromatic fluorides, chlorides, bromides and iodides
inorganics	ammonia, hydrogen cyanide, hydrogen sulfide, nitrogen dioxide, phosphine, hydrogen chloride, hydrogen fluoride, carbon dioxide, sulfur dioxide, ozone

Reproducible analysis – long-term stability



Why ethylene oxide?

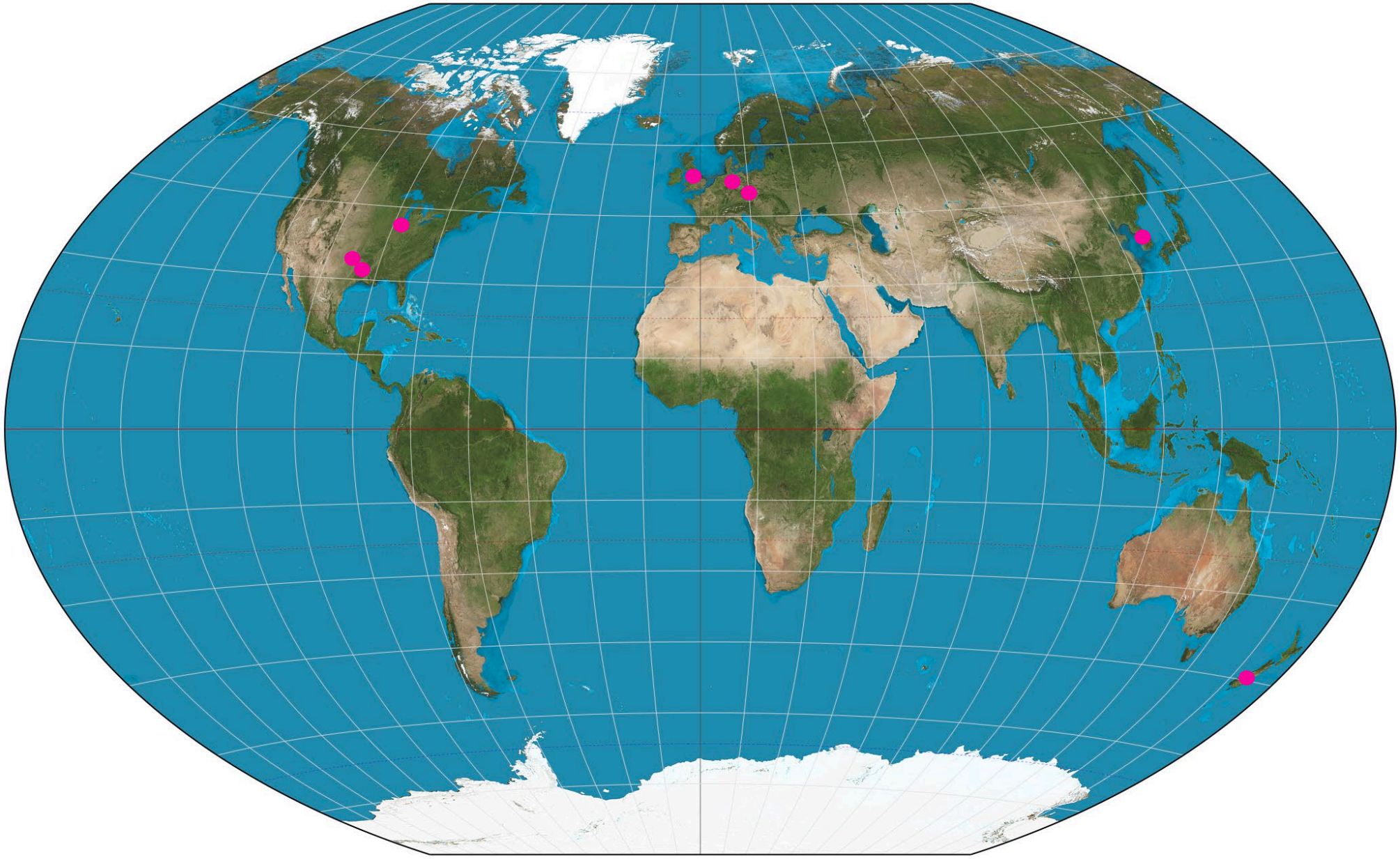
- Epoxide used in the production of many consumer products.
    - Detergents, thickeners, plasticizers, ethylene glycol
- In smaller quantities used as a pesticide and sterilizing agent.
- At room temperature is a flammable, carcinogenic, irritating and an anesthetic gas.
- HON rule announced April 2024 aiming to reduce emissions of toxic air pollutants, including ethylene oxide (EtO), from plants that produce synthetic chemicals, polymers and resins.

