

High Performance EO Analysis by MS Plant Trial Data

A Monitor cycloid mass spectrometer (MS) was run in parallel with existing analytical tools in an ethylene oxide production facility. The chemistry of this catalytic reaction is relatively simple with two reactants, ethylene and oxygen, and two major products, ethylene oxide and carbon dioxide. Ethylene oxide is the desired product, whereas carbon dioxide is an undesirable side reaction product consuming valuable raw material. Therefore, the goal is to minimize carbon dioxide formation while maximizing ethylene oxide production. The existing plant analytical instrumentation included multiple paramagnetic oxygen analyzers (inlet & outlet) on each reactor, IR instrumentation for inlet ethylene feed control, and process GC to measure all components (with correction of Ar for O₂ using the O₂ analyzer data) with a 15 minute cycle time. The plant instrumentation was within their normal calibration parameters. The Monitor MS was not calibrated on-site: a calibration established at our factory several months prior was found to be precise and accurate.

The carbon and oxygen balances shown in Figures 1 & 2 are good indicators of the quality of analysis for the respective analytical data. These data were acquired after a 6 hour warm-up period for the mass spectrometer, although meaningful data were seen after just 2 hours' operation. The Monitor MS performed better than the combined plant analytical data in all cases. There are several reasons for this; the plant data depends upon several different analyzers, the relative error between each analyzer, and the timing mismatches in attempting to correlate the data. In this respect, the mass spectral data is more coherent, all data elements are from a single analyzer in the same time domain with a single calibration process. This is extremely important when using the data for process understanding, since the fine detail of process trends can be appreciated for their full value.

The following Table shows a comparison of the quality of data achieved with the Monitor process MS as compared with the plant analytical tools used in this trial. This table is an average over the 16 hour comparison period, however as stated above, Monitor MS data is much more revealing with regard to the rapidly changing process trends.

Parameter	Monitor MS	Plant Analytical
O ₂ balance	98.8	94.2
C balance	100.8	96.3
delta EO	1.98	1.96
delta O ₂	2.73	2.79
delta C ₂ H ₄	2.53	2.61
delta CO ₂	1.14	1.10

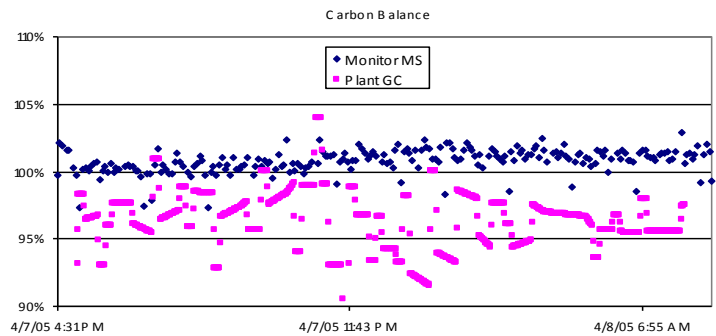


Figure 1

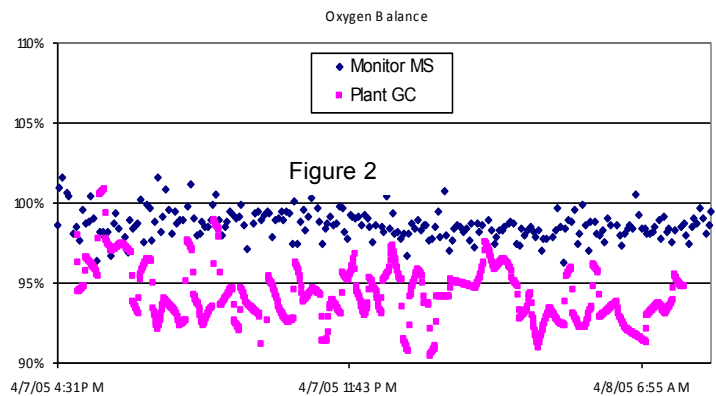
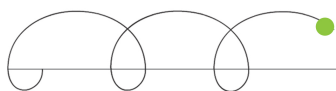


