Application Note

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One-Minute Natural Gas Analysis Using the Agilent 990 Micro GC Backflush-to-Detector Option

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Introduction

Natural gas analysis (NGA) is a key application area for the Agilent 990 Micro GC. Today, there are four NGA analyzers based on the 990 Micro GC platform.[1] The configuration of each analyzer depends on the composition of natural gas and compounds of interest. Each of the four analyzers adopts the same principle for hydrocarbons analysis: completely resolving individual target hydrocarbons for qualification and quantitation purposes. Based on this principle, the analysis time for the 990 Micro GC NGA Analyzers is determined by how long it takes the heaviest hydrocarbons to elute from the CP-Sil 5CB columns. It is difficult to reduce the analysis time on the 990 Micro GC NGA analyzer to under one minute because during this short period of time the heavy components cannot elute from the analytical column completely. In addition, the late-eluting hydrocarbon is often observed as a broadening peak, impacting its integration and detection.

In traditional natural gas analysis solutions based on a conventional GC platform, C_6/C_6-plus hydrocarbons (heavy components) are commonly analyzed by being backflushed as a bundled peak for detection. The backflush (BF) approach can significantly reduce the overall analysis time. A similar idea has been implemented by Agilent in the 990 Micro GC. A backflush-to-detector option on an 8 m CP-Sil 5CB channel was developed to resolve C_3 to C_5 hydrocarbons in a fingerprint profile within one minute, and backflush C_6/C_6-plus hydrocarbons as one bundled peak for natural gas analysis.[2]

For users who require faster turnaround and accept the concept of quantifying C_6-plus hydrocarbons using hexane response factor, the backflush-to-detector (BF2D) option is an ideal choice for faster natural gas analysis. This Application Note demonstrates a one-minute natural gas analysis solution using the 990 Micro GC BF2D option.

Table 1. The composition of simulated natural gas.

Compound	Concentration (mol%)
Nitrogen	1.01
Oxygen	0.02
Carbon Dioxide	5
Methane	Balance
Ethane	1.5
Propane	0.4
Isobutane	0.05
Butane	0.05
2,2-Dimethylpropane	0.01
Isopentane	0.03
Pentane	0.03
2,2-Dimethylbutane	0.01
Hexane	0.005
Heptane	0.005
Octane	0.005
Nonane	0.005

Instrumentation

Channel 1

A 40 cm, HayeSep A, straight channel, was used for air, methane, carbon dioxide, ethane, and propane analysis. Figures 1A and 1B show the chromatograms of simulated natural gas on channel 1. Air, methane, carbon dioxide, ethane, and propane are well resolved. Propane elutes at 53 seconds, and it can be used as a bridge component between channels 1 and 2. The analysis is completed within one minute.

Table 2. Method for NGA on HayeSep A and BF2D CP-Sil 5CB channels.

Channel Type	40 cm, HayeSep A, Straight	8 m, CP-Sil 5CB, BF2D
Carrier Gas	Helium	Helium
Injector Temperature	110 °C	110 °C
Injection Time	40 ms	40 ms
Column Head Pressure	280 kPa	150 kPa
Column Temperature	80 °C	72 °C
Backflush Time	NA	7 seconds
Invert Signal	NA	From 9 to 19 seconds

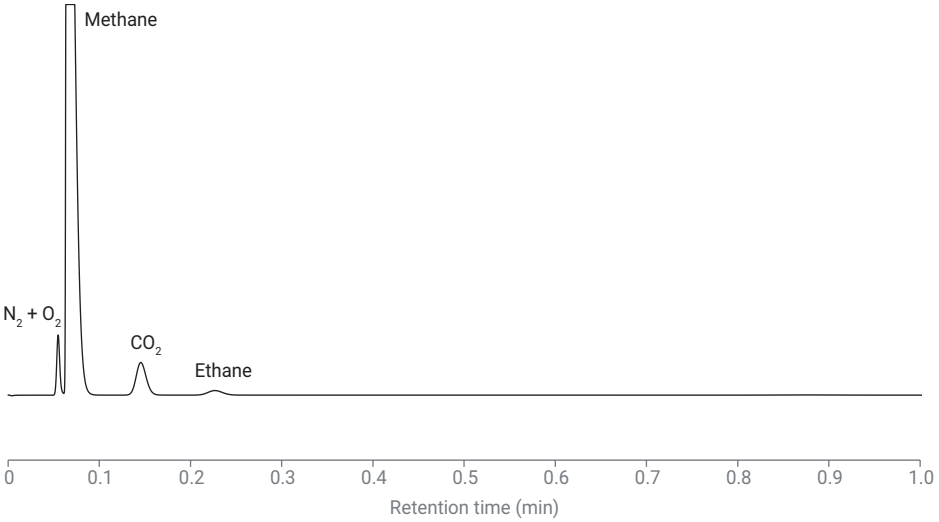

Figure 1A. Chromatogram of air, methane, carbon dioxide, and ethane on the HayeSep A channel.