Goals of the ASTM test method:

WK67973 *New Standard Measurement of Ethylene Oxide in Ambient Atmospheres*

- Create a robust, reproducible method for the measurement of ethylene oxide from ambient air.
- Measure ethylene oxide with an independent product ion or using a subtraction approach to remove acetaldehyde interference.
- Compare LODs and MDLs achieved across multiple labs around the world (various iterations of 8-reagent ion instruments ranging from Voice200*ultra* to the Syft Tracer i8).

Labs participating in the ASTM Test Method Comparison



Labs participating in the ASTM Test Method Comparison

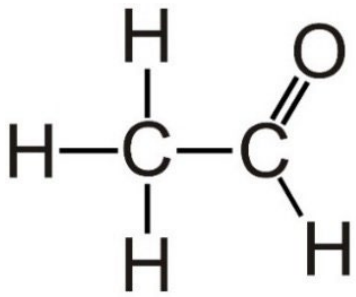
Country	Number of Labs	Configuration
United States	5	Ultra i8 (2), Tracer i8 (2), Tracer i3 (1)
Germany	1	Tracer i3
England	2	Ultra i8 (1), Tracer i8 (1)
Czech Republic	1	Ultra i8
South Korea	1	Ultra i3
New Zealand	2	Tracer i8

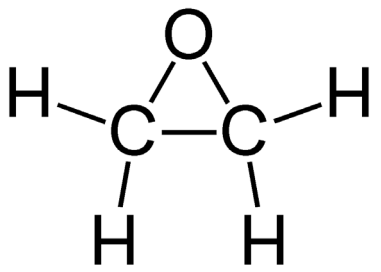
Multiple reagent ions can selectively measure ethylene oxide

	Reagent ion	Acetaldehyde	Ethylene Oxide
METHOD 1	H_3O^+	$C_2H_4O.H^+$ 1 m/z 45	$C_2H_4O.H^+$ 1 m/z 45
	NO^+	$C_2H_3O^+$ 1 m/z 43	$C_2H_4O.NO^+$ 1 m/z 74
METHOD 2	OH^-	$C_2H_3O^-$ 1 m/z 43	$C_2H_3O_2^-$ 1 m/z 59

Used together where acetaldehyde is subtracted from the $H3O^+$ measurement of the sum

