Fast Analysis of Coal Mine Gas by Agilent 3000 Micro GC

Application

Hydrocarbon Processing

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Abstract

The Agilent 3000 Micro GC provides two fast and accurate solutions for the analysis of coal mine gas components. When configured with two channels, the GC baseline separates the individual components in less than 60 seconds and within 70 seconds when configured with four channels. The developed methods provide fast, real-time coal mine gas-detection solutions to provide rescuers with critical information before they attempt rescues in the mine. In this application, the trace indicators of coal mine explosion are also detected by the performance-enhanced Agilent 3000 Micro GC by reducing the big tailing of the main component. Using this method can help prevent coal mine explosions when they are in their early stages.

Introduction

Coal mine gas releases when coal beds are mined are serious safety hazards. When the released gases are mixed with air to the critical concentration, they are highly explosive and have been responsible for explosions in underground coal mines all over the world.

Because the concentration of carbon monoxide, ethylene, and acetylene and the ratio of paraffin and olefin in gas from coal mines can be used to predict a fire or explosion in an early stage, it is important to determinate the concentration of these gases in coal mines in real time.

The Agilent 3000 Micro GC is a powerful tool for the fast analysis of gas components [1–3]. Its robust construction and multiple channel configurations make analyzing complex samples quick and easy. Each channel or module is a self-contained GC comprising a micro-machined injector and detector and a high-resolution capillary column. Up to four different channels expand separation capabilities without compromising speed, making troubleshooting and repair simple. The performance-enhanced Agilent 3000 Micro GC is specially designed for the analysis of low-concentration components. The analytes in coal mine gas, ethylene and ethane compounds, are the indicators of coal mine explosion. When the concentration of these components approaches approximately 1 ppm, an explosion could occur if effective precautions are not taken. However, on a normal µGC these peaks are on the tailing of air peaks, affecting the detection limits of these indicators, as well as the reliability and repeatability of the tests.

In this application, two fast solutions are developed to analyze gas components from a coal mine by the Agilent 3000 Micro GC. The trace-level (1 ppm) spontaneous-combustion indicators, such as ethylene, ethane, and acetylene in coal mine gas, are also analyzed on performance-enhanced modules.

Experimental

Sample

The concentration and composition of the coal mine gas standards for the fast solution are shown in Table 1.

Table 1. Standard Compositions and Their Concentrations

Component	Concentration (µL/L)	Component	Concentration (µL/L)
O_2	400	C_2H_4	200
H_2	300	C_2H_6	600
CH_4	650	C_3H_6	500
CO	500	C_3H_8	450
CO_2	400	C_4H_{10}	400
C_2H_2	350		

The concentrations of low-level coal mine gas components are ethylene, 1.1 ppm; ethane, 1.1 ppm; acetylene, 1.1 ppm; and air as the balance. These standards were all provided by Beijing AP BAIF Gases Industry Co. Ltd. (Beijing, China).

Configurations and Analytical Conditions

Two-Channel Configured Micro GC Solution for Fast Coal Mine Gas Analysis

Two channels are used for the fast analysis solution on the Micro GC. The first channel is a molecular sieve column (molecular sieve 5Å, 10 m × 0.32 mm × 12 µm) for the separation of O_2, N_2, CH_4, and CO. The second channel is a PLOT U column (PLOT U, 8 m × 0.32 mm × 30 µm) for the separation of CO_2, C_2H_4, C_2H_6, C_2H_2, and C_3 compositions. The injectors used are all variable-volume type and helium is used as carrier gas. The analytical conditions are shown in Table 2.

Table 2. Analytical Conditions of Two-Channel Micro GC

Channel	Molecular Sieve 5Å (10 m × 0.32 mm × 12 µm)	PLOT U (8 m × 0.32 mm × 30 µm)
Sample inlet temperature (°C)	60	60
Injector temperature (°C)	80	80
Column temperature (°C)	100	85
Inject time (ms)	20	20
Run time (s)	100	100
Column pressure (psi)	40	40

Four-Channel Configured Micro GC Solution for Comprehensive and Fast Coal Mine Gas Analysis

A four-channel configuration of the Micro GC is listed in Table 3. The first channel is used to analyze H_2 in coal mine gas, and Ar is used as the car-

Table 3. Four-Channel Configuration of Micro GC

Channel	Molecular Sieve 5Å (10 m × 0.32 mm × 12 µm)	Molecular Sieve 5Å (8 m × 0.32 mm × 12 µm)
Sample inlet temperature (°C)	60	60
Injector temperature (°C)	80	80
Column temperature (°C)	60	100
Inject time (ms)	60	20
Run time (s)	100	100
Column pressure (psi)	40	40
Carrier gas	Ar	He
Channel	**PLOT U (10 m × 0.32 mm × 30 µm)**	**PLOT Q (10 m × 0.32 mm × 10 µm)**
Sample inlet temperature (°C)	60	60
Injector temperature (°C)	80	100
Column temperature (°C)	80	120
Inject time (ms)	20	20
Run time (s)	100	100
Column pressure (psi)	40	40
Carrier gas	He	He

rier gas because of the sensitivity problem. The fourth channel is for the separation of C_3H_6, C_3H_8, and C_4H_{10} in coal mine gas. The second and third channels are the same as those in the two-channel configured Micro GC.

