Ambient Sample Transport of Ethylene Oxide

Background

In a 2018 update to the National Air Toxics Assessment (NATA), the US EPA significantly increased its risk value for ethylene oxide (EO). EO is commonly used in the chemical industry, particularly as a sterilant for medical products and as a reaction intermediate in the production of ethylene glycol. In response to NATA, industry and state regulatory agencies have expressed an interest in monitoring ambient air quality in and around these sterilization and chemical manufacturing facilities.

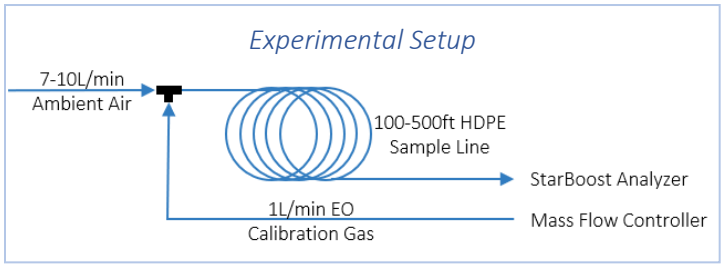
Problem

Collecting a stable sample of ethylene oxide for laboratory analysis may be challenging due to its reactivity, particularly with acids. Scrubber systems at sterilization facilities may utilize aqueous acids to convert ethylene oxide to ethylene glycol, and if residual acid mists are present in the sample canister or bag, losses can be expected. Ethylene oxide is also difficult to trap and concentrate due to its low boiling point. Because of these issues with sample stability, an analytical technology optimized for **real-time direct measurement of ethylene oxide** is necessary.

Solution

The MAX-iAQ is an ambient air monitoring system capable of sequentially analyzing up to 20 sample lines for ethylene oxide. The comprehensive design provides complete control of sample streams via an integrated multiplexer that maintains continuous sample gas flow on each channel. The core of this system is Max Analytical's novel FTIR enhancement technology, called StarBoost™, that dramatically increases the sensitivity, linearity and dynamic range of the FTIR over narrow spectral bands of interest. The SNR improvements provide a **limit of detection (LOD) as low as 5 ppb for ethylene oxide in real time**. In addition, the analyzer never requires calibration and utilizes a Peltier-cooled MCT detector that allows for continuous operation. The MAX-iAQ can be installed centrally at a plant or along a fence line, with up to 20 sample lines terminating at various locations.

Results



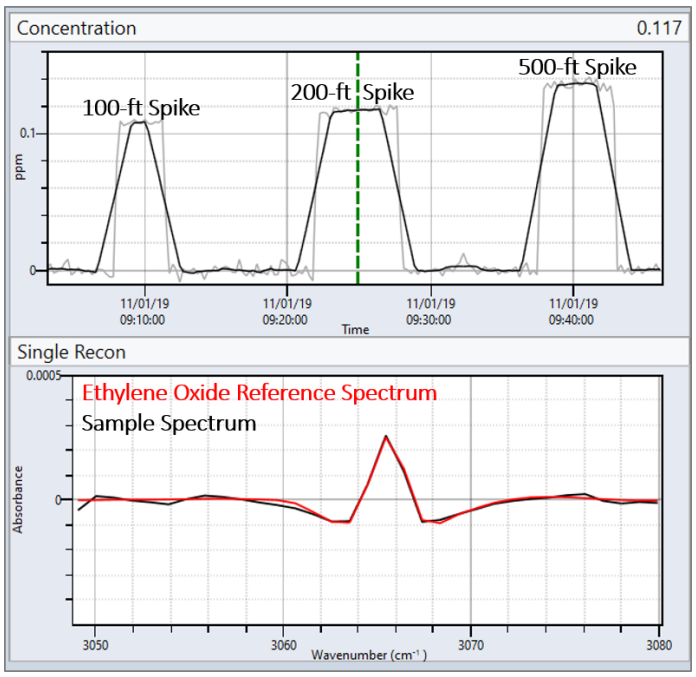
To demonstrate the effective transport of a low-level reactive gas through ambient sample lines, ethylene oxide spike recovery testing was performed using various lengths of tubing. A sample stream of ambient air was pushed using a diaphragm pump through 100-500ft of HDPE tubing to the inlet of the StarBoost™ FTIR. A certified calibration cylinder containing 2ppm EO and 500ppm ethane (tracer gas) was injected at 1L/min into the sample stream at the terminus of the HDPE sample line, with a dilution factor of 10-15% of the total sample flow. A diagram of this experimental setup is shown in the figure above.

Prior to spiking, the EO cylinder was measured directly to demonstrate the instrument was in calibration, and the EO response was within 1.5% of the certified cylinder value.

Ethylene Oxide Spike Recovery			
Dilution Factor	100-ft	200-ft	500-ft
Ethane Sample Average (ppm)	0.052	0.008	0.015
Ethane Spike Average (ppm)	24.990	27.016	31.592
Dilution Factor	0.050	0.054	0.063
Ethylene Oxide Spike Recovery			
EO Sample Average (ppm)	-0.002	0.000	0.000
EO Spike Average (ppm)	0.108	0.117	0.136
Predicted EO Spike Conc (ppm)	0.111	0.122	0.143
Percent Recovery (%)	96.30%	94.85%	94.52%

For all three ethylene oxide spikes, the percent recovery was greater than 94%, well within the acceptable tolerance of 70-130% specified by US EPA Method 320. This demonstrates adequate transport of EO through the sampling system and the accuracy of the EO measurement. While the recovery declined slightly as the length of tubing increased, 94.5% of the ethylene oxide was transmitted through 500-ft of HDPE. This high percent recovery suggests that longer lengths of sample line could be used.

The flow of calibration gas remained constant at 1L/min for all 3 sample line lengths, but the flow of ambient air sample varied due to flow restrictions at longer lengths of tubing. At 100-ft, sample flow was approximately 10L/min, and at 500-ft, flow was reduced to 7L/min. For this reason, the dilution factors and predicted EO spike concentration increase with increasing sample line length. A concentration plot of the spike recovery test is shown below to illustrate the precision of the measurement at low ppb levels. Scans were collected every 15 seconds, shown with an 8-scan (2 min) rolling average.



Below the concentration plot, a single reconstruction of the ethylene oxide spectrum is displayed at the time point associated with the green hashed line. The single reconstruction is the raw sample absorbance spectrum with all compounds in the analysis method subtracted, except the analyte of interest. This reconstruction matches the EO reference spectrum exceedingly well, demonstrating specificity for EO.

Conclusions

Ethylene oxide recoveries of 94-96% were achieved when spiking concentrations of 111-143ppb through 100-500ft of unheated HDPE tubing, demonstrating adequate transport of EO through the sampling system and the accuracy of the EO measurement. These results also indicate that the length of sample line could be increased beyond 500ft while still achieving an acceptable recovery. Max Analytical will continue to perform experiments to further optimize the sample transport of EO, prevent condensation of moisture in humid environments, and determine the maximum length of sample line.