Acetaldehyde

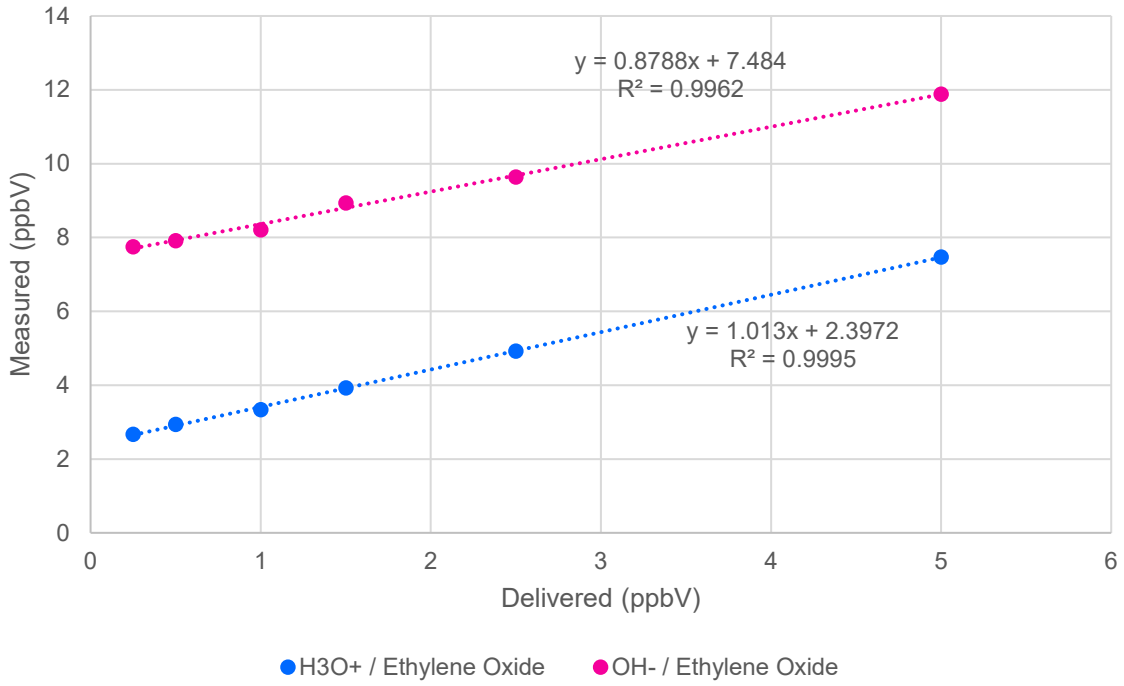




Ethylene Oxide

Swift et al. Analytical Methods, 2023.

Calibrating for Ethylene Oxide



250 pptV – 5 ppbV delivered (6-point standard curve)

Determination of Method Detection Limits

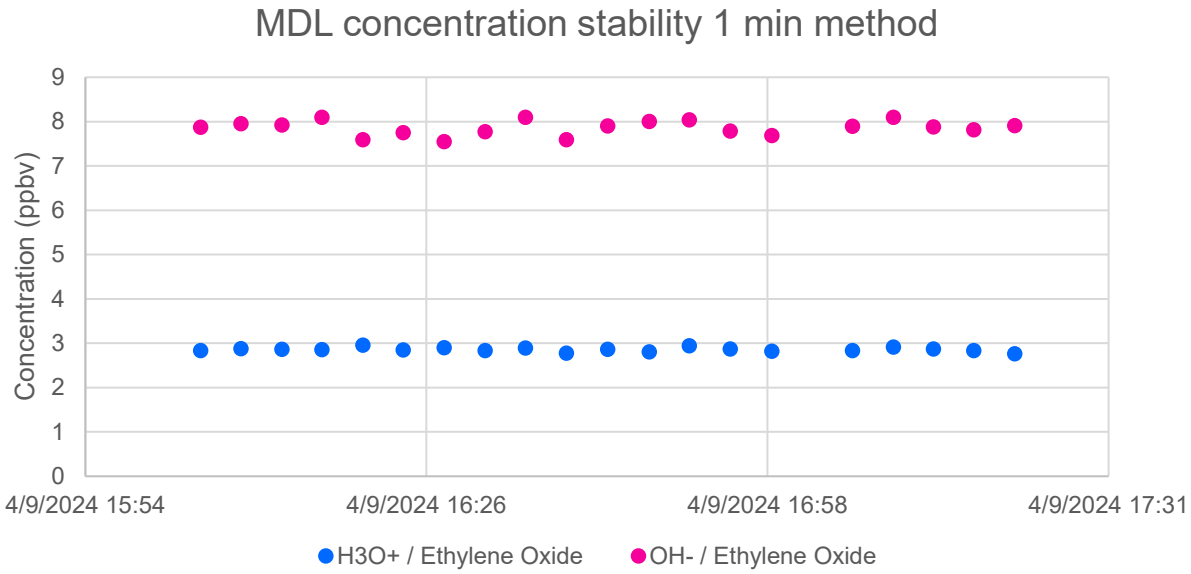
- MDL = 3.143 x SD of 7 replicate samples at 3-5 times the LOD

