Ethylene Oxide Monitoring with StarBoost™ Technology

Background

Ethylene oxide (EtO) is a carcinogenic and mutagenic compound commonly used in the chemical industry, particularly as a sterilant for medical products and hospitals and as a chemical intermediate in the manufacture of ethylene glycol. Following a recent assessment, the EPA has updated its risk estimate for ethylene oxide and expressed a need for monitoring very low levels of EtO, < 50 parts-per-trillion (ppt), in and around these chemical and medical diagnostic manufacturers.

Problem

The molecular weight of ethylene oxide is identical to CO_2, but CO_2 is present in ambient air at a level that is 8 million times higher than the level of EtO to be monitored, 400ppm versus 50ppt. Ethylene oxide is also highly reactive and difficult to trap and concentrate due to its low boiling point. These chemical and physical traits make it exceedingly difficult to detect by mass spectrometry. Few analytical technologies can directly measure any compound present in ambient air at 50 ppt. To achieve this low detection limit requires some sample concentration methodology followed by an analytical method optimized for EtO detection.

Solution

Max Analytical has developed a novel FTIR enhancement, called StarBoost™ technology, that dramatically increases the sensitivity, linearity and dynamic range over narrow spectral bands of interest. The SNR improvements provide minimum detection limits (MDLs) that are 10 to 20 lower than the commercially available FTIR gas analyzers without having to use extremely long pathlength gas cells. **Utilizing this optimization technology allows for single digit ppb ethylene oxide MDLs in real-time.**

To access even lower detection limits in the ppt range, ethylene oxide in ambient air can be concentrated onto a thermal desorption tube (TDT) and desorbed in the StarBoost™ FTIR gas cell. Max Analytical Technologies has developed a novel thermal desorption tube (TDT) concentration method in which TDTs are enclosed within a Peltier cooler during sampling. The Peltier cooler, or "tube chiller," is designed to maintain the TDT at a temperature near freezing to improve the trapping efficiency of volatile compounds, like EtO, on the sorbent material.

Results

To illustrate the viability of StarBoost™ technology for EtO detection, a certified EtO cylinder (49.85ppm) was diluted to 100ppb, 50ppb and 10ppb in ambient air and analyzed in real-time (1-min averages) using an FTIR equipped with StarBoost™. Spike recoveries for each of the three levels in ambient air are shown in Table 1, below.

Table 1 - Ethylene Oxide Spike Recoveries in Ambient Air

Ambient Air Flow (sccm)	**Flow from EtO Cylinder** (sccm)	**EtO Concentration in Stream, Calculated** (ppb)	**EtO Concentration in Stream, Measured** (ppb)	**Percent Recovery**
500	1.00	98.7	93.2	94.4%
1000	1.00	49.3	42.6	86.4%
5000	1.00	9.77	7.2	73.7%

This experiment demonstrates the ability to concentrate and quantitatively analyze for EtO at low ppb levels in ambient air. For all three spike levels, recoveries were greater than 70%. For 1-minute scans, the standard deviation in the EtO measurement was ~1ppb and the detection limit was ~3ppb.

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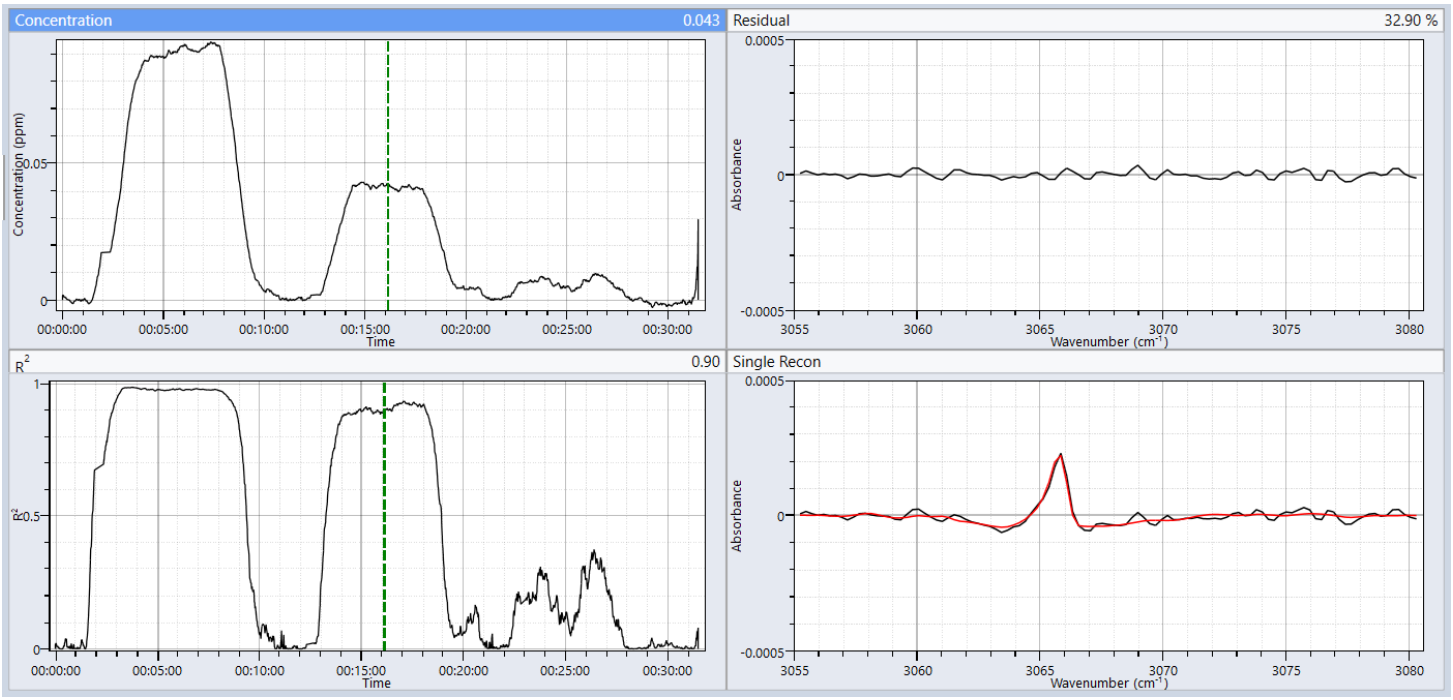
For a detailed view of the analysis, refer to Figure 1 below. The concentration plot for EtO is displayed in the top left panel. At bottom left is a plot of the R^2 analysis, or how well the model fits the data. The green hashed line indicates the selection of a scan during the 50ppb EtO spike. For this selected spectrum, the top right panel displays the IR residual and the bottom right panel shows the single reconstruction of EtO, with all other gases in the method subtracted. The reference spectrum of EtO (red) is overlaid on the sample spectrum (black). These results demonstrate single-digit ppb sensitivity for EtO in ambient air using an FTIR with StarBoost™ technology. Even at 50ppb, the EtO peak is clearly visible in the single reconstruction, and the reference spectrum matches the sample, indicating a good fit quality.



Conclusions

These data show that a FTIR with StarBoost™ can measure single digit ppb levels of ethylene oxide in ambient air. The ease of sampling and data collection allow for efficient workflow, and results can be achieved in real-time. Additionally, the increased precision compared to traditional FTIR assures the client or regulator that the result truly reflects the EtO present in the source stream. For even lower detection limits, samples can be concentrated onto TDTs using a Peltier cooler and immediately desorbed into the gas cell. This would provide a near real-time solution with sensitivity in the low ppt range.

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