Application Brief

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Coal Mine Gas Analysis with the Agilent 990 Micro GC

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Abstract

In this application brief, a fast coal mine gas analysis was performed on an Agilent 990 Micro GC configured with four analytical channels. The permanent gases and tracer gas SF_6 were analyzed on two Agilent J&W CP-Molsieve 5Å channels. C_2 hydrocarbons and hydrogen sulfide were analyzed on an Agilent J&W PoraPLOT U (PPU) channel. C_3 hydrocarbons and CO_2 were analyzed on an Agilent J&W PoraPLOT Q (PPQ) channel. The analysis cycle was less than 3 minutes. The excellent system performance was demonstrated by assessment results of the retention time/response precision and detection limit.

Introduction

Gas detection in the coal mine industry is critical to ensure protection from the hazardous gases generated in the mining environment. Gases that should be monitored include permanent gases, such as hydrogen, carbon monoxide and methane, C_2 to C_3 hydrocarbons, hydrogen sulfide, and tracer gas SF_6. Among them, the concentration change of hydrogen, methane, and C_2 light hydrocarbons can indicate whether there is an explosion risk. The inhalation of CO and H_2S threatens the health and safety of miners. From methane wick wall testing, use of canaries and flame lamps, to modern-day detection tubes, gas sensors, and GC techniques, many different methods have been used and developed for mine gas detection. Among them, GC-based measurement can provide accurate gas composition and concentration information in one analysis. The 990 Micro GC is a compact GC based on a micro thermal conductivity detector with minimized electricity and carrier gas consumption. These features reduce the risk of explosions, making the system suitable for use in the mining environment. The subsets of target analytes are analyzed on different channels of the 990 Micro GC. The separation performed on high-efficiency, narrow-bore PLOT columns under isothermal mode provides accelerated analysis compared with a conventional benchtop GC. In this application brief, the analysis speed, quantitation, and qualification precision of coal mine gas analysis on a 990 Micro GC are demonstrated.

Experimental

Analytical instrument

A 990 Micro GC was configured with four analytical channels. The standard gas composition is shown in Table 1. The channel types, analytical conditions, and corresponding target analytes on each channel are listed in Table 2.

Table 1. Coal mine gas standard composition information.

Compound Number	Compounds	Concentration (mol%)	Compound Number	Compounds	Concentration (mol%)
1	He	100 ppm	8	C_2H_4	150 ppm
2	H_2	100 ppm	9	C_2H_6	100 ppm
3	O_2	21%	10	C_2H_2	150 ppm
4	N_2	74%	11	H_2S	150 ppm
5	CH_4	2%	12	C_3H_8	200 ppm
6	CO	300 ppm	13	C_3H_6	200 ppm
7	CO_2	2%	14	SF_6	200 ppm

Table 2. Analytical methods for coal mine gas analysis.

Conditions	Channel Type			
	Agilent J&W CP-Molsieve 5Å, 10 m, RTS, Backflush, 1 m Precolumn	Agilent J&W CP-Molsieve 5Å, 10 m, RTS, Backflush, 1 m Precolumn	Agilent J&W PoraPLOT U, 10 m, Backflush	Agilent J&W PoraPLOT Q, 10 m, Backflush
Carrier Gas	Argon	Helium	Helium	Helium
Injector Temperature	80 °C	80 °C	80 °C	80 °C
Column Temperature	80 °C	60 °C	52 °C	60 °C
Column Pressure	150 kPa	150 kPa	150 kPa	150 kPa
Injection Time	150 ms	150 ms	40 ms	40 ms
Backflush Time	7.2 s	5.7 s	7.0 s	12.0 s
Invert Signal	Yes	No	No	No
Target Compounds	H_2	O_2, N_2, CH_4, CO, SF_6	C_2H_6, C_2H_4, C_2H_2, H_2S	SF_6, CO_2, C_3H_8, C_3H_6