- Linearity indicates no issue down to 250 pptV and MDL is probably below this

- MDL evaluated at 500 pptV

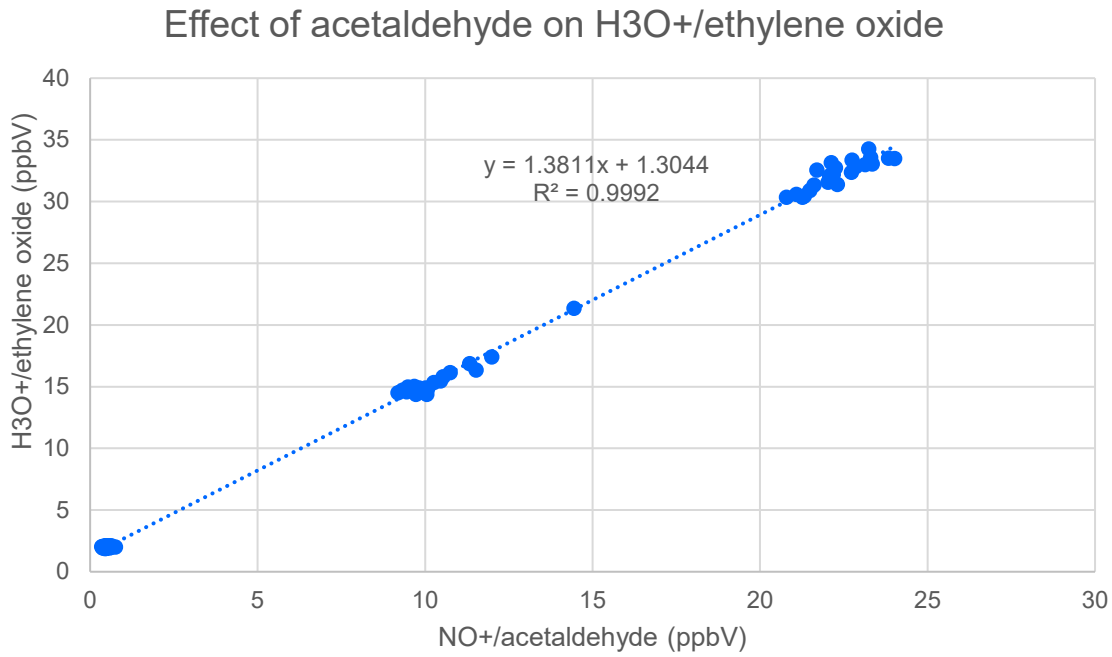
- H_3O^+ 7-scan MDLs from total of 20 scans
  160 pptV (60 second method)

- OH^- 7-scan MDLs from total of 21 scans
  575 pptV (60 second method)



