Inert Sample Path for the Agilent 490 Micro GC - Analysis of Low ppm Levels of Hydrogen Sulfide and Carbonyl Sulfide

Application Note

Micro Gas Chromatography, Sulfur Analysis

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Abstract

To prevent adsorption of sulfur compounds, deactivation of the stainless steel surface of the sample path is required for the analysis of low ppm levels.
This application note shows the separation, low level detection, and analysis performance of hydrogen sulfide and carbonyl sulfide using the Agilent 490 Micro GC in combination with a fully deactivated sample inlet.

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Introduction

Low ppm level analysis

Trace gas analysis is a challenge in today's world. The ability to analyze lower component levels permits better quality control, and gives more reliable results. Because you require fast and accurate gas analysis results, we made product quality improvements resulting in lower limits of detection for our gas analysis platform, the Agilent 490 Micro GC [1].

An example of this improvement is the use of UltiMetal deactivation of stainless steel surfaces. For low ppm level analysis of sulfur compounds, these stainless steel surfaces need to be deactivated to prevent adsorption.

Experimental

To match your gas application requirements, you can equip the 490 Micro GC with one to four independently controlled column channels. Each column channel is a complete, miniaturized GC with:

- Electronic carrier gas control
- Micro-machined injector
- Narrow-bore analytical column
- Micro thermal conductivity detector (µTCD).

This setup provides fast gas analysis, with typical run times of 30 to 90 seconds.

For this experiment, an Agilent 490 Micro GC (p/n G3581A) equipped with a J&W PoraPLOT U column channel was used. Micro GC systems are easy to operate, just a handful of settings are required to complete the run method. Table 1 gives an overview of the settings used for this study.

Table 1. Just a Few Settings Completes the Run Method

Agilent 490 Micro GC	
Column	Agilent J&W PoraPLOT U (10 m)
Injector temperature	50 °C
Column temperature	80 °C
Column pressure	150 kpa, helium
Injection time	200 ms
Sample line temperature	50 °C
Sampling time	10 seconds

Improved sample inlet design with UltiMetal surface deactivation

The system features an improved sample inlet design (Figure 1) with a fixed 1/16 inch heated sample connector at the rear of the instrument, or an optional unheated inlet on the front. The new sample inlet uses proprietary Agilent UltiMetal technology for metal surface deactivation of the sample flow path. The surface treatment is applied to the entire inlet, from the sample connector union to the tubing connected to the MEMS injector. Recent sample inlet enhancements include vacuum-brazing connections to minimize nut and ferrule connections, and features an o-ring free design for a leak-free sample path [2].