Low-Level Coal Mine Indicators Analysis on Performance-Enhanced Agilent 3000 Micro GC

The low-concentration coal mine gas indicators were detected on the performance-enhanced module using a PLOT U column. The separation conditions are shown in Table 4.

Table 4. Separation Conditions of Coal Mine Indicators on Performance-Enhanced Module

Analytical column	PLOT U (8 m × 0.32 mm × 30 µm)
Injector	Large volume injector
Carrier gas	He
Sample inlet temperature (°C)	50
Injector temperature (°C)	50
Column temperature (°C)	50
Sampling time (s)	10
Inject time (ms)	200
Run time (s)	90
Column pressure (psi)	30

Results and Discussion

Two-Channel Configured Micro GC for Fast Analysis of Coal Mine Gas

O_2, CO, and CH_4 were separated within 40 seconds and CO_2, C_2H_4, C_2H_6, C_2H_2, and C_3 within 60 seconds. With this configuration, the fast, real-time analysis of the main concerned gas compositions was achieved. Figures 1A and 1B show the chromatograms of channels A and B, respectively.

Table 5 shows the run-to-run repeatability by relative standard deviations (RSDs) of the peak area. The RSDs of separated compositions are all less than 1 percent, which means that the Agilent Micro GC with this configuration is stable and reliable.

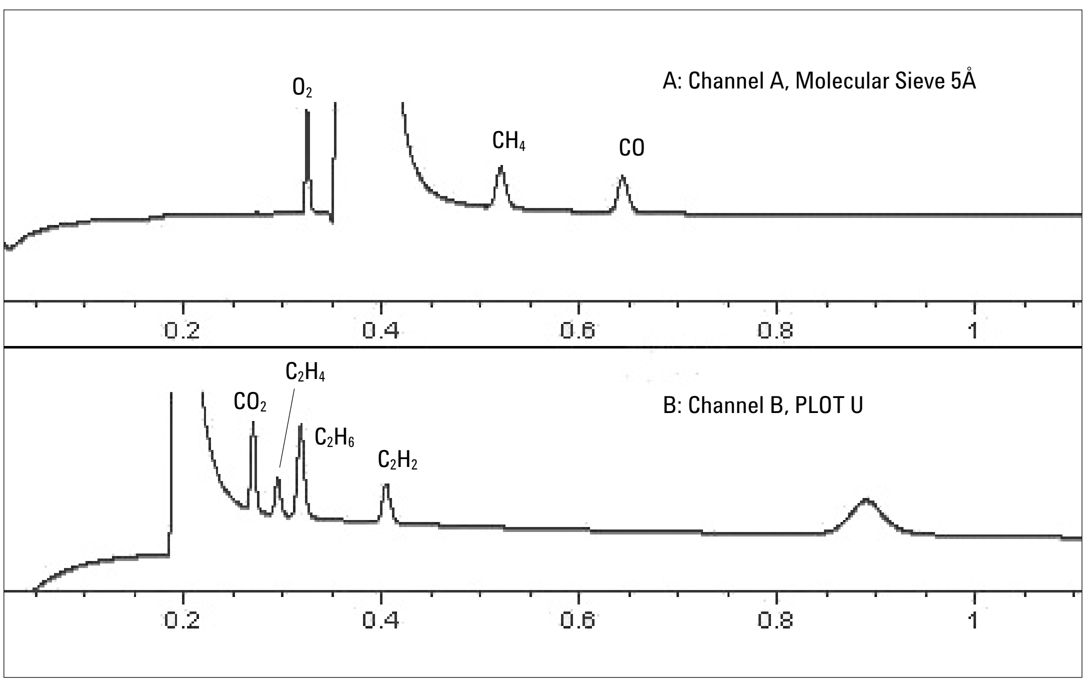

Figure 1. Chromatograms of coal mine gases.