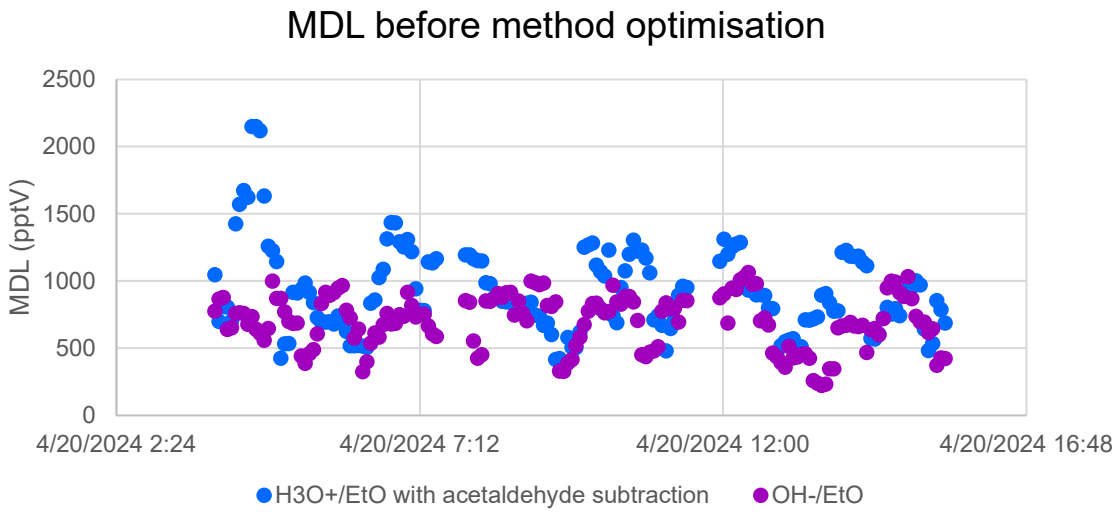
Ethylene oxide acetaldehyde subtraction

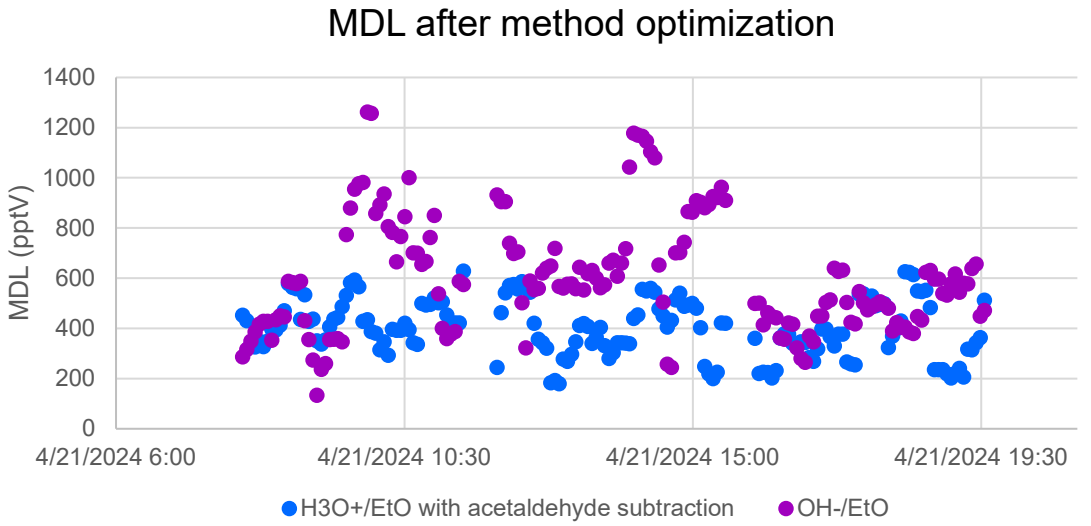
- Ethylene oxide and acetaldehyde both react rapidly to form H3O+/45
- Always expect some acetaldehyde to be present in samples so a subtraction is needed - use NO+/acetaldehyde concentration
- In order to subtract acetaldehyde concentration from EtO, the relationship between H3O+/ethylene oxide and NO+/acetaldehyde concentrations needs to be evaluated.
- First step is to find relationship between H3O+/ethylene oxide and NO+/acetaldehyde concentration
- **Subtraction is 1.38 x NO+/acetaldehyde concentration**



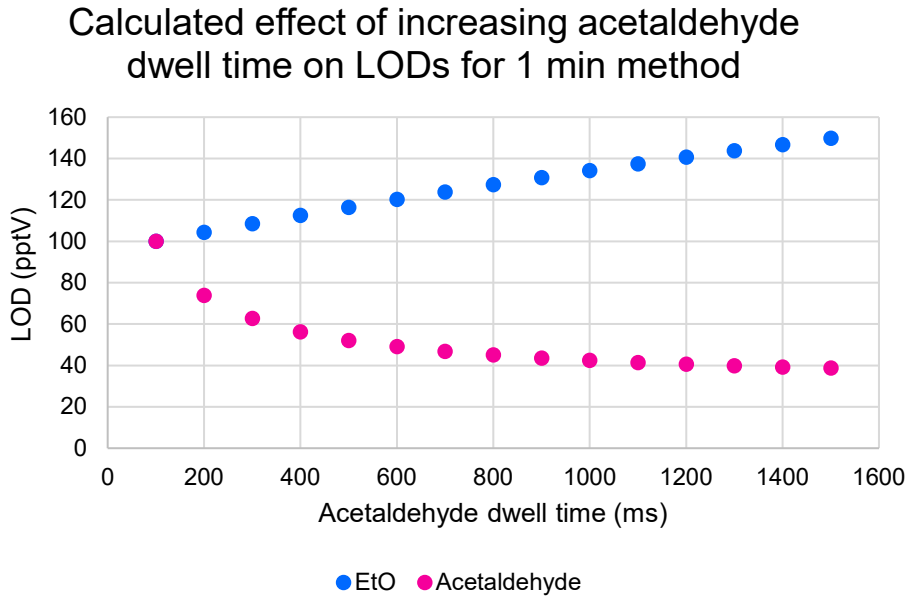
How does acetaldehyde presence affect EtO measurement?

