Ethylene Oxide Process Monitoring

On line process mass spectrometry is a critical element in the efficient operation of modern ethylene oxide (EO) production processes. Fast and accurate analysis of feedstock and effluent streams are needed to maximize conversion efficiency, catalyst selectivity ratios and carbon balances. Monitoring these parameters has a direct impact on bottom line performance.

In addition to the ethylene and oxygen reagents, EO processes streams commonly include either nitrogen or methane as balance gases. A mixture of oxygen, ethylene, the balance gas and recycle gases are supplied to a heated multi-tubular catalytic reactor. The reactor effluent is cooled and scrubbed to extract EO as an aqueous solution, and CO2 is removed before the waste gases are recycled.

A Monitor Instruments IND 3000 process mass spectrometer, equipped with a molecular leak inlet system[1], was installed in an EO pilot test facility as a reactor characterization, diagnostics, and process monitoring tool. A quantitative method was created [2] using Gas-Wizard™ software to monitor the eight compounds of interest in EO process streams: methane (using indicator mass 15 amu), ethylene (27amu), nitrogen (28 amu), ethylene oxide (29 amu), ethane (30 amu), oxygen (32 amu), argon (40 amu) and carbon dioxide (44 amu). The matrix inversion capability in Gas-Wizard was used to deconvolute interferences in the eight component matrix. Six fragmentation binary mixtures and two sensitivity mixtures were used to calibrate the system.

Prior to installation, IND 3000 performance was evaluated with a standard gas mixture containing four key components in the EO process (ethylene, nitrogen, ethylene oxide, and carbon dioxide). Eight hours of ethylene and EO data, shown in Figure 1, demonstrate excellent long term stability. The linearity of the oxygen response from 4% to 21% in nitrogen was also measured. Calibration at 5% with subsequent challenge from 4% to 21% produced a quantitative recovery within 0.07%. This is well within the certified accuracy of the standards.

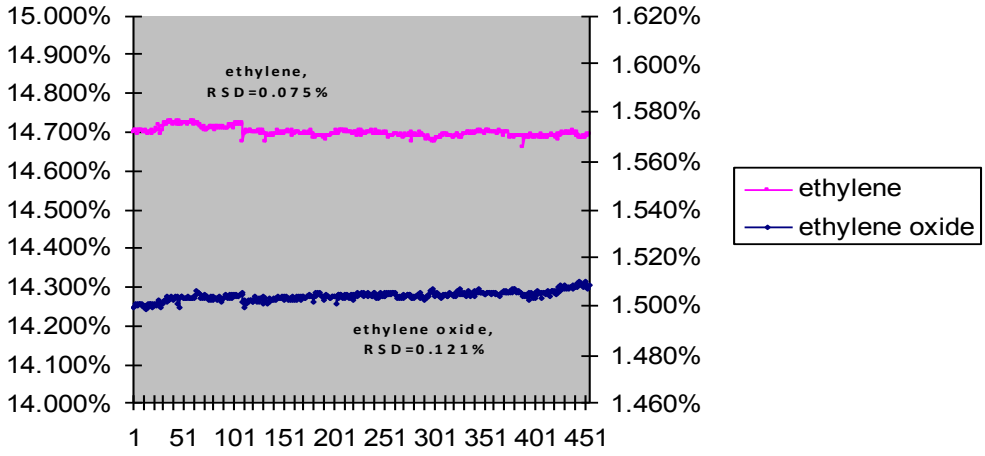

Figure 1. Eight hour analytical stability test using a certified EO gas standard containing 14.7% ethylene and 1.5% EO.

[1] See Monitor Instruments Application Note: Precision Inlet Systems
[2] See Monitor Instruments Application Note: Quantitative Method Development

The EO process pilot plant was equipped to run both N_2 and CH_4 balance processes. While it is uncommon to switch between these two extremes in a production plant, it is desirable in a development facility, even though so doing presents a challenge to the overall dynamic range, precision, and accuracy of a process analyzer. The data in Table 1 compare process stream measurements made by the Monitor IND 3000 with data from an older and more expensive legacy mass spectrometer.

	Balance/ Stream Indicator>	**CH_4** 15amu	**C_2H_4** 27 amu	**N_2** 28 amu	**EO** 29 amu	**C_2H_6** 30 amu	**O_2** 32 amu	**Ar** 40 amu	**CO_2** 44 amu
3000I	N_2/feed	0.03	15.10	69.97	0.00	0.016	7.10	0.044	7.75
Legacy	N_2/feed	0.16	15.19	69.59	0.02	0.00	7.10	0.09	7.86
3000I	N_2/exit	0.07	13.49	70.85	1.51	0.031	5.47	0.046	8.53
Legacy	N_2/exit	0.16	13.46	70.17	1.52	0.00	5.45	0.10	8.53
3000I	CH_4/feed	57.93	24.91	1.06	0.19	0.277	7.60	0.144	7.90
Legacy	CH_4/feed	58.08	24.36	1.15	0.23	0.24	7.41	0.21	8.32
3000I	CH_4/exit	58.92	23.03	1.04	2.13	0.299	5.47	0.150	8.97
Legacy	CH_4/exit	58.65	22.28	1.14	2.09	0.29	5.28	0.21	9.23

Table 1. Summary of typical performance data. Note that a small amount of EO was added to the CH_4 balance feed stock when these data were run in an experimental process.

These data are routinely used to calculate parameters that characterize reactor efficiency, such as oxygen and carbon balances, and catalyst conversion selectivity. Another common parameter, "spread," quantifies the quality and consistency of the analytical data. The lower the spread, the better the data fit the overall reactor process stream mass balance. In the nitrogen balance system data shown above the carbon and oxygen mass balances are 1.01 and the spread is 1.61% for the Monitor Series 3000I. The Legacy system data gave 0.98 oxygen and carbon mass balances along with a 2.30% spread.

Monitor Instruments' Series 3000 cycloidal mass spectrometers provide process analysis in a wide variety of industries. Our application specific inlet systems, versatile Gas-Wizard™ software, and stable analyzers assure cost effective, high quality process control information. We invite you to visit our website (www.monitorinstruments.com), to request information via e-mail at info@monitorinstruments.com, or post at 290 East Union Rd., Cheswick, PA 15024, USA, or to contact us by telephone at +1.724.265.1212 or fax at +1.274.265.1199. We will give your application the careful, confidential consideration it deserves.

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