Analysis of Liquefied Ethane Product Using the Agilent Process Micro Gas Chromatograph

Application Note

Micro Gas Chromatography, Petrochemical Analysis, Hydrocarbon Analysis

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Abstract

This application note describes the use of the Agilent 490-PRO Micro GC for the analysis of liquefied ethane product. The ethane stream analysis involves the detection of nitrogen, methane, carbon dioxide, ethane, and propane, with trace detection of hydrogen sulfide, and methanol. The 490-PRO micro GC uses a single column channel to analyze all components of interest in 180 seconds. Results are calculated by the on-board data handling and calculation module and delivered to plant processing and pipeline operating systems.

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Introduction

Ethane product in Alberta, Canada is collected from gas processing facilities in the province and delivered to two petrochemical facilities, Dow Chemical in Ft. Saskatchewan and Nova at Joffre. Delivery capacity of the pipeline system is 320,000 barrels per day, with a pipeline length of more than 1,300 km. Pipeline specification calls for a limit of 60 parts per million (ppm) of hydrogen sulfide and 1,000 ppm of methanol. Hydrogen sulfide is highly corrosive, which demands its limitation. The methanol limit exists to protect catalysts at the receiving facilities.

The 490 Micro GC is designed for fast and accurate analysis for a wide range of gas samples. The system can hold up to four independently controlled chromatographic channels. Each channel is a complete miniaturized gas chromatograph and includes a MEMS-based injector and thermal conductivity detector and narrow-bore analytical columns. This setup results in results in seconds, rather than minutes, for faster and better informed decision making.

Instrument setup and sample information

A 490-PRO Micro GC, equipped with a 10-m PoraPLOT Q column channel, was used for the analysis of all compounds of interest in an ethane stream. Table 1 gives an overview of the instrument settings.

The 490-PRO Micro GC, designed to be a system component, does not require a connection with a chromatography data system to generate data and results. It features on-board data collection, peak integration, result calculation and generation. The powerful PROstation set-up tool was used to program these method parameters in the 490-PRO Micro GC. After setup, PROstation was disconnected and user defined analysis results and information was automatically passed to an external system in an unattended manner.

Three different sample streams, two product streams and one calibration mixture, were coupled to the 490-PRO Micro GC. This setup features scheduled and fully automated calibration verification or, when required, instrument recalibration.

The sampling system, designed by QC LAB Inc., includes sample vaporizers and multiple filtration steps (coalescer and genie membrane). QC LAB Inc. has been proving the Agilent Micro GC instrumentation and services to the gas processing industry in Canada for over 20 years now. The vaporizer expands the application range of the Micro GC the liquefied gas streams (LNG and LPG). In addition, the vaporizer ensures the sample pressure does not exceed the Micro GC inlet maximum of one bar overpressure. The coalescer and genie membrane filter removes water droplets and particles from the sample stream before entering the 490 Micro GC. Figure 1 shows a photo of the filter setup.

Table 1.   Micro GC Instrument Settings

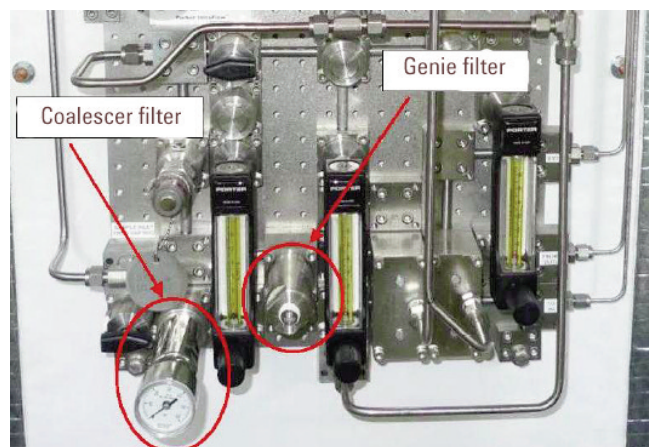|	PoraPLOT Q, 10 m
Column temperature	80 °C
Carrier gas	helium, 15 psi (~ 103 kPa)
Injection time	80 milliseconds
Detector sensitivity	auto ranging
Sample line temperature	60 °C
Sampling time	20 seconds

Figure 1.   Sampling system with coalescer and genie membrane filter.