- Remeasured the MDL in the presence of 2.5 ppbV acetaldehyde using the subtraction method.
- Initially found the H3O+/EtO MDL was significantly higher, higher than OH-/EtO
- The acetaldehyde measurement was the dominant contribution to the increased MDL
- Improving the acetaldehyde measurement should lower MDL
- H3O+/EtO MDL much improved in optimised method
- Now better than OH-/EtO





Dwell time adjustment to account for the slower reaction rate of acetaldehyde

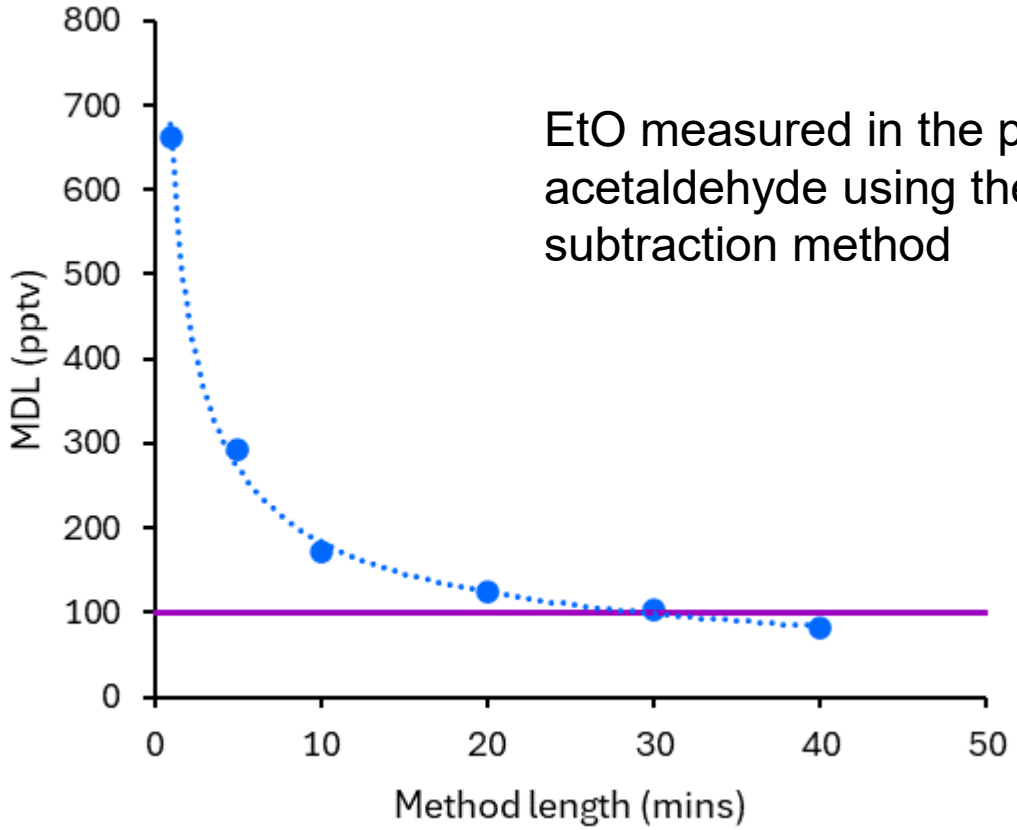


Compound	Reagent	Reaction rate	Branching ratio (%)	Mass (m/z)	Product	Scan	Calculate	Time limit (ms)	Count limit
Ethylene Oxide	H3O+	3.3E-9	100	45	C2H5O+	☑	☑	**300**	10000
Ethylene Oxide	H3O+	3.3E-9		63	C2H4O.H3O+	☑	☑	**300**	10000
Ethylene Oxide	H3O+	3.3E-9		81	C2H4O.H+.2H2O	☐	☐	100	10000
acetaldehyde	NO+	7.4E-10	80	43	CH3CO+	☑	☑	**750**	10000