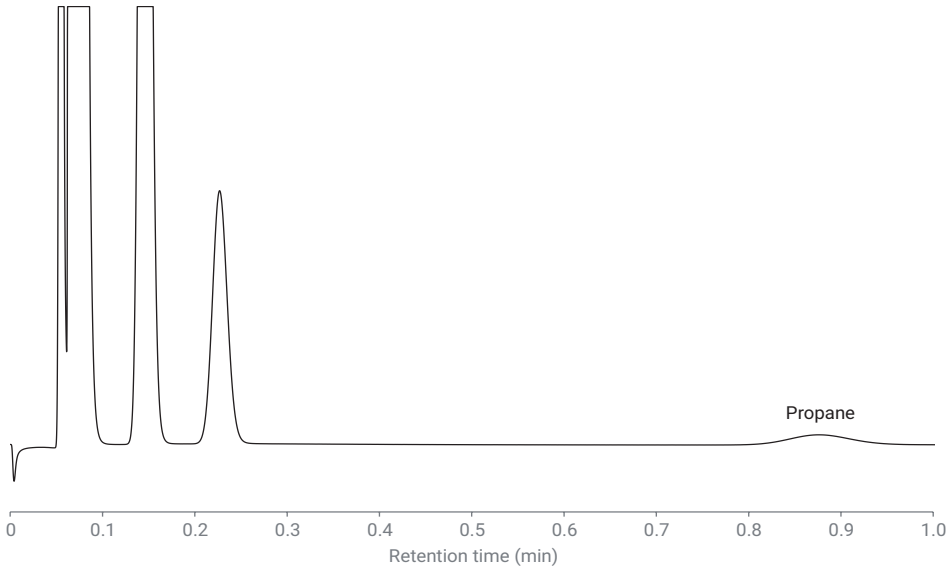

Figure 1B. Enlarged chromatogram of propane on the HayeSep A channel.

Channel 2

An 8 m, CP-Sil 5CB, BF2D option, for propane, butane, isobutane, pentane, isopentane, 2,2-dimethylpropane, C_6/C_6-plus hydrocarbons analysis was used.

Figure 2 shows the chromatogram of C_3 to C_6/C_6-plus hydrocarbons. The C_3 to C_5 compounds are well separated after ethane. The C_6/C_6-plus bundled peak elutes before the peak of methane/air. A specially selected precolumn is used in the 8 m CP-Sil 5CB, BF2D channel to facilitate the effective separation of C_6/C_6-plus and methane. Before pentane enters the analytical column, carrier gas goes from the precolumn to the analytical column. After pentane enters the analytical column, with the activation of the backflush valve, the carrier gas enters the precolumn and the analytical column simultaneously. The flow in the precolumn is reversed to flush the C_6/C_6-plus bundled peak to the reference column for detection. A negative peak will be detected when the bundle of compounds flows through the TCD detector. This negative peak is inverted in real time to a positive peak for easy integration. This signal-inverse function is realized in a preset time range—a feature designed for the BF2D channel in the Agilent chromatography data system: OpenLab CDS, OpenLab Chemstation, OpenLab EZChrom and Prostation for 990 PRO Micro GC.

