Application Note

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Trace-Level Hydrogen Sulfide and Carbonyl Sulfide Analysis on the Agilent 990 Micro GC

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Abstract

This application note demonstrates how to analyze low-ppm level hydrogen sulfide (H_2S) and carbonyl sulfide (COS) on the Agilent 990 Micro GC system. The targeted analysis was completed on a 10 m Agilent J&W PoraPLOT U channel with backflush in less than 1 minute. Retention time and response repeatability were evaluated based on 30 consecutive analyses of 10 ppm H_2S and COS gas standard.

Introduction

Hydrogen sulfide (H_2S) is a deleterious gas, which is harmful to the environment. If existing in the hydrocarbon processing process, H_2S will poison the catalysts and severely reduce the down-stream product yield. Considering its impact on these industries, it is necessary to monitor H_2S emission and its abundance. Accurate and reliable hydrogen sulfide analysis achieved by a conventional thermal conductivity detector (TCD) is usually only possible at concentrations above 100 ppm. To measure H_2S at low-ppm and ppb levels, a chemiluminescence detector is often a good choice. However, the ownership cost and usability of chemiluminescence detectors is not comparable with TCDs. The 990 Micro GC system has the most sensitive TCD in the industry, which features a 200 nL detection cell for the detection of most gas components at low-ppm levels.

In this application note, a 990 Micro GC configured with a 10 m J&W PoraPLOT U channel with backflush was used for 10 ppm H_2S and COS analysis. The peak shape and signal-to-noise ratio (S/N) of targeted components were evaluated. The absolute response, retention time repeatability, and analysis speed were also demonstrated for fast H_2S and COS analysis.

Experimental

A 990 Micro GC was configured with a single 10 m J&W PoraPLOT U channel with backflush. The analytical parameters are shown in Table 1. The calibration sample was purchased from Air Liquide, Inc; its composition is shown in Table 2.

Table 1. Channel configuration of the Agilent 990 Micro GC system and the analytical parameters.

Channel Type	Injection Time (ms)	Column Temperature (°C)	Column Pressure (kPa)	Backflush Time (s)	Carrier Gas
10 m Agilent J&W PoraPLOT U with Backflush	50	80	150	9.0	He

Table 2. Calibration gas composition.

Compound Name	Concentration (mol/mol)
Hydrogen Sulfide	10.0 ppm
Carbonyl Sulfide	10.0 ppm
Nitrogen	Balance

Results and discussion

Figure 1 shows a chromatogram of the calibration standard. Figure 2 is a chromatogram with H_2S and COS detected in simulated natural gas. H_2S and COS are well separated on the PoraPLOT U channel. Propane is the last compound to elute on the PoraPLOT U channel and does so within 60 seconds. Peak shapes are excellent, with peak symmetry for H_2S and COS at 1.06 and 0.97, respectively. The retention time (RT) and area precision for 30 consecutive analyses of the calibration standard are shown in Table 3. The area repeatabilities for 10 ppm H_2S and COS are 6.5% and 3.7%. The RT relative standard deviations (RSD) are 0.035% and 0.018% for H_2S and COS, respectively. The results demonstrate good qualification reliability and quantitation precision. The S/N for the two compounds in Figure 1 is 14:1 and 18:1, respectively. Estimated from the S/N achieved with 10 ppm standard gas, the analysis of 2 to 3 ppm H_2S and COS is possible on the 10 m PoraPLOT U channel with the same method parameters. For higher response and area precision of a low-ppm H_2S and COS sample, an injection time of 150 to 200 ms is recommended.

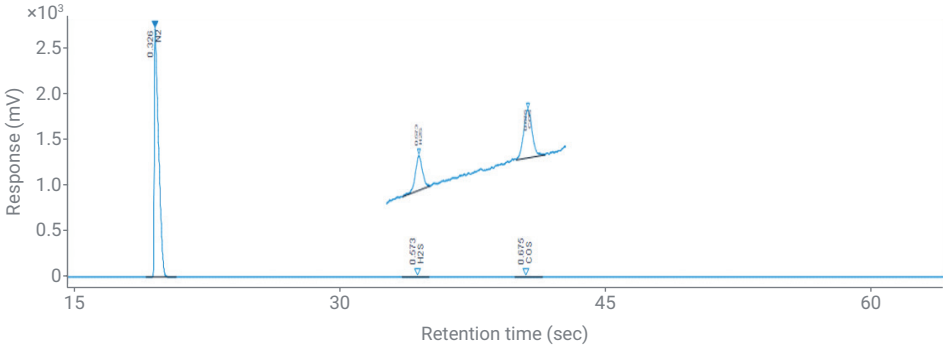

Figure 1. Chromatogram of 10 ppm H_2S and COS standard gas.

Conclusion

This application demonstrates the analysis of 10 ppm H_2S and COS on the Agilent 990 Micro GC system within 60 seconds. The 990 Micro GC delivered the H_2S analysis at ppm level with good retention time and peak response repeatability. The inert gas flow path, the high sensitivity of 990 microcell TCD, and the high precision of the thermal and pneumatic controls contributed to the excellent performance of the 990 Micro GC.

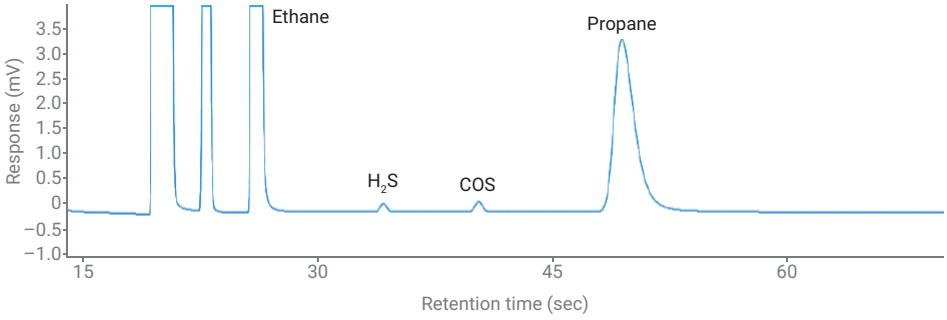

Figure 2. Chromatogram of H_2S and COS in natural gas.

Table 3. Area and retention time (RT) repeatability of 30 calibration gas analyses.

Compound	RT (min)	RT %RSD	Area (mV × s)	Height (mV)	Area %RSD	S/N
H_2S	0.574	0.035	0.006	0.014	6.42	14:1
COS	0.675	0.018	0.011	0.020	3.68	18:1
N_2	0.326	0.009	758.69	2,710.178	0.146	2.7×10^6:1

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