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Application News

Gas Chromatograph Nexis™ GC-2030

Simplifying Multicomponent Quantitative Analysis of Organic Compounds with the Polyarc™ Microreactor for GC

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User Benefits

- The Polyarc system converts organic compounds eluting from the column into methane, enabling sensitivity to be normalized to carbon concentrations.
- Even for compounds containing heteroatoms or unsaturated bonds, which tend to show lower sensitivity with FID, the Polyarc reduces response differences.
- Because the response is normalized, fewer calibration curves are required even for multicomponent sample analysis.

■ Introduction

The Flame Ionization Detector (FID), the most widely used detector for GC, is used for quantitative analysis across a wide range of fields because it can detect almost all organic compounds.

However, FID can exhibit sensitivity differences depending on the compound type, such as compounds containing heteroatoms (e.g., oxygen or nitrogen) or those with unsaturated bonds (for details, refer to Application News No.01-01033-EN). As a result, conventional quantitative methods using calibration curves—including external standard methods— typically require a separate calibration curve for each target analyte.

This application introduces a case study analyzing a mixed solution of eleven compounds with different functional groups using the Polyarc, a GC microreactor. While compounds exhibit varying sensitivity with FID detection, the Polyarc system reduces sensitivity differences between compounds and facilitates quantitative analysis through simplified calibration curve creation.

■ Overview of the Polyarc

The Polyarc is a post-column microreactor installed between the column and the FID. The main unit is mounted on the GC's top panel. A schematic is shown in Fig. 1.

Organic compounds introduced into the Polyarc via the column are fully converted to methane in two steps—oxidation followed by reduction (Fig. 2)—and are ultimately detected by FID.

Because the FID detects methane, sensitivity is determined by the number of carbons, regardless of the original organic compound's functional groups. For example, 1 mol of 1-propanol (C_3H_8O) is detected as 3 mol of methane.

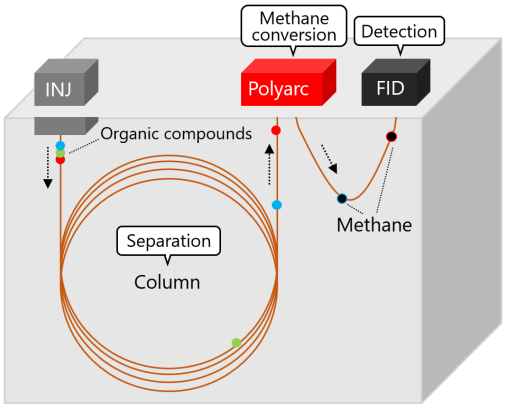

Fig. 1 Schematic of Polyarc

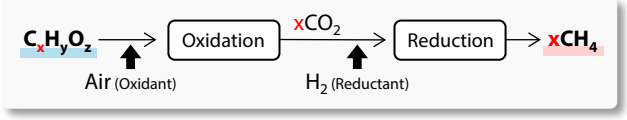

Fig. 2 Methane conversion reactions in Polyarc

■ Analytical Conditions

In this application, the sample was prepared by dissolving n-heptane, cyclohexane, toluene, o-xylene, methyl ethyl ketone (MEK), tetrahydrofuran (THF), *N*,*N*-dimethylformamide (DMF), butyl acetate, propylene glycol monomethyl ether acetate (PMA), pyridine, and 1,2-dichloroethane in methanol so that each compound was 500 ppm (v/v%).

Analytical conditions are shown in Table 1. The instrument used was a Nexis GC-2030 equipped with the Polyarc Ultra was used.(Fig. 3) Polyarc can be installed on both the Nexis GC-2030 and the Brevis™ GC-2050.

Additionally, in this application, N_2 was used as the carrier gas for affordability and safety, enabling cost-effective analysis.

Fig. 3. (Left) External view of Nexis™ GC-2030;
(Right) External view of the Polyarc™ system

Table 1 Analytical conditions

Model	:	Nexis GC-2030
Inj. Temperature	:	250 °C
Inj. Mode	:	Split 1:50
Carrier Gas	:	N_2
Line Velocity	:	25 cm/s
Column	:	SH-1 (60 m, 0.32 mm I.D., 1.00 µm) (P/N 221-75725-60)
Column Temperature	:	40 °C – 4 °C/min – 140 °C
Detector	:	FID
FID Temperature	:	300 °C
Makeup Gas Flow	:	24 mL/min
H_2 Flow	:	1.5 mL/min (FID only: 32 mL/min)
Air Flow	:	200 mL/min
Polyarc Temperature	:	450 °C
Polyarc H_2 Flow	:	35 mL/min
Polyarc Air Flow	:	2.5 mL/min

■ Results 1. Chromatogram

Fig. 4 shows the obtained chromatogram. When a Polyarc system is installed, eluting compounds pass through the Polyarc reactor, producing slightly broader peak widths. However, because the Polyarc reactor is designed with a microstructured architecture, the impact on separation is minimized. As shown in Fig. 4, good separation was confirmed in this analysis.