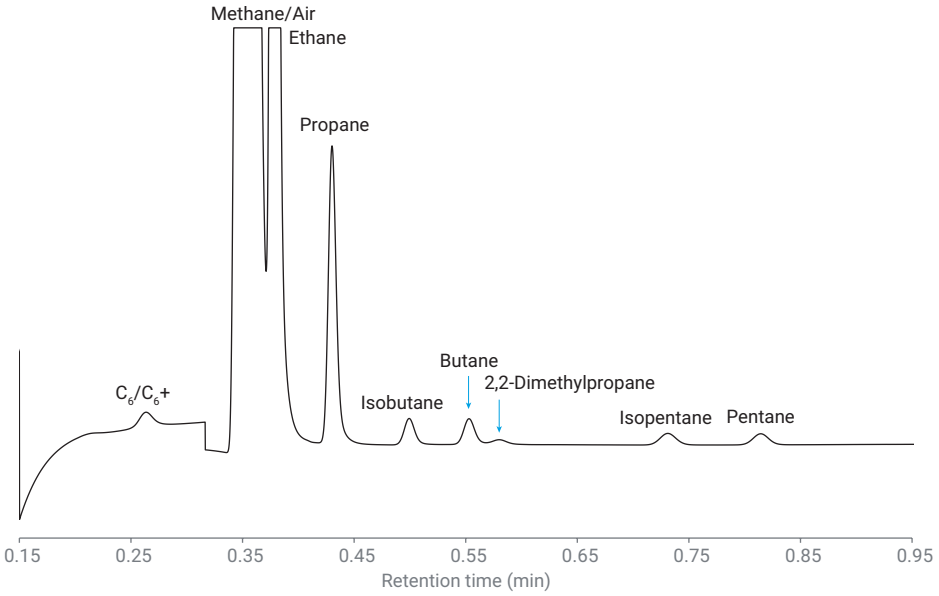

Figure 2. Chromatogram of stimulated natural gas on the 8 m, CP-Sil 5CB, BF2D channel.

Finding the right backflush time is critical for bundled C_6/C_6-plus peak detection. The appropriate backflush time on each BF2D channel usually occurs within a short time window. The shipped BF2D channel has a preset backflush time, which is factory-tuned under a given set of conditions. Users can use the same conditions and the preset backflush time for their own analysis, or use the factory-tuned value as reference for further optimization when they use different analytical conditions. In this experiment, the backflush time was seven seconds, and pentane eluted at approximately 52 seconds. The total analysis time was less than 60 seconds, with baseline separation of the C_6/C_6-plus, propane, butane, isobutane, pentane, and isopentane peaks.

Among the current four 990 Micro GC NGA analyzers, the NGA analyzer A extended can provide the fastest separation for heavy hydrocarbons. We ran the simulated natural gas (up to C_9) on the NGA analyzer A extended and compared its speed with the BF2D solution. The NGA analyzer A extended took approximately 75 seconds to detect the peak of n-C_9. The BF2D-based approach took one minute to complete the analysis, with a speed improvement of 20%. The heavier the hydrocarbons that need to be analyzed, the more the speed benefit to be achieved with the BF2D-based solution.

Table 3 shows the area and retention time (RT) repeatability of natural gas analysis based on the 8 m, CP-Sil 5CB BF2D option. The area repeatability was between 0.1% and 3.1%, depending on component concentration. RT repeatability was between 0.005% and 0.1%. The good instrument repeatability showed that the one-minute analysis for natural gas is a reliable solution.

Table 3. RT and area repeatability of 20 runs on HayeSep A and BF2D CP-Sil 5CB channels.

Peak No.	Compound	RT (min)	RT RSD%	Area (mv × s)	Area RSD%
1	Nitrogen/Oxygen	0.055	0.046	10.54	0.66
2	Methane	0.065	0.061	414.66	0.06
3	Carbon dioxide	0.146	0.035	19.323	0.08
4	Ethane	0.227	0.034	4.04	0.15
5	Propane	0.430	0.009	3.503	0.65
6	Isobutane	0.499	0.008	0.404	0.56
7	Butane	0.553	0.007	0.418	0.74
8	2,2-Dimethylpropane	0.580	0.012	0.111	3.1
9	Isopentane	0.731	0.005	0.274	1.0
10	Pentane	0.814	0.006	0.257	1.2
11	C_6/C_6-plus	0.264	0.09	0.338	3.0

Conclusion

A fast solution for natural gas analysis was developed on the Agilent 990 Micro GC platform. A two-channel configuration: the first a straight HayeSep A channel, the second a CP-Sil 5CB BF2D channel, was used to analyze natural gas within one minute. Methane, air, carbon dioxide, ethane, and propane were analyzed on the HayeSep A channel. Propane, butane, isobutane, pentane, isopentane, 2,2-dimethyl propane, and C_6/C_6-plus hydrocarbons were resolved on the BF2D CP-Sil 5CB channel. The system repeatability is good. Compared to other 990 Micro GC NGA analyzers, this fast solution can further improve natural gas analysis speed.

References

1. Fast Analysis of Natural Gas Using the Agient 990 Micro GC Natural Gas Analyzer, *Agilent Technolgoies Applicaiton Note*, publication number 5994-1040EN, **2019**.
2. One-Minute NGA Analysis Based on a Backflush-to-Detector Channel, *Agilent Technologies Application Note*, publication number 5991-9401EN, **2018**.

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