

High-Speed Analysis of Total Petroleum Hydrocarbons (TPH) using Brevis GC-2050

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User Benefits

- ◆ The compact model, Brevis GC-2050 (universal power supply: 230 V) enables a high-speed temperature rise. It is possible to reduce analysis time by using Brevis GC-2050 and hydrogen gas.
- ◆ Brevis GC-2050 and dual injectors save space and provides high productivity.

Introduction

Total petroleum hydrocarbons (TPH) refers to mixtures of various types of hydrocarbons. In the production of petroleum hydrocarbon products, there are concerns about environmental pollution caused by TPH remaining in water and soil. There are also health risks, so it is very important to control the concentration of TPHs.

ISO 9377-2-2000 describes a method of analysis using solvent extraction and gas chromatography. In this method, the separation of C10 from extraction solvent and elution of C40, while keeping the relative response(peak area) of n-tetracontane (C40) to n-eicosane (C20) at least 0.8, are required. For that reason, it is necessary to lower the initial temperature and increase the thickness of the column, but the analysis time takes about 20 to 30 minutes.

In the tests described here, the initial temperature was not lowered, and a column with a thick film and a hydrogen carrier were used. Brevis GC-2050 supports universal power supply, and the device is prepared with a wiring configuration for a 230 V power supply voltage to achieve high-speed temperature increase, realizing high-speed analysis within 6 minutes.

This article introduces an example of the high-speed analysis of TPH using hydrogen as the carrier gas. A system performance test and the linearity of the calibration curve was checked, based on ISO 9377-2-2000.

* Detailed information regarding analysis of TPH is described in Application News 01-00355-JP, "High-Speed Analysis of Total Petroleum Hydrocarbons (TPH)." Please also refer to this.



Fig. 1 Brevis™ GC-2050 (Left) and Dual Injectors (Right)

Sample Preparation

The samples (1) to (3) were prepared as follows.

- (1) Extraction Solvent/Background Correction Sample
Heptane was prepared.
- (2) System Performance test Sample
50 µg/mL alkane standard mixture (Sigma-Aldrich, P/N: 94234-2ML) was prepared.
- (3) Calibration Curve Samples (Mineral Oil)
A diluting solvent was prepared by dissolving 2.0 mg of n-tetracontane and 2.0 µL n-decane in 100 mL of heptane. Using the prepared diluting solvent, a 5,000 µg/mL QC standard solution (Sigma-Aldrich, P/N: 51706-1ML) was diluted to prepare 50, 100, 250, 500, and 1000 µg/mL calibration curve samples.

Analytical Conditions

The Brevis GC-2050 (Universal power supply: 230 V) was used, with two analysis lines and dual AOC™-30i. A standard insert was used for the injection port.

Hydrogen created by Precision H2 SL was used as carrier gas and flame ionization detector combustion gas.

Table 1 shows the analytical conditions.

Table 1 Analytical Conditions	
Model:	Brevis GC-2030 (230 V)/ AOC-30i × 2
Syringe:	Xtra Life Microsyringe (P/N 227-35400-01)
Injection Mode:	Sampler Navigator – Standard mode
Injection Volume:	1 µL
Injection Temp.:	280 °C
Injection Mode:	Split
Split Ratio:	1:5
Carrier Gas:	H ₂
Carrier Gas Control:	Column flow (7.00 mL/min)
Column:	SH-I-1-1MS (P/N: 227-36004-03) (12 m × 0.2 mm I.D. × 0.33 µm)
Column Temp.:	100 °C (0.3 min) - 65 °C/min - 175 °C - 45 °C/min - 300 °C - 35 °C/min - 340 °C (0.5 min)*1
Detector:	Hydrogen flame ionization detector (FID)
Detector Temp.:	350 °C
Detector Gas:	H ₂ 32 mL/min, Air 200 mL/min
Makeup Gas:	N ₂ 24 mL/min

System Performance Test

A system performance test was conducted using the 50 µg/mL alkane standard mixture.

The chromatogram is shown in Fig. 2, and the area repeatability and average area ratio (C40/C20) are shown in Table 2.

The area ratio (C40/C20) was over 0.94, satisfying the standard requirement.

C10 eluted in 0.4 min and separation from the extraction solvent was good.

Table 2 C20 and C40 Area Repeatability and Average Area Ratio (n = 3)

Line	C20 Area Repeatability (%RSD)	C40 Area Repeatability (%RSD)	C40/C20 Average Area Ratio
1	0.16	0.49	0.91
2	0.26	0.23	0.95

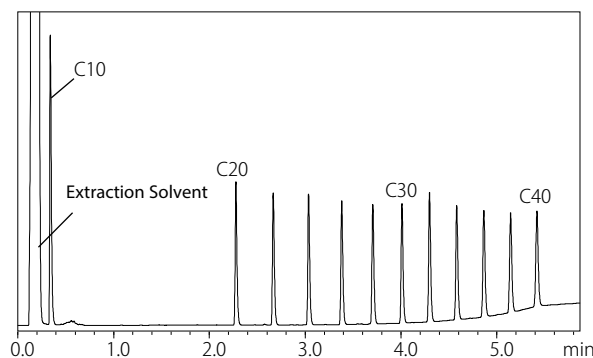


Fig. 2 Chromatogram of alkane standard mixture

Calibration Curve

Fig. 3 shows the chromatograms of QC standard solutions. The area for mineral oil was calculated by totaling the peak areas for all the components detected between C10 and C40.