Figure 2



Fast, accurate MS data provide real-time process control information. For example, the data in Figures 3 and 4 illustrate the reactant consumption in one case (Fig. 3) and the product formation in the other (Fig. 4). These are delta plots, that is the inlet minus outlet for the reactant plot, and the outlet minus inlet for the product plot. When the reaction is profiled in this fashion we can see nearly perfect coherence in the reactant plot where the consumption of both oxygen and ethylene track each other very closely. However, that is not the case for the product formation trend plots. Clearly, at around midnight the reaction efficiency for ethylene oxide formation improved measurably relative to the undesirable carbon dioxide side reaction product. The improvement is on the order of 500 ppm. The data points to a window of opportunity for improved process control with better understanding of the cause of the reaction dynamic in this case.

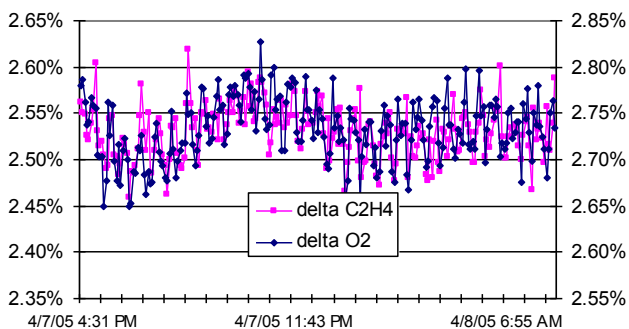


Figure 3

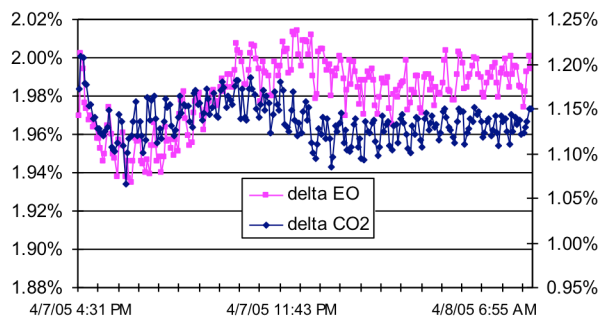


Figure 4

This effect could easily be explained as a precipitous change in catalyst selectivity. However, other analytical data are available for stream components. For example, the absolute value of inlet ethylene concentration in Figure 5 shows a variation of around 1% even though delta ethylene varies only 0.25% min. to max. throughout the 16 hour period. Both delta product and inlet ethylene trend plots show a clear perturbation at 11:40pm. Since the inlet oxygen concentration varies around 0.25%, and delta oxygen by less than that, reaction stoichiometry cannot be ruled out as the underlying cause for the observed change in ethylene oxide production efficiency observed here.

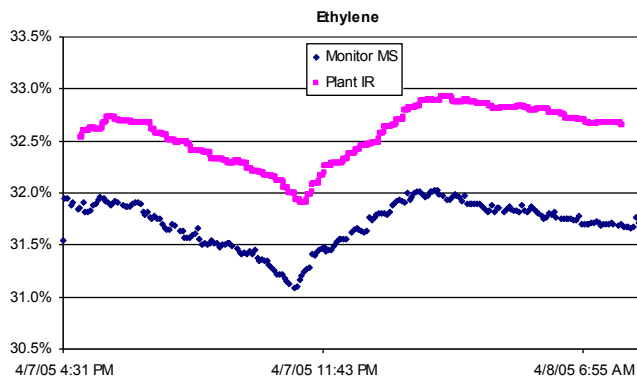


Figure 5

Monitor's IND3000 process mass spectrometry data reveals rapidly changing trends in the process. The fact that high quality MS data is generated so quickly after startup, and without re-calibration, is further supporting evidence that Monitor can supply fast, durable and yet cost effective process analyzers.

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), or to request information via e-mail at info@monitorinstruments.com, or post at 290 East Union Rd., Cheswick, PA 15024, USA. You could also 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.