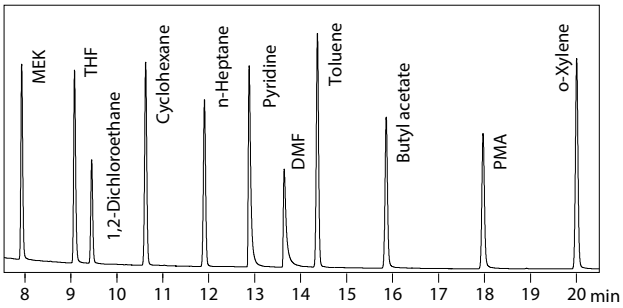

Fig. 4 Chromatogram obtained with Polyarc

■ Results 2. Comparison of Relative Response to FID

To verify the Polyarc's methane conversion effectiveness, the same analysis was performed without the Polyarc, using FID alone. Table 2 shows, for each case (FID and Polyarc), the average area values from five analyses and the relative response ratio (%) when the relative response* of n-heptane with FID is set to 100%. The relative response ratios are presented as a radar chart in Fig. 5.

With FID alone, reduced relative response is observed for compounds containing heteroatom functional groups, including DMF. In contrast, when the Polyarc is used, these compounds exhibit sensitivity comparable to that of hydrocarbons.

(*Relative response was calculated by dividing the average area value by the carbon molar concentration.)

Table 2 Average peak area values (N = 5) with FID and with Polyarc, and relative response ratios when the relative response of n-Heptane with FID alone is set to 100%

Compounds	Average peak area (N = 5)		Relative response ratio [%]	
	FID	Polyarc	FID	Polyarc
n-Heptane	118792	116139	100 (Standard)	98
Cyclohexane	141990	136720	103	99
Toluene	177174	168654	108	103
o-Xylene	180959	168597	110	102
MEK	95259	114982	86	103
THF	101794	122053	83	100
DMF	58251	96699	61	100
Butyl acetate	99299	117812	88	104
PMA	80047	111958	73	102
Pyridine	157947	157806	102	102
1,2-Dichloroethane	70588	63819	112	102

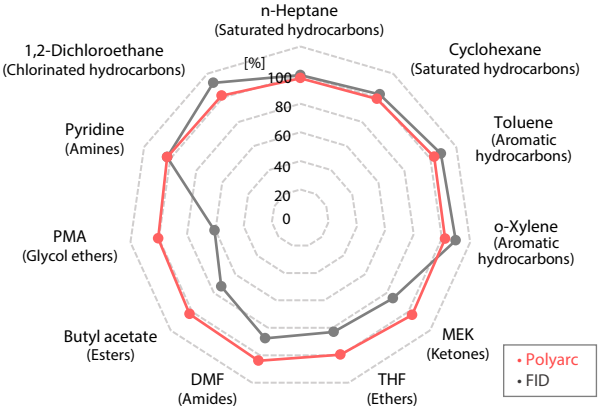

Fig. 5 Relative response ratios [%] with FID and with Polyarc

■ Results 3. Reproducibility

Peak area reproducibility (%RSD) for five consecutive analyses of the sample using the Polyarc is shown in Table 3. Even with Polyarc, high reproducibility (RSD < 1%) was confirmed.

Table 3 Peak area reproducibility with Polyarc (N = 5)

Compounds	Reproducibility (%RSD)
n-Heptane	0.491
Cyclohexane	0.526
Toluene	0.693
o-Xylene	0.746
MEK	0.761
THF	0.753
DMF	0.780
Butyl acetate	0.729
PMA	0.634
Pyridine	0.708
1,2-Dichloroethane	0.601

■ Quantitative Analysis Using Polyarc (Simplifying Calibration Curve Creation)

The procedure for quantitation using a Polyarc to simplify calibration curves creation is introduced below. When a Polyarc is used, sensitivity is normalized to carbon concentration; therefore, it is not necessary to prepare a separate calibration curve for each compound.

Fig. 6 shows, for each compound, a plot with carbon molar concentration [Cmol/L] on the x-axis and average area value on the y-axis. In this application, the carbon molar concentration was calculated as the amount of carbon [mol] in a given compound per unit sample volume. With Polyarc, as shown in Fig. 6, the plot of area values versus carbon molar concentration falls on a single straight line regardless of the compound.

From this, by analyzing at least one compound of known concentration and creating a calibration line, as in Fig. 6, it is possible to calculate the molar carbon concentration of components with unknown concentration from the measured peak area, and thus determine their solution concentrations.