For details on data processing parameters, please refer to Application News 01-00355.

The average area and area repeatability for mineral oil are shown in Table 3. The calibration curves are shown in Fig. 4. The calibration curve linearity R^2 was over 0.9998, so good results were obtained.

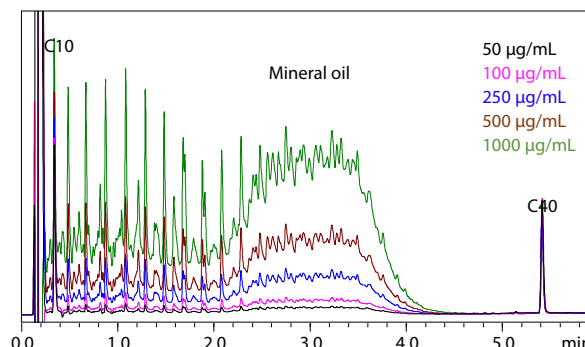
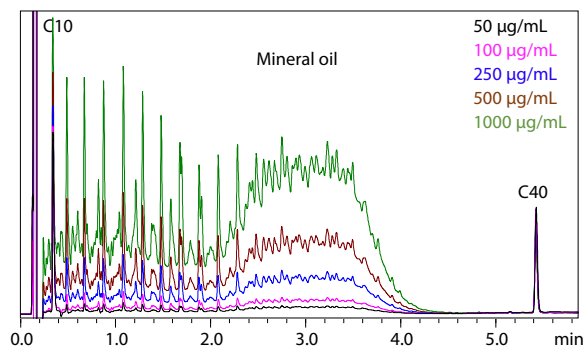


Fig. 3 Chromatograms of QC Standard Solutions (Left: Line 1, Right: Line 2)

Table 3 Average Area and Area Repeatability of Mineral Oil (n = 3, Left: Line 1, Right: Line 2)

No.	Concentration (µg/mL)	Average Area for Mineral Oil	Area Repeatability (RSD%)
1	50	411,037	1.02
2	100	793,609	0.45
3	250	2,098,153	0.45
4	500	4,163,042	0.13
5	1000	8,375,165	0.12

No.	Concentration (µg/mL)	Average Area for Mineral Oil	Area Repeatability (RSD%)
1	50	433,517	1.09
2	100	832,573	0.19
3	250	2,183,440	0.15
4	500	4,266,727	0.16
5	1000	8,783,025	0.40

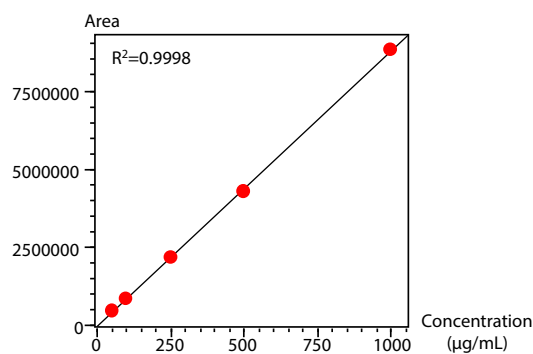
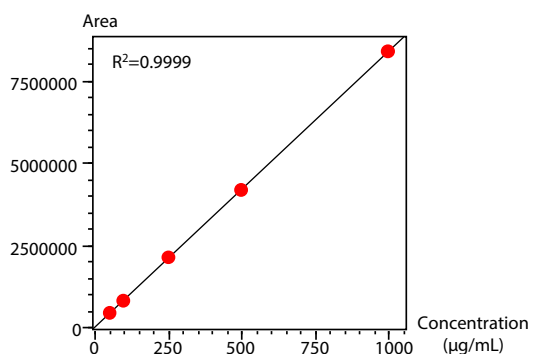


Fig. 4 Calibration Curve of Mineral Oil (Left: Line 1, Right: Line 2)

■ Compact Hydrogen Generator -Precision SL 200-

FID uses hydrogen gas as combustion gas. However, in the case of cylinders, it takes cost and effort of maintenance because of high pressure combustible gas.

Precision H2 SL Series produce hydrogen by electrolyzing water. It supply 99.9995% pure hydrogen up to 200 mL/min. It is lightweight and compact, and the amount of storage is smaller than cylinders so that it is safer.

In this study, it took 45 mL/min x 2 for hydrogen carrier gas and 32 mL/min x 2 for FID hydrogen gas, a total of 154 mL/min. Therefore, the gas supply was within the maximum hydrogen flow rate of the Precision H2 SL200 model.



Fig. 5 Precision H2 SL200

■ Conclusion

In this article, high-speed TPH analysis was achieved using the Brevis GC-2050 (230 V) with an oven insert to enable a high-speed temperature rise, and hydrogen as the carrier gas.

The analysis time was within 6.0 min. In the system performance test, the area ratio (C40/C20) was over 0.91, satisfying the standard requirement. The calibration curve linearity R^2 was over 0.9998, so good results were obtained.

TPH analysis by the flagship model Nexis™ GC-2030 reported in 01-00355-EN is even faster than compact model Brevis GC-2050.

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