Application Note

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One-Minute NGA Analysis Based on a Backflush-to-Detector Channel

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Introduction

Natural gas analysis is a key application for the Agilent 490 micro GC. Currently, there are four types of NGA analyzers available based on different configurations to address various NGA analysis requirements. Application Note 5991-0275 lists currently available analyzer configurations, their targeted NGA composition, and typical analysis time. The total analysis time for the 490 Micro GC NGA analyzer is usually determined by how long it takes for the heavy hydrocarbons to elute from the 5CB columns. The more complex the matrices and the heavier the hydrocarbons, the longer the analysis time. If the analysis time is not long enough, the heavier components from the previous injection will interfere with the baseline of the following injection. That is why it takes 200–300 seconds for samples with components up to C12.

For components heavier than C6 (up to C9 or even C12), some customers are interested in detailed identification and corresponding concentration of each heavy component, and some only need to know the total concentration of C6 and C6+ components. In the latter situation, there is no need to separate each heavy component.

For customers who are only interested in the total concentration of C6 and heavier components, and are sensitive to analysis speed, there is a column channel designed for fast and complete elution of C6/C6+ components without interfering with the separation of lighter hydrocarbons such as propane, *i*-butane, *n*-butane, and *neo*, *i*-, and *n*-pentane on the same column. This developed backflush-to-detector channel, coupled with existing channels specializing in methane analysis, results in a new configuration for faster NGA analysis with total analysis times under 60 seconds.

Instrumentation

The configuration of the 490 Micro GC NGA analyzer is comprised of one straight Hayesep channel and one CP-Sil 5CB backflush-to-detector (BF2D) channel.

The separation of air, C1, and C2 components is performed on an HSA channel. Figure 1 depicts the chromatogram of an NGA sample on this Hayesep channel, completing the analysis within 60 seconds.

Table 1. Detailed method for NGA analysis on HSA and BF2D 5CB channels.

Channel type	40 cm, straight Hayesep	8 m BF2D CP-Sil 5CB
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
BF time	NA	6 s

Sample

Peak no.	Component	Concentration
1	N_2	5 %
2	CH_4	Bal
3	CO_2	1.50 %
4	C_2H_6	9 %
5	n-C_6H_{14}	0.10 %
6	C_3H_8	6 %
7	i-C_4H_{10}	3 %
8	n-C_4H_{10}	2 %
9	i-C_5H_{12}	0.50 %
10	n-C_5H_{12}	0.50 %

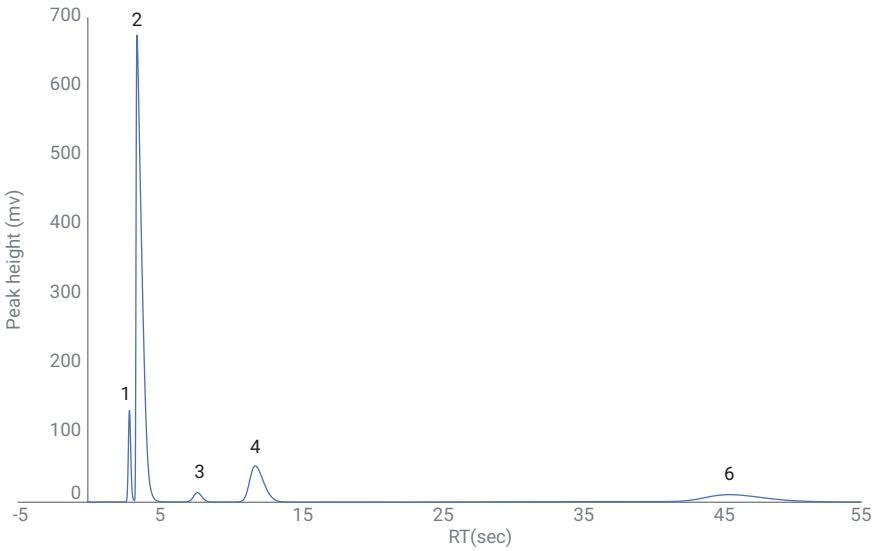

Figure 1. Nitrogen, methane, carbon dioxide, and ethane separation on a straight HSA channel.