Results and Discussion

Single run for ethane composition and impurity analysis

The 490 Micro GC, equipped with a single 10-m PoraPLOT Q column channel, analyzed ethane composition and its low level impurities in a single run. Detection of trace components to ppm levels was possible through the use of the auto ranging feature of the micro thermal conductivity detector (µTCD). This functionality enabled automatic switching from ultra-high sensitivity (low ppm range) to normal settings (percentage levels).
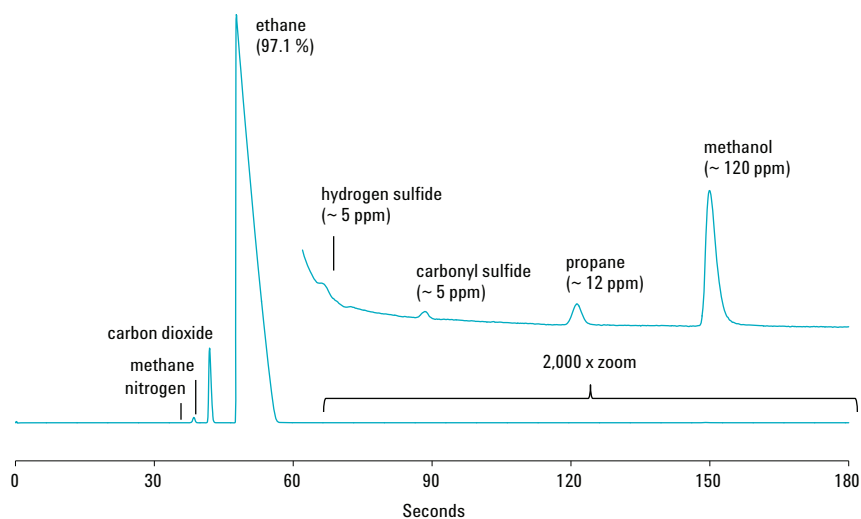

Figure 2.   Chromatogram for ethane on a 10-m PoraPLOT Q column channel, showing baseline separation for all compounds of interest.

For the characterization of the ethane product, nitrogen, methane, carbon dioxide, ethane, and propane is analyzed. Impurity analysis includes hydrogen sulfide and methanol characterization. Chromatograms of an ethane sample stream are displayed in Figure 2 and Figure 3 (zoom) and shows baseline separation for all compounds of interest.
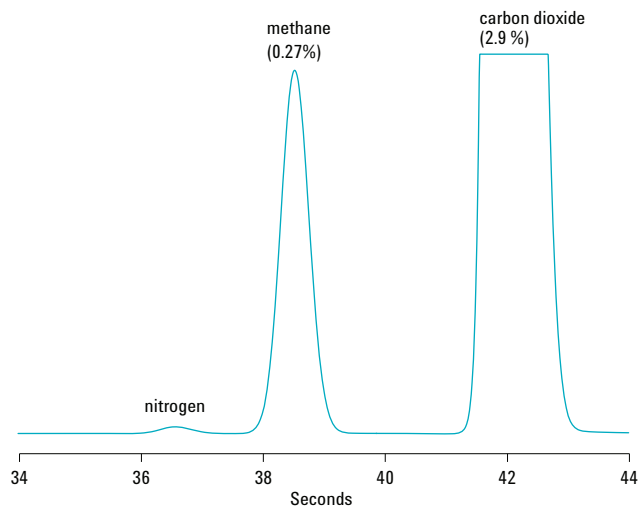

Figure 3.   Chromatogram (zoom) for nitrogen and methane separation.