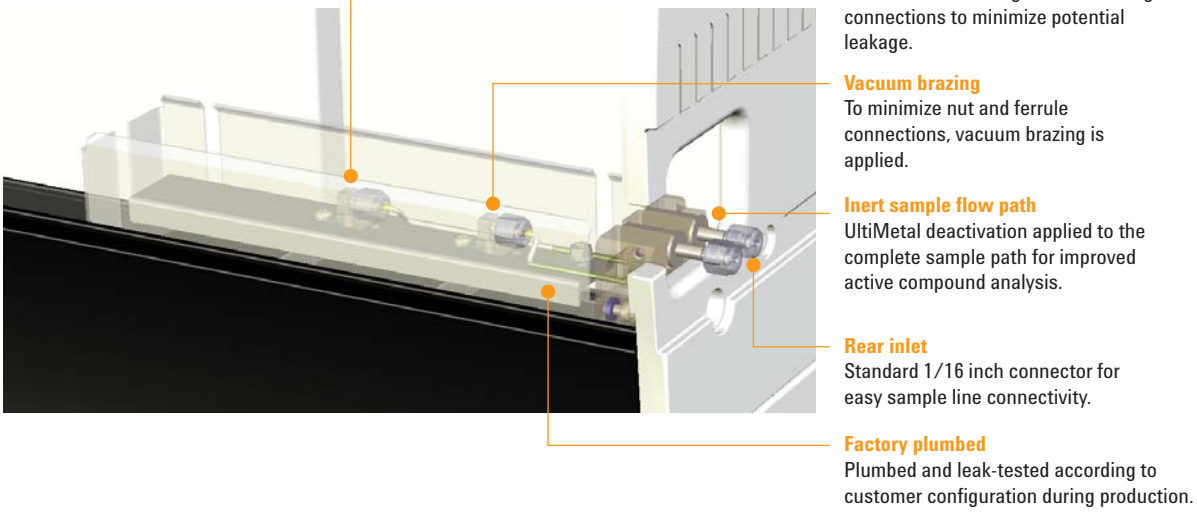

Figure 1. Agilent 490 Micro GC with improved sample inlet design.

Results and Discussion

Hydrogen sulfide and carbonyl sulfide separated from the biogas and natural gas matrix

To separate hydrogen sulfide and carbonyl sulfide from the matrix of energy-rich sample streams, such as biogas or natural gas, an Agilent J&W PoraPLOT U column is required. Figure 2 shows that these sulfur compounds elute between ethane and propane. The separation of hydrogen sulfide and carbonyl sulfide from the biogas or natural gas matrix compounds enables easy integration and peak identification, resulting in more reliable analyses.

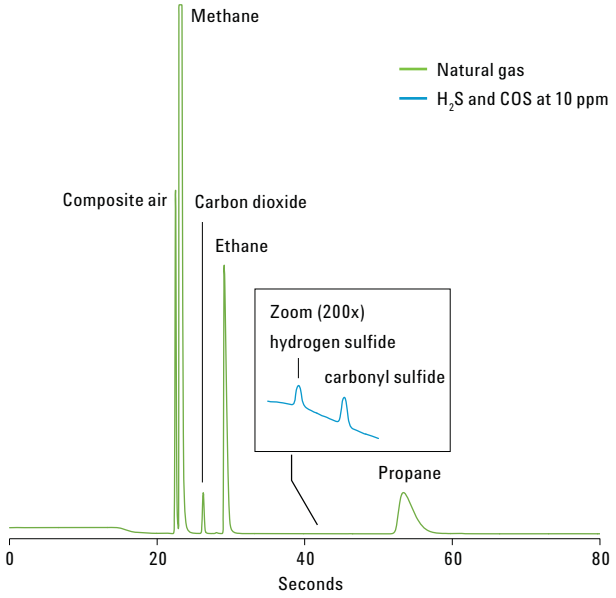

Figure 2. Separation of hydrogen sulfide and carbonyl from the other compounds in biogas and natural gas is done on the Agilent J&W PoraPLOT U column channel.

The Agilent Biogas and Natural Gas Analyzers (p/n G3582A) are equipped with the same PoraPLOT U column channel and sample path deactivation [3,4]. Therefore, results and conclusions shown in this application note are applicable to these preconfigured and factory-tuned instruments as well.

Sulfur compound detection down to 3 ppm

Figure 3 shows an example of 3.0 ppm hydrogen sulfide and 3.2 ppm carbonyl sulfide. Even though baseline noise is visible in these zoomed chromatograms, it clearly shows that the 490 Micro GC with an inert sample inlet is capable of analyzing down to low ppm levels of these sulfur compounds.

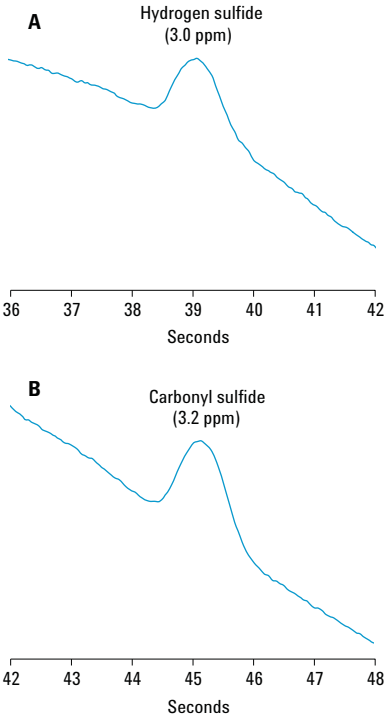

Figure 3. Analysis of 3 ppm level hydrogen sulfide (A) and carbonyl sulfide (B) on the Agilent 490 Micro GC with inert sample path.