Table 5. RSDs for Peak Area

Compound	Avg. RT	Avg. peak area	Peak area SD	RSD (n = 10)
O_2	0.322	1140.63	4.62	0.41
CH_4	0.519	1269.57	2.42	0.19
CO	0.644	1190.11	10.12	0.85
CO_2	0.270	918.22	3.38	0.37
C_2H_4	0.295	453.82	2.14	0.47
C_2H_6	0.317	1478.03	4.43	0.30
C_2H_2	0.404	695.19	2.53	0.36
C_3H_6 and C_3H_8	0.889	2519.28	3.10	0.12

Four-Channel Configured Micro GC for Fast and Comprehensive Analysis of Coal Mine Gas

For Micro GC thermal conductivity detector (TCD) systems, helium is the best choice of carrier gas, because it has the highest sensitivity for all other analytes except for hydrogen. When hydrogen is the target analyte, argon, not helium, is chosen as the carrier gas. This is described in the application for the detection of hydrogen in fuel cell reformer gases by Micro GC [1], in which argon shows the most linear performance and the most sensitivity for hydrogen. In this solution for coal mine gas analysis, hydrogen and n-butane were separated by the four-channel configured Micro GC; hydrogen analysis was realized in channel A and argon was used as the carrier gas. Channel D with a PLOT Q column was used for the analysis of C_3H_6, C_3H_8, and n-C_4H_{10}. Figure 2 shows the chromatograms of the analytes in the four channels. The 11 coal mine components are separated in less than 70 seconds.

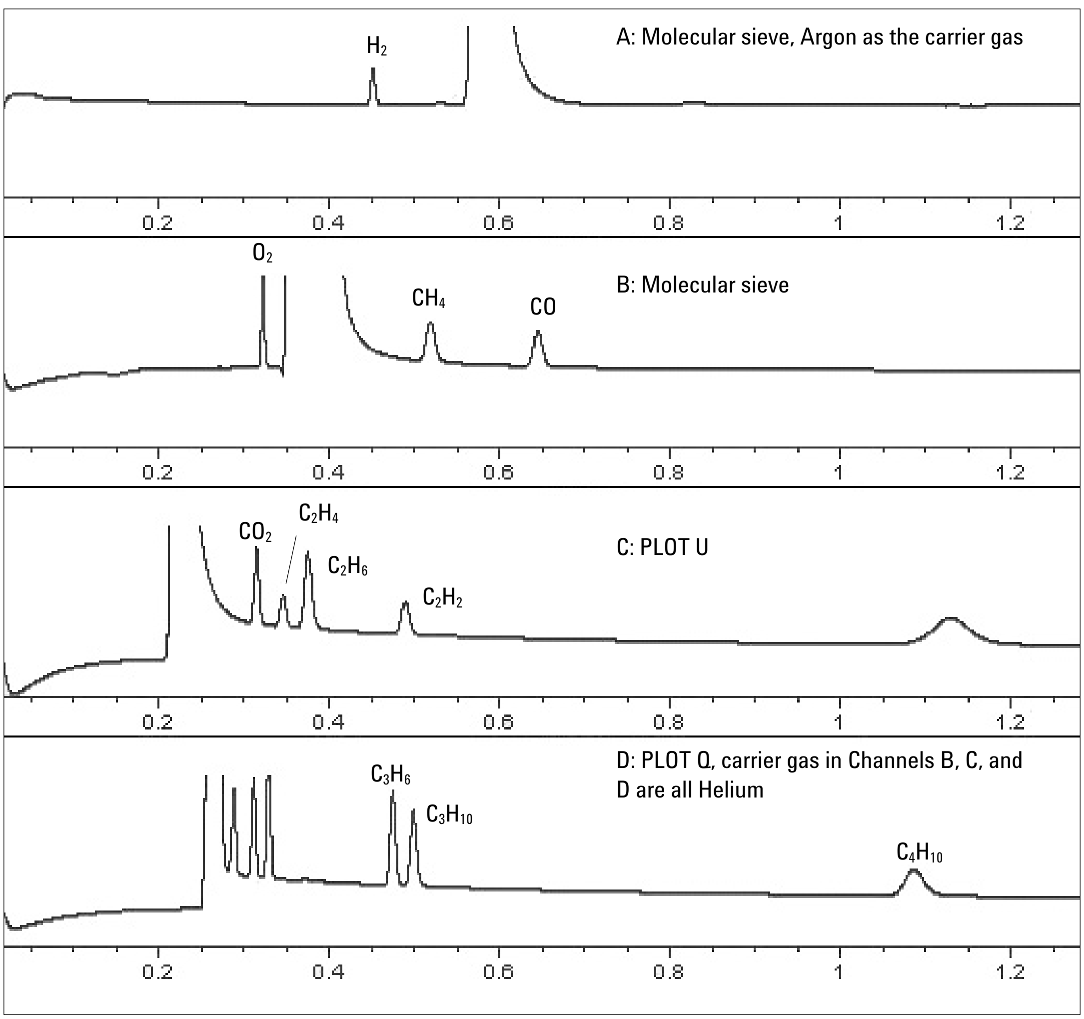

Figure 2. Chromatograms of coal mine gases by a four-channel Micro GC.