The chromatograms of each channel are shown in Figure 1. SF_6, methane, and carbon monoxide showed satisfactory signal-to-noise ratios (S/N) on the Molsieve channel using helium as carrier gas. However, the hydrogen peak was very small, with a peak height of approximately 5 to 6 µv at a test concentration of 100 ppm. For this reason, hydrogen was analyzed using argon carrier gas on a second Molsieve channel for better sensitivity. Propane and propylene were well resolved on the PPQ channel, but not on the PPU channel. The backflush (BF) time on PPU was optimized to reduce analysis time by backflushing propane and propylene before they entered the main PPU analytical column. Ethylene and acetylene coeluted on the PPQ channel, but the two compounds and ethane can be well separated on the PPU channel. H_2S analysis is recommended to be performed on the PPU channel because of the improved inertness of the fused silica PPU column; this column has lower adsorption of H_2S and gives good peak shapes especially at the single-digit ppm concentration level. The PPQ column used in this application was based on metal tubing; the H_2S peak on the metal column is tailing, which will impact the detection limit. SF_6 and CO_2 cannot be separated at the baseline level on the PPU channel; their separation was much better on the PPQ and Molsieve channels. The best SF_6 peak was seen on the Molsieve channel, where SF_6 eluted very early, even before H_2. The SF_6 peak shape was very sharp, which benefits SF_6 identification and quantitation.

The system retention time (RT) and response precision of 20 consecutive injections are shown in Table 3. The average RT %RSD was 0.015%. This result is excellent considering the short retention time, which is due to the highly precise pneumatic and thermal control of the 990 Micro GC. For most analytes, the response precision was better than 1.0%. The peaks with area %RSD bigger than 1% are mainly wide asymmetric peaks with low response, which make it difficult to achieve reproducible integration. The baseline noise of each analytical channel was usually below 1 µv. The average peak height of the key compounds and 1 µv baseline noise were used for MDLs calculation. The MDLs were generated at an S/N ratio of 3:1 and are shown in Table 3. The MDLs on the PPU and PPQ channels were obtained with 40 ms injection time. If the injection time were increased to 150 ms, these MDLs could be improved approximately threefold. The SF_6 results on the PPQ channel were also included for reference.

Table 3. The RT and response results of target compounds in a coal mine gas standard.

Compound	RT (min)	RT RSD%	Area (mV × s)	Area RSD%	Peak Height (mV)	MDL (ppm) (S/N = 3)	Channel Number
H_2	0.463	0.0179	1.200	0.197	2.267	0.14	1
SF_6	0.346	0.0055	0.239	0.274	0.758	0.792	2
O_2	0.551	0.0042	296.8608	0.0161	240.251	NA	
N_2	0.857	0.0063	1,111.9806	0.0104	297.439	NA	
CH_4	1.360	0.0063	26.2467	0.0559	11.820	5.07	
CO	2.743	0.0163	0.4114	1.6160	0.118	7.63	
C_2H_4	0.4509	0.0067	0.0930	0.2201	0.231	1.95	3
C_2H_6	0.4932	0.007	0.0652	0.3779	0.113	2.66	
C_2H_2	0.5917	0.0085	0.0777	0.4795	0.149	3.02	
H_2S	0.8016	0.0121	0.0671	2.3441	0.095	4.74	
CO_2	0.4061	0.0127	13.6154	0.0673	54.973	NA	4
SF_6	0.4601	0.0185	0.1794	0.1742	0.256	2.34	
C_3H_6	1.3680	0.0438	0.1740	0.8989	0.118	5.09	
C_3H_8	1.5239	0.0517	0.1719	1.5300	0.082	7.32	