Excellent repeatability

For reliable results, instrument repeatability and limit of detection are important concerns. To confirm the instrument's performance, repeatability is determined at three concentration levels (approximately 3, 5, and 10 ppm) for both concentration (ESTD) and retention time (RT).

Retention time repeatability over 10 consecutive runs was calculated between 0.03 to 0.08 %. Peak area repeatability, depicted as external standard concentration, was determined at 0.5 to 1.0 % RSD for the 10 ppm level, and 1.5 to 2.5 % RSD for the 3 ppm level. Table 2 gives an overview showing excellent performance for both concentration and retention time repeatability. Figure 4 depicts an overlay of five consecutive runs for hydrogen sulfide at the 10 ppm level.

Table 2. Excellent Repeatability is Determined for Both Concentration (ESTD) and Retention Time (RT)

Hydrogen sulfide			Carbonyl sulfide		
ESTD (ppm)	RSD % Concentration (ESTD)	RSD % (RT)	ESTD (ppm)	RSD % Concentration (ESTD)	RSD % (RT)
3.0	2.5 %	0.07 %	3.2	1.5 %	0.05 %
5.2	1.8 %	0.07 %	6.6	0.78 %	0.08 %
10.4	0.52 %	0.04 %	12.0	0.99 %	0.03 %

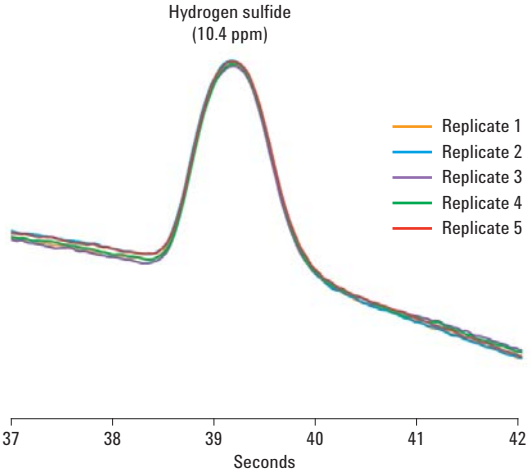

Figure 4. Overlay of five replicate runs for 10 ppm hydrogen sulfide.

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Conclusion

The Agilent 490 Micro GC's new sample inlet design, with Agilent proprietary UltiMetal treatment for stainless steel surface deactivation, provides accurate analysis of low ppm level sulfur compounds. Hydrogen sulfide and carbonyl sulfide can be analyzed down to a 3 ppm level with excellent concentration (1.5–2.5 % RSD) and retention time (0.03–0.08 % RSD) repeatability on the Agilent 490 Micro GC with Agilent J&W PoraPLOT U column channel.

As a result of its speed of analysis, the 490 Micro GC provides reliable results quickly for better and faster product and process quality control.

References

1. Fast and Reliable Trace Gas Analysis – Improved Detection Limits for the Agilent 490 Micro GC, *Agilent Technologies Technical note*, publication number 5991-6201EN (**2015**).
2. Agilent 490 Micro GC Solution, *Agilent Technologies Datasheet*, publication number 5991-6034EN (**2015**).
3. Analysis of Biogas using the Agilent 490 Micro GC Biogas Analyzer, *Agilent Technologies Application Note*, publication number 5990-9508EN (**2011**).
4. Fast Analysis of Natural Gas using the Agilent 490 Micro GC Natural Gas Analyzer, *Agilent Technologies Application Note*, publication number 5991-0275EN (**2012**).

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