EtO MDL in the presence of acetaldehyde is dependent on method length

Scan Time (mins)	EtO MDL (pptV)*
1	660
5	295
30	102

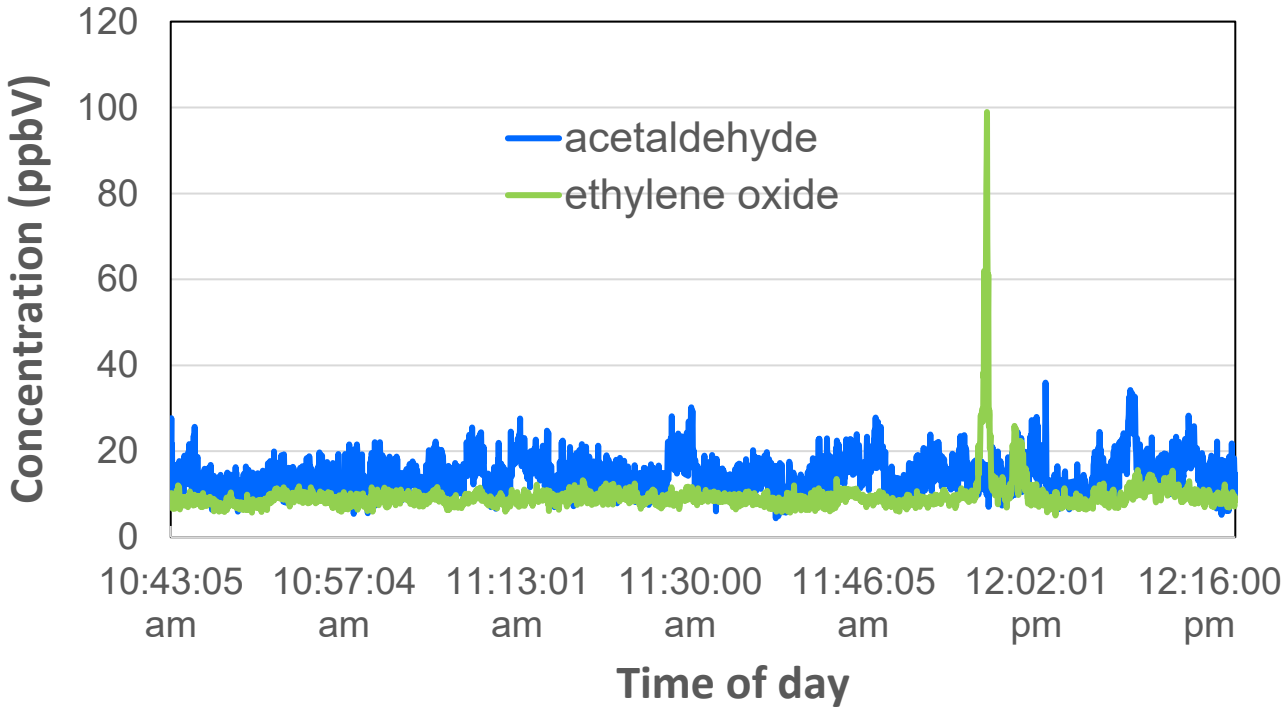
* With acetaldehyde present and subtraction applied



EtO measured in the presence of 2.5 ppbV acetaldehyde using the positive ion subtraction method