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The product gas contains more than 95% ethane, hydrogen sulfide elutes close after ethane on a PoraPLOT Q column. As a result, hydrogen sulfide elutes on the tailing of the bulk peak. However, Figures 4 and 5 clearly show that low ppm levels of hydrogen sulfide can be quantified.

With micro gas chromatography technology, very fast analysis times were achieved. The total analysis time of this application was just 180 seconds; this provided near real-time data transfer to the plant process control system for faster trend analysis and instant process optimization.

In addition to lower investment costs, the single column setup has another important benefit. Compared to a regular gas chromatograph, a Micro GC typically consumes a factor of 10 to 20 less carrier gas. With the single column channel setup, this application uses even less carrier gas; approximately 4 mL/min of helium. One bottle lasts for approximately 3 years in continuous operation.

The 490-PRO Micro GC provides outstanding accuracy and longevity, requiring no maintenance over long periods of time. Two 490-PRO Micro GCs, installed by QC LAB in 1994, are still functioning and providing over 450 analyses per day, without any repair or loss of service.
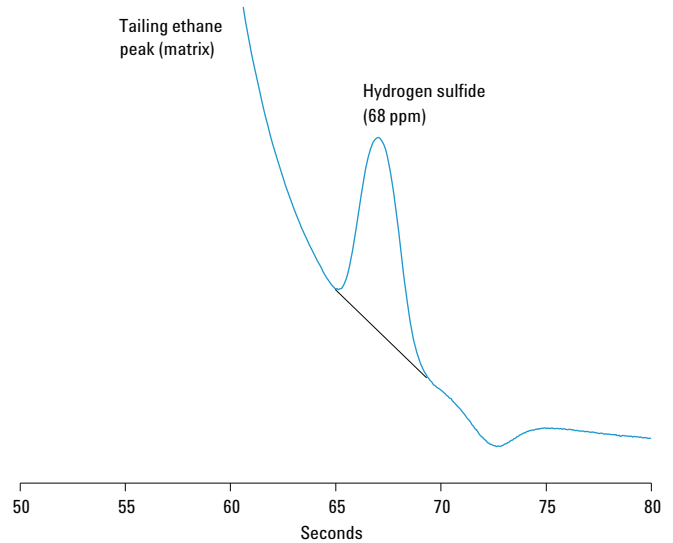

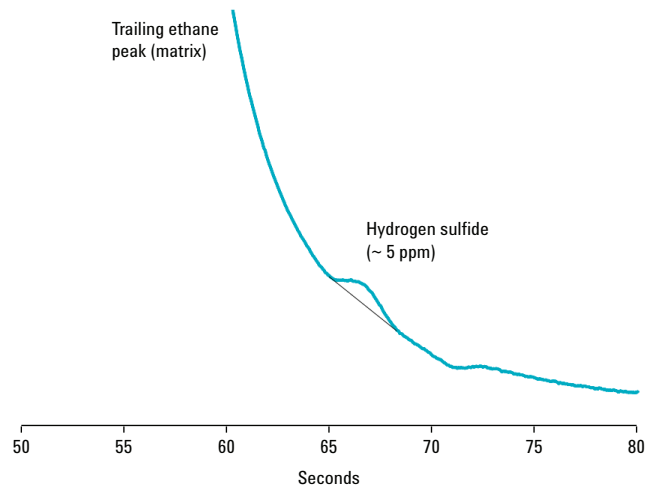Figure 4.   Chromatogram for hydrogen sulfide at 68 ppm level.

Figure 5.   Chromatogram for hydrogen sulfide at 5 ppm level.

Ethane product characterization

QC LAB uses 10 consecutive calibration runs to calibrate a GC. The results are then averaged and a response factor is determined. Table 2 shows the results of 10 consecutive hydrocarbon calibration runs and the calculated statistics. Excellent short term accuracy was obtained; this usually indicates long term reliability.