Figure 2 shows the chromatogram of C6/C6+ components, propane, *i*-butane, *n*-butane, *iso*-pentane, and *n*-pentane separated on an 8 m BF2D 5CB channel. C6 and C6+ components are trapped first by the precolumn, then backflushed through the reference column to the TCD, resulting in one peak, shown in the chromatogram (peak 5). The peak was inverted real-time to a positive peak for easy integration in 490 Micro GC instrument control software: Agilent OpenLab CDS, OpenLab ChemStation, OpenLab EZChrom, and Prostation for the 490-PRO Micro GC. This signal-reverse function can be realized in a preset time range—a feature designed for the backflush-to-detector channel. Compared to the previous signal-reverse function applicable only to the whole run time, it is more flexible.

The backflush (BF) time should be adjusted to ensure that interested hydrocarbons enter the analytical column completely, and that C6/C6+ components are backflushed into the precolumn without any penetration into the analytical column. This BF time was adjusted in-factory based on a given set of conditions. This can be used as a reference/starting point for the customer's further optimization, based on their own test parameters. In this experiment, the BF time was six seconds, and the total analysis time was below 60 seconds, with good separation of the C6/C6+ combined peak, as well as air/methane, propane, *iso*-/*n*-butane, and *iso*-/*n*-pentane peaks.

Table 2 lists the RT and area repeatability of individual components on two channels. The area repeatability was approximately 0.1 %. RT repeatability was between 0.03 % and 0.1 %, demonstrating the excellent repeatability of the new configuration. Such precision guarantees qualification and quantitation results with level a high confidence.

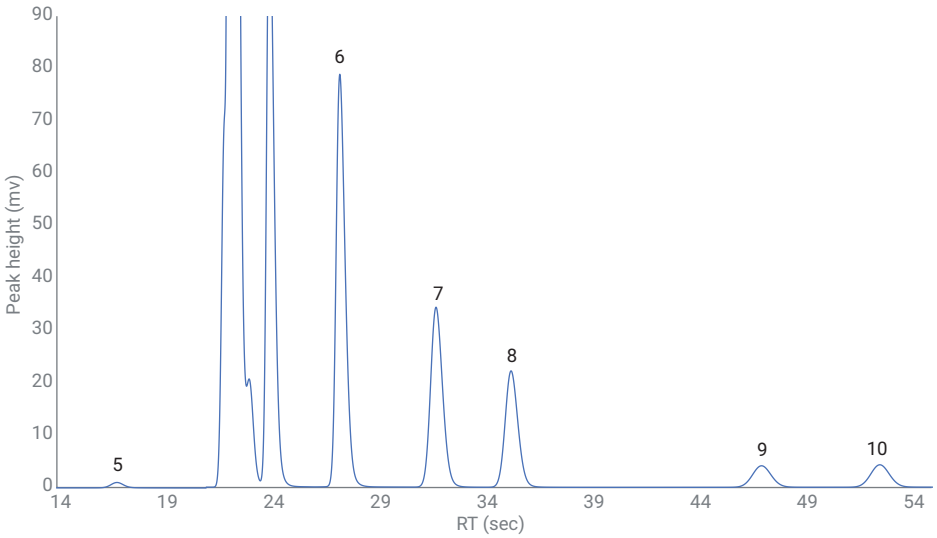

Figure 2. Chromatogram of C6/C6+ compounds, propane, *iso*-butane, *n*-butane, *iso*-pentane, and *n*-pentane.

Table 2. RT and area repeatability of 15 injections using the BF2D configuration.