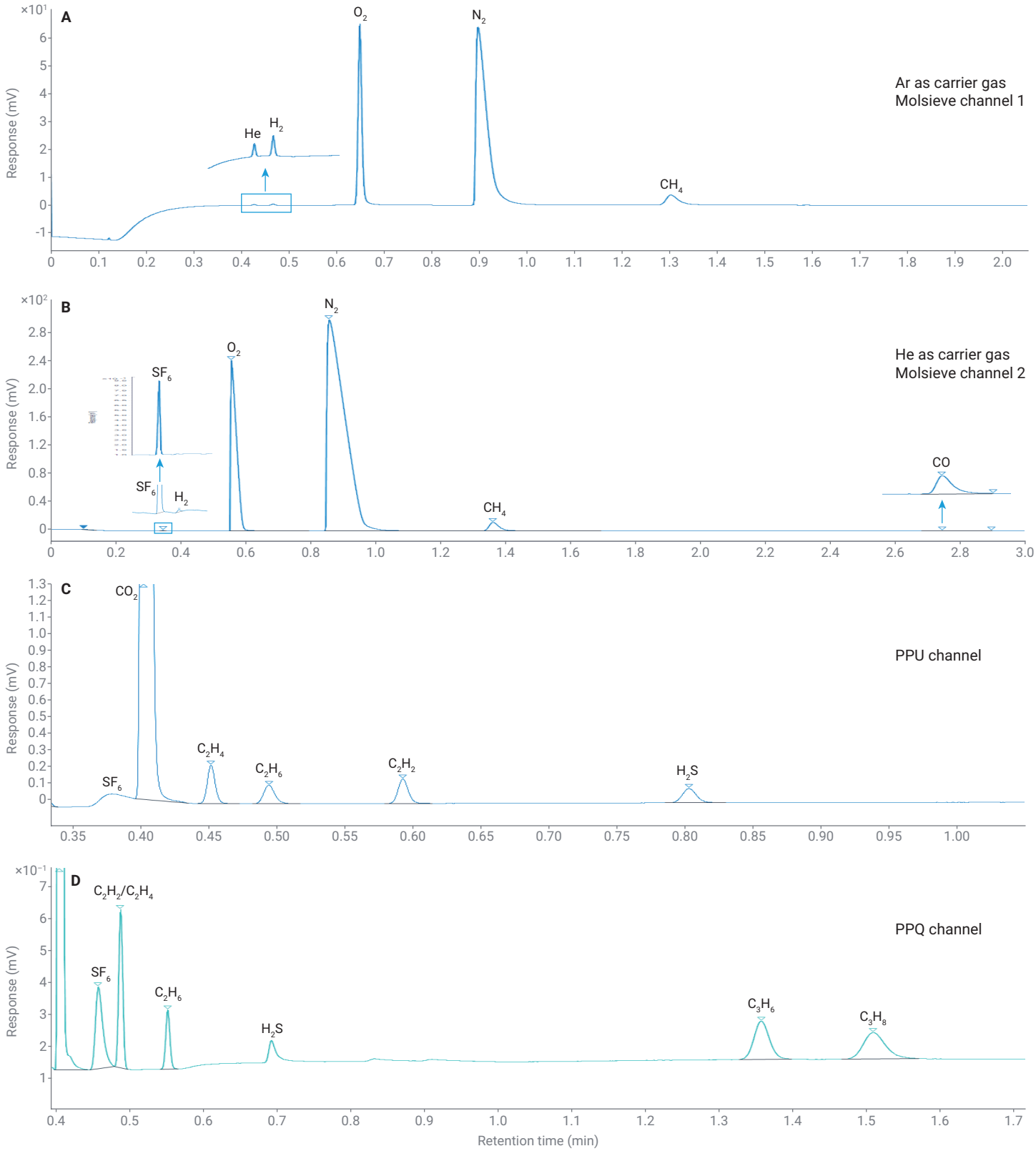

Figure 1. Chromatograms on four analytical channels. (A) H_2 analysis on the Agilent J&W CP-Molsieve 5Å channel with argon as carrier gas. (B, C, D) Analysis of other compounds using helium as the carrier gas.

Conclusion

In this application brief, an Agilent 990 Micro GC, configured with four analytical channels, including one PPU, one PPQ, and two Molsieve channels, was used for coal mine gas analysis. The channels were all of the backflush type, which can prevent heavier components entering analytical columns. This feature can reduce analysis time and generate a cleaner baseline for better detection limits. Thirteen target analytes were identified and quantitated based on the complementary results from the four analytical channels. Excellent retention time/response precision and MDL demonstrated that the 990 Micro GC can provide fast, accurate, and reliable results for coal mine gas analysis.

Reference

1. Brady, D.; van Loon, R. Fast On-Site Mine Safety Analysis by the Agilent 490 Micro GC. *Agilent Technologies application note*, publication number 5991-0438EN, **2012**.

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