Application Brief

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One-Minute Analysis of Mud Logging Gas Using the Agilent 990 Micro GC

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Introduction

Mud logging gases are the compounds found in the drilling mud collected during the oil and gas exploration process. The quantitative monitoring of mud logging gas is critical for identifying zones of producible oil and gas. It requires a rapid, sensitive, and accurate gas analysis method. The Agilent 990 Micro GC has already been proven to be an ideal platform for the rapid quantitative analysis of the hydrocarbons C_1 to C_8 in mud logging gas.[1]

This application introduces a new method that can also provide a full separation of the permanent gases (hydrogen, oxygen, nitrogen, and methane). All components of interest, including separated permanent gases, carbon dioxide, sulfides, and hydrocarbons up to C_8, can be quantitatively analyzed within 40 seconds. The total analysis cycle is less than one minute.

Experimental

The 990 Micro GC was equipped with three channels: a 1 m + 10 m Agilent J&W CP-Molsieve 5Å with backflush channel with retention time stability (RTS) filters, a 1 m + 10 m Agilent J&W PoraPLOT U with backflush channel, and a 4 m Agilent J&W CP-Sil 5 CB straight channel. The RTS filters are used to filter out the water and carbon dioxide contaminants in the carrier gas. This filtration protects the J&W CP-Molsieve 5Å column and mitigates the retention time shift. Table 1 shows the experimental conditions of this application.

Results and discussion

The compounds measured in mud logging operations are listed in Table 2. The experimental data, including repeatability and detection limit, is shown in Table 3. The lower detection limits (LDL) were calculated as two times the four-sigma noise. For channels 1 and 2, ppm level LDLs were achieved (except for hydrogen). For channel 3, sub-ppm LDLs were obtained. As listed in Table 1, the typical injection time was 40 ms for channels 1 and 2, and 20 ms for channel 3. The injection time could be increased when lower detection limits are desired. For all components, the retention time (RT) precisions were less than 0.1% RSD, and the area precisions were less than 1% RSD, showing excellent stability of the system.

The 10 m J&W CP-Molsieve 5Å with backflush channel was equipped to separate hydrogen, oxygen, nitrogen, and methane. All components eluted within 40 seconds and were well separated. Figure 1 shows the chromatogram of the 10 m CP-Molsieve 5Å channel. It should be noted that the hydrogen peak will be split or negative at high concentration (>10%). Therefore, the hydrogen peak area is no longer linear when using helium as the carrier gas. A more detailed discussion can be found here.[2]

The 10 m J&W PoraPLOT U with backflush channel was equipped to separate carbon dioxide, ethane, hydrogen sulfide, carbonyl sulfide, and propane. The whole sample line inside the 990 Micro GC was inert, enabling the ppm level analysis of hydrogen sulfide. Figure 2 shows the chromatogram of this channel.

The 4 m J&W CP-Sil 5 CB channel was equipped to separate C_4 to C_8 hydrocarbons. All peaks eluted within 40 seconds with the well optimized method. Figure 3 shows the chromatogram of this channel.

Table 1. Test conditions for mud logging gas.

Channel Type	1 m + 10 m Agilent J&W CP-Molsieve 5Å, Backflush, RTS	1 m + 10 m Agilent J&W PoraPLOT U, Backflush	4 m Agilent J&W CP-Sil 5 CB, Straight
Carrier Gas	Helium	Helium	Helium
Column Pressure	300 kPa	250 kPa	300 kPa
Injector Temperature	80 °C	80 °C	80 °C
Column Temperature	100 °C	80 °C	90 °C
Injection Time	40 ms	40 ms	20 ms
Backflush Time*	4.2 s	7.0 s	NA
Sampling Time	15 s		
Run Time	40 s		

*The backflush time should be tuned for each new channel.

Table 2. Composition of the mud logging gas standard.

Component	Concentration
Hydrogen	0.980%
Oxygen	0.204%
Methane	2.02%
Carbon Dioxide	0.199%
Ethane	0.259%
Hydrogen Sulfide	95.08 ppm
Carbonyl Sulfide	97.45 ppm
Propane	972.30 ppm
i-Butane	488.04 ppm
n-Butane	292.67 ppm
i-Pentane	136.15 ppm
n-Pentane	179.93 ppm
n-Hexane	55.24 ppm
Methylcyclopentane	51.86 ppm
Benzene	60.08 ppm
Cyclohexane	52.57 ppm
n-Heptane	57.77 ppm
Methylcyclohexane	58.46 ppm
Toluene	55.70 ppm
n-Octane	58.65 ppm
Nitrogen	Balance

Table 3. Retention time, peak area repeatability, and lower detection limits for 10 replicate analyses of the standard gas.