Compound	RT (min)	RT RSD%	Area (mv*s)	Area RSD%
Nitrogen	0.049	0.1	24.03	0.06
Methane	0.058	0.071	321	0.06
Carbon dioxide	0.128	0.041	9.60	0.12
Ethane	0.193	0.034	62.00	0.09
Propane	0.455	0.034	34.48	0.12
i-Butane	0.531	0.034	19.80	0.12
n-Butane	0.589	0.032	13.59	0.12
i-Pentane	0.786	0.032	3.71	0.12
n-Pentane	0.879	0.031	3.92	0.13
C6/C6+	0.282	0.030	0.62	0.14

If the same sample was run on NGA analyzer A extended, the general analysis time was approximately 200 seconds (up to *n*-dodecane, according to AN:5991-0257). The analysis speed, based on the BF2D configuration, is approximately 60 % faster than that of analyzer A extended. Table 3 shows the quantitation results and calorific value calculations from NGA analyzer A extended and the BF2D configuration. For high-concentration components, the difference in quantitation results is as small as 0.03 %. For C6/C6+ components, the quantitation differences are as low as 0.3 %, as shown in Table 3. By following ISO 6976 standard, the calculated calorific value difference based on NGA analyzer A extended and the BF2D configuration is negligible.

Table 3. C3-C5 hydrocarbon quantitation results and NGA calorific value based on NGA analyzer A extended and an 8 m CP-SIL 5CB BF2D channel.

Compound	ESTD concentration by NGA analyzer A extended	ESTD concentration by BF2D channel		Quantitation result comparison (%)	
Propane	6.0000	6.0056		99.91 %	
i-Butane	3.0005	3.0015		99.97 %	
n-Butane	2.0004	2.0012		99.96 %	
i-Pentane	0.50011	0.50024		99.97 %	
n-Pentane	0.49989	0.49982		100.01 %	
C6/C6+	0.10059	0.10025		100.34 %	
Calorific value	Unit	traditional		Backflush to detector	
		Dry	Saturated	Dry	Saturated
Compressibility		0.9968	0.9962	0.9968	0.9962
Water mole.	%		2.31		2.31
Mole mass	kg/kmol	21.2939	21.2182	21.2953	21.2195
Relative density, ideal		0.7352	0.7326	0.7353	0.7327
Relative density, real		0.7373	0.7351	0.7374	0.7352
Gas density, ideal	kg/m^3	0.8852	0.8821	0.8853	0.8821
Gas density, real	kg/m^3	0.8881	0.8854	0.8881	0.8855
Superior heating value (volume real)	MJ/m^3	44.06	43.07	44.07	43.07
Inferior heating value (volume real)	MJ/m^3	39.98	39.08	39.98	39.08
Superior heating value (volume ideal)	MJ/m^3	43.92	42.91	43.93	42.91
Inferior heating value (volume ideal)	MJ/m^3	39.85	38.93	39.85	38.93
Superior heating value (mass)	MJ/kg	49.62	48.64	49.62	48.64
Inferior heating value (mass)	MJ/kg	45.02	44.13	45.02	44.13
Superior heating value (molar)	kJ/mol	1,056.56	1,032.15	1,056.63	1,032.22
Inferior heating value (molar)	kJ/mol	958.58	936.44	958.64	936.5
Wobbe Index (real)	MJ/m^3	51.32	50.24	51.32	50.24
Wobbe Index inferior	MJ/m^3	46.56	45.58	46.56	45.58

Conclusion

A natural gas analyzer was developed based on a new type of CP-Sil 5CB backflush-to-detector channel coupled with a straight Hayesep channel. This is the fastest NGA analyzer based on a 490 Micro GC platform. It can provide total concentration for C6/C6+ components, and detailed information about air, methane, carbon dioxide, and C2–C5 compounds in NGA. It is a choice for customers who need a fast and reliable solution for NGA analysis.

Reference

1. Fast analysis of natural gas using the Agilent 490 micro GC natural gas analyzer, publication number 5991-0257.

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