The run-to-run repeatability of the four-channel configured Micro GC is shown in Table 6. This table illustrates that the Micro GC shows excellent precision, with all RSDs below 1%.

Table 6. Reproducibility of Four-Channel Micro GC Solution

Compound	Avg. RT	Avg. peak area	Peak area SD	RSD (n = 10)
H_2	0.451	606.61	5.89	0.97
O_2	0.322	1140.60	4.58	0.40
CH_4	0.519	1269.59	2.43	0.19
CO	0.644	1190.11	10.11	0.85
CO_2	0.314	918.22	3.37	0.37
C_2H_4	0.345	453.89	2.24	0.49
C_2H_6	0.375	1478.07	4.45	0.30
C_2H_2	0.489	695.19	2.59	0.37
C_3H_6	0.474	1397.50	5.28	0.38
C_3H_8	0.498	1283.31	4.49	0.38
C_4H_{10}	1.085	1274.80	8.42	0.66

Low-Level Coal Mine Indicators Analysis on Performance-Enhanced Agilent 3000 Micro GC

The Agilent 3000 Micro GC improves the ability to produce chromatograms with peaks generated by 1 ppm analytes that are well-resolved and well-shaped with flat baselines to reliably detect and accurately integrate such peaks. This helps make the accurate quantitation of trace-level analytes possible and reliable. Figure 3 shows the 1 ppm ethylene, 1.1 ppm ethane, and 1.1 ppm acetylene analysis on a PLOT U module. The Agilent 3000 Micro GC exhibits high-sensitivity performance, as the 1 ppm gas is easily detected with an excellent signal-to-noise ratio (more than 3).

Conclusions

The Agilent 3000 Micro GC provides flexible and reliable configurations for fast coal mine gas analysis. These configurations achieve fast, dependable results, which ensures the real-time analysis of coal mine gases.

By using the performance-enhanced Agilent 3000 Micro GC system, the concentration of spontaneous combustion indicators lower than 1 ppm are well-detected, with a signal-to-noise ratio of more than 3. The result meets the requirement of coal mine safety, which can help mine operators detect explosion potential at an early stage.

References

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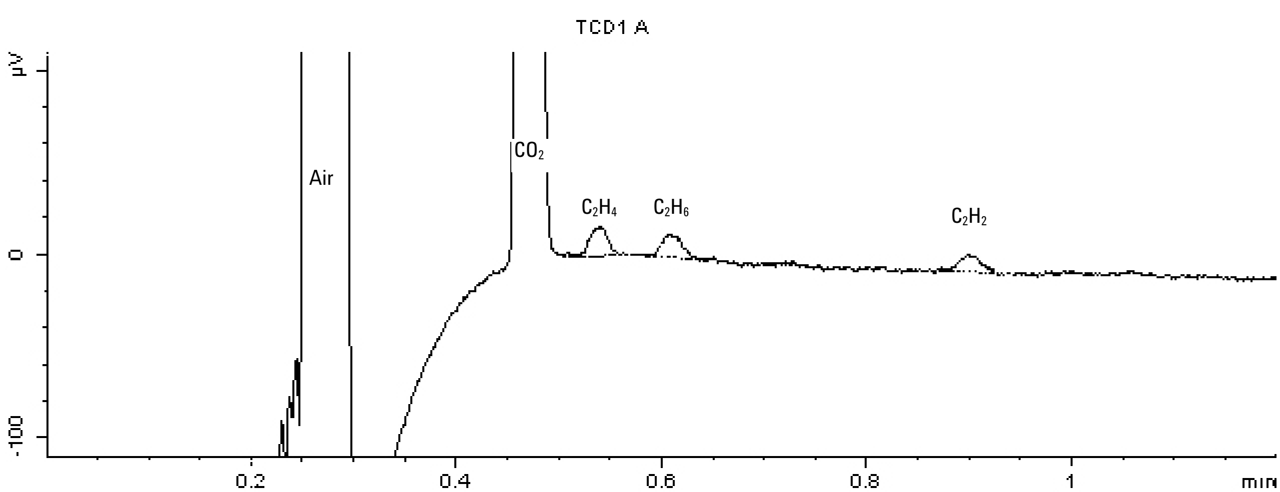

Figure 3. Chromatogram of low-level coal mine indicators analyzed on a performance-enhanced PLOT U column.

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