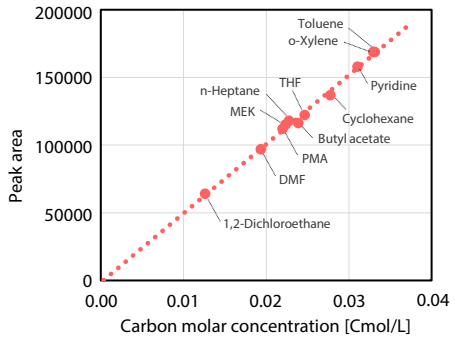

Fig. 6 Plot of carbon molar concentration vs. average peak area with Polyarc

Below is a concrete example of quantitation using the Polyarc. In this application, the sample was prepared with all eleven compounds at 500 ppm; however, for demonstration purposes, only o-xylene is assumed to have a known concentration (500 ppm), and the concentrations of the other ten compounds are treated as unknown. Accordingly, o-xylene is used as the internal standard (IS in the equation below), and the remaining 10 compounds are quantified. Because the Polyarc normalizes the detector response (peak area) to carbon concentration, the concentration of an unknown compound in solution can be calculated using the following equation when an internal standard of known concentration is included.

$$C_A = C_S * \frac{Area_A}{Area_S} * \frac{MW_A}{MW_S} * \frac{d_S}{d_A} * \frac{\#C_S}{\#C_A}$$

(C_A: Mass concentration of unknown sample, C_S: Mass concentration of IS,
$Area_A$: Peak area of unknown sample, $Area_S$: Peak area of IS,
MW_A : Molecular weight of unknown sample, MW_S : Molecular weight of IS,
d_A : Density of unknown sample, d_S : Density of IS,
$\#C_A$: Carbon number of unknown sample, $\#C_S$: Carbon number of IS)

Quantitation results for the ten compounds of unknown concentration, obtained from the equation, are shown in Table 4. Although the actual concentration of each compound is 500 ppm, all quantitative errors relative to 500 ppm were within ±5%, demonstrating good performance.

Table 4 Quantitation results when o-xylene is used as the internal standard

Compounds	Concentration [ppm]	Quantitative error [%]
n-Heptane	478	- 4.44
Cyclohexane	484	- 3.16
Toluene	503	+0.63
o-Xylene (Internal standard)	-	-
MEK	506	+1.23
THF	487	- 2.70
DMF	491	- 1.87
Butyl acetate	508	+1.68
PMA	501	+0.09
Pyridine	500	- 0.04
1,2-Dichloroethane	497	- 0.67

Thus, using the Polyarc enables simple multicomponent quantitation by adding an internal standard without the need to prepare calibration curves for each compound. Although one internal standard was used in this application, preparing multiple internal standards can further improve quantitation accuracy.

Note that for compounds for which standard samples cannot be obtained (i.e., compounds that cannot be precisely quantified), quantitation values obtained using a Polyarc should be treated as reference values only.

■ Conclusion

Analyzing multicomponent samples with a Polyarc confirmed that sensitivity differences due to functional groups can be reduced. Compounds containing heteroatoms or unsaturated bonds can be analyzed with relative response comparable to hydrocarbons when a Polyarc is used.

Peak area reproducibility was excellent, with %RSD < 1.

Additionally, it was confirmed that analyzing at least one compound of known concentration enables quantitation of compounds with unknown concentration—i.e., calibration curve preparation can be omitted.

Table 5 summarizes the benefits of using a Polyarc system. The Polyarc simplifies multicomponent quantitative analysis and enables high-sensitivity analysis of compounds that are poorly detected by FID.

Note that for certain compounds, adsorption in the inlet or similar effects may prevent achieving a relative response equivalent to that of saturated hydrocarbons.

<Related Applications>

1. Analysis of Ethanol in E10 Gasoline Using Polyarc™ Microreactor for GC Application News No.01-01033-EN

Table 5 Benefits of using a Polyarc

Cases	Benefits of using a Polyarc
Analyzing multicomponent samples and needing to prepare multiple calibration curves	Quantitation can be performed with a single calibration curve (or with fewer calibration curves)
Already reducing the number of calibration curves by using "XX-equivalent" quantitation	Quantitation accuracy is improved
Qualitative and quantitative analysis of compounds containing heteroatoms such as oxygen and nitrogen is challenging	Sensitivity differences due to functional groups are eliminated, improving sensitivity
Need to analyze formic acid, formaldehyde, CO, and CO_2 with GC-FID	Converted to methane, enabling detection by FID
Target compounds require quantitation, but standard materials for calibration curve preparation are unavailable (or are expensive)	Quantitation can be performed using any readily available standard compound

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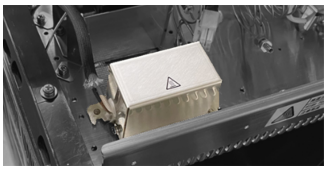

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A Microreactor for Gas Chromatography

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