Table 2.   Ethane Product Calibration Results and Statistics

	nitrogen	methane	carbon dioxide	ethane	propane
run 1	0.1027%	2.0187%	1.3079%	96.2683%	0.3024%
run 2	0.1021%	2.0189%	1.3079%	96.2681%	0.3031%
run 3	0.1017%	2.0191%	1.3079%	96.2695%	0.3019%
run 4	0.1013%	2.0190%	1.3081%	96.2701%	0.3015%
run 5	0.1009%	2.0190%	1.3080%	96.2705%	0.3017%
run 6	0.1005%	2.0190%	1.3080%	96.2710%	0.3016%
run 7	0.1004%	2.0189%	1.3081%	96.2711%	0.3015%
run 8	0.1001%	2.0191%	1.3080%	96.2705%	0.3023%
run 9	0.1003%	2.0192%	1.3082%	96.2705%	0.3018%
run 10	0.1002%	2.0191%	1.3080%	96.2704%	0.3023%
average	**0.101%**	**2.019%**	**1.308%**	**96.270%**	**0.302%**
accuracy	**± 0.0013%**	**± 0.0002%**	**± 0.0002%**	**± 0.0015%**	**± 0.0008%**

Hydrogen sulfide and methanol impurities

In addition to the characterization of the ethane product itself, low level hydrogen sulfide and methanol impurities were monitored as well. Table 3 displays the calculated average and accuracy for 10 consecutive runs for calibration.

The hydrogen sulfide specifications are limited to 60 parts per million (ppm) as it is highly corrosive and can damage, the more than 1,300 km long, pipeline from the production site to the final processing plant. The methanol limit of a maximum 1,000 ppm exists to protect catalysts used at the receiving facilities. Figure 6 shows an example of a 20-hours trend analysis for both compounds.

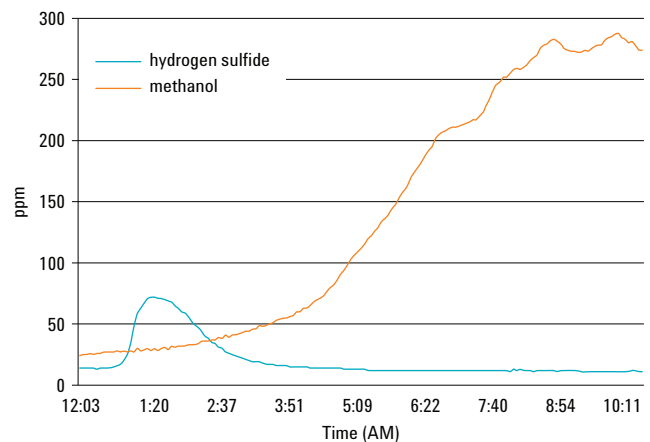

Figure 6.   Example of a 20-hours trend analysis of hydrogen sulfide and methanol impurities in the ethane product.

Table 3.   Hydrogen Sulfide and Methanol Statistics (n = 10)

	hydrogen sulfide	methanol
average	52.2 ppm	500 ppm
accuracy	± 1 ppm	± 13 ppm

Conclusion

This application note clearly shows that the Agilent 490-PRO Micro GC, equipped with a 10-m PLOT Q column, is capable of analyzing ethane streams in a process environment. The single channel setup and low carrier gas consumption of the 490-Micro GC keeps your investment and operating costs under control.

The MEMS-based injector and detector of the Micro GC, in combination with short and narrow-bore analytical columns, results in very fast analysis times. Getting results in seconds rather than minutes leads to more accurate trend analysis and faster process control.

The 490-PRO Micro GC brings lab quality instrument to on-line/at-line applications. Build-in on-board data handling and result generation controls the complete operation of the instrument. It runs standalone, and does not require a local operator. All required results and information are automatically passed to external systems for optimal process control.

For More Information

These data represent typical results. For more information on our products and services, visit our Web site at www.agilent.com/chem.

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