Component	Channel	RT (min)	RT RSD	Area (mV × s)	Area RSD	LDL (ppm)
Hydrogen	1	0.239	0.0047%	0.0639	0.692%	47.1
Oxygen	1	0.314	0.0038%	0.700	0.047%	0.957
Nitrogen	1	0.388	0.0022%	350.4	0.054%	4.66
Methane	1	0.555	0.0020%	6.52	0.033%	3.38
Carbon Dioxide	2	0.242	0.0034%	1.22	0.065%	0.797
Ethane	2	0.274	0.0033%	1.78	0.035%	0.796
Hydrogen Sulfide	2	0.362	0.0036%	0.0502	0.406%	1.27
Carbonyl Sulfide	2	0.424	0.0027%	0.0780	0.685%	1.08
Propane	2	0.532	0.0042%	0.836	0.489%	2.92
i-Butane	3	0.082	0.011%	0.331	0.074%	0.262
n-Butane	3	0.089	0.011%	0.203	0.043%	0.264
i-Pentane	3	0.111	0.010%	0.105	0.124%	0.353
n-Pentane	3	0.121	0.010%	0.144	0.088%	0.337
n-Hexane	3	0.184	0.0095%	0.0517	0.355%	0.436
Methylcyclopentane	3	0.219	0.014%	0.0432	0.407%	0.608
Benzene	3	0.249	0.0065%	0.0454	0.536%	0.635
Cyclohexane	3	0.264	0.014%	0.0436	0.540%	0.711
n-Heptane	3	0.310	0.015%	0.0566	0.415%	0.643
Methylcyclohexane	3	0.371	0.016%	0.0503	0.658%	0.928
Toluene	3	0.446	0.013%	0.0472	0.467%	0.895
n-Octane	3	0.560	0.036%	0.0668	0.312%	0.884

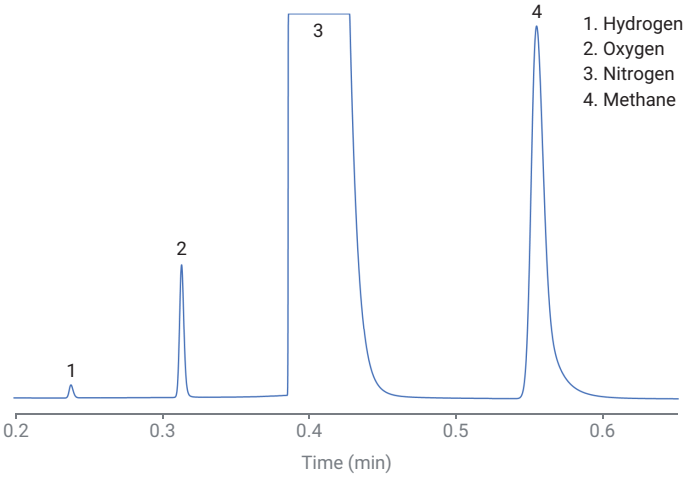

Figure 1. Chromatogram of hydrogen, oxygen, nitrogen, and methane on the 10 m Agilent J&W CP-Molsieve 5Å with backflush channel.

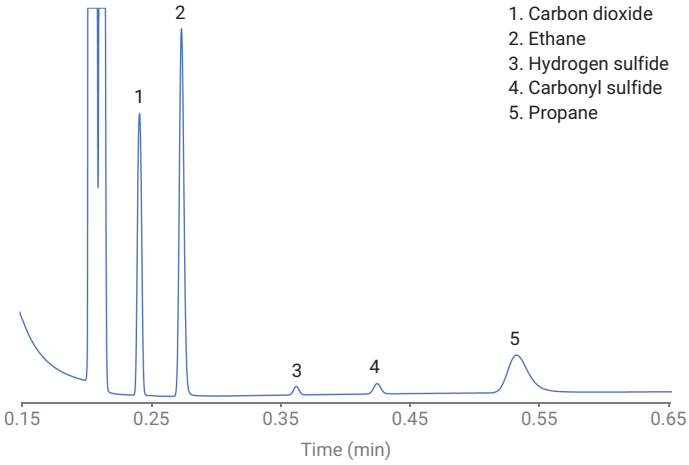

Figure 2. Chromatogram of carbon dioxide, ethane, hydrogen sulfide, carbonyl sulfide, and propane on the 10 m Agilent J&W PoraPLOT U with backflush channel.

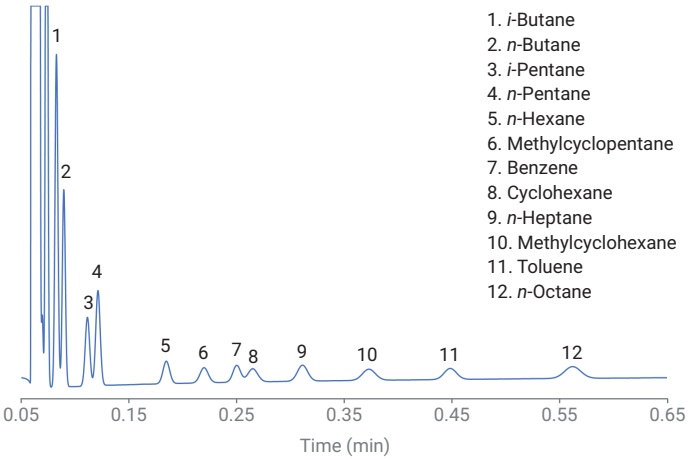

Figure 3. Chromatogram of C_4 to C_8 hydrocarbons on the 4 m Agilent J&W CP-Sil 5 CB channel.

Conclusion

This application shows the chemical performance of the Agilent 990 Micro GC system for the one-minute analysis of mud logging gas. The analysis includes the separation of permanent gases and hydrocarbons up to C_8. All components of interest have excellent repeatability and detection limits, making the 990 Micro GC an excellent platform for the rapid analysis of mud logging gas.

References

1. Zhang, J. Rapid Analysis of Mud Logging Well Gas Using the Agilent 990 Micro GC. *Agilent Technologies application note*, publication number 5994-1039EN, **2019**.
2. Toonen, A. Hydrogen Detection with a TCD Using Mixed Carrier Gas on the Agilent Micro GC. *Agilent Technologies application note*, publication number 5991-3199EN, **2013**.

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