HON action level

Stationary measurement of ethylene oxide outside a chemical production plant

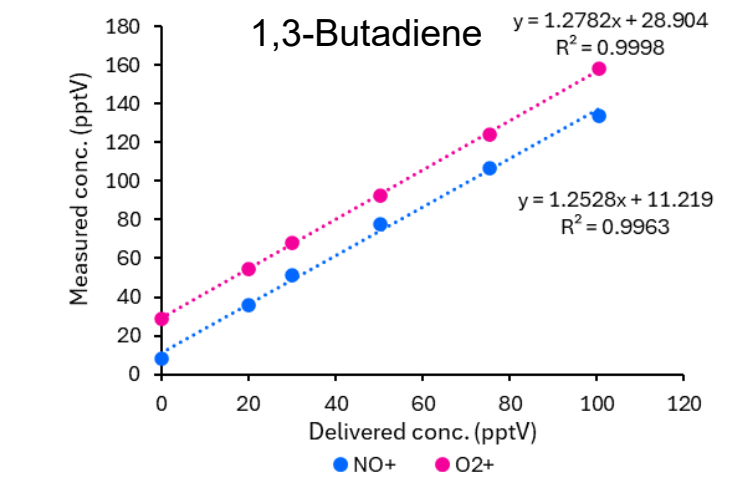


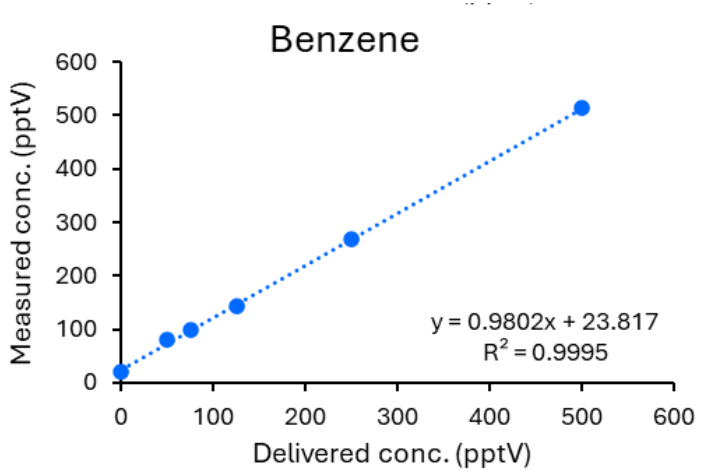
Other HON regulated VOCs

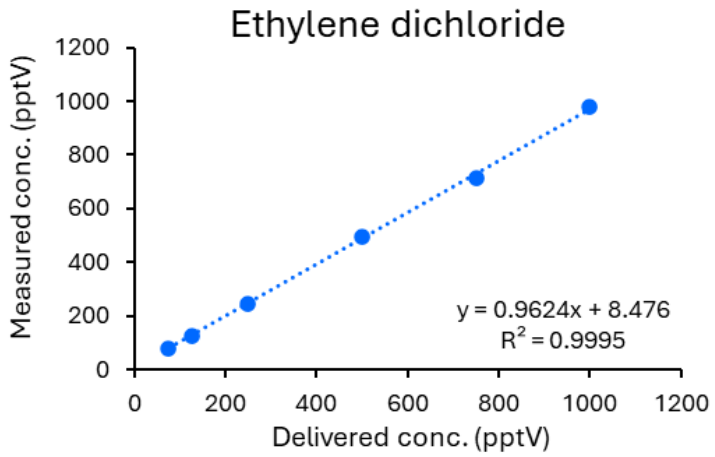
Compound	MDL (pptV)	HON Action Level (pptV)
Vinyl Chloride	660	1170
1,3-Butadiene	60	1360
Benzene	85	2820
Ethylene Oxide	102[+]	110
Ethylene Dichloride	260	990

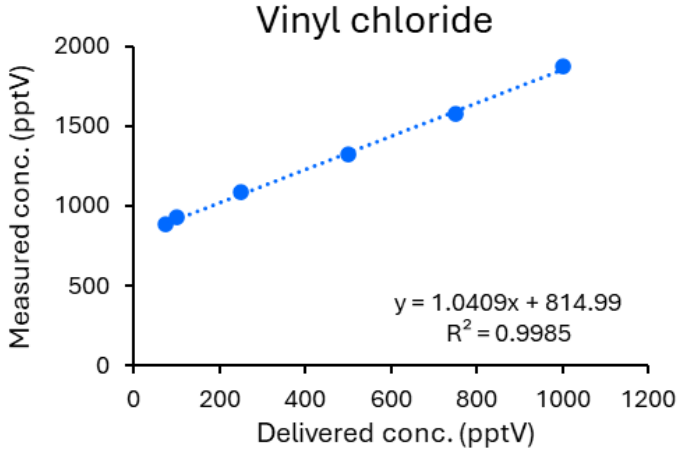
[+] Ethylene oxide measured over 30 minutes in the presence of 2.5 ppbV acetaldehyde

Other HON-regulated VOCs









Timeline for ASTM Method Testing

1. In-house repeatability – Germany, New Zealand, England
2. Other USA sites – mobile lab + customers
3. Other European sites – England, Czech Republic
4. South Korea – government agency mobile labs to be tested at Syft HQ

Summary/Take Aways

- Required MDL of 110 pptV is possible with stationary measurements and a longer sampling time.
- Mid pptV MDLs can be expected in a mobile setting, where acetaldehyde is present and subtracted from the ambient matrix.
- SIFT-MS has unique characteristics that make it well suited to air quality measurement. Most notably the selectivity that comes from multiple rapidly switchable reagent ions.
- Syft instruments have a track record of robust performance in mobile and stationary settings

Acknowledgements

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Labs participating in the ASTM Method Evaluation

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Thank you

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