

Application News

Gas Chromatograph

3.5 min Total Petroleum Hydrocarbons (TPH) Analysis Using the Nexis™ GC-2030 and H₂ Carrier

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User Benefits

- ◆ Analysis times can be significantly shortened by using the Nexis GC-2030 (230 V), an oven insert, and hydrogen as the carrier gas.
- ◆ Using dual injectors, throughput can be greatly improved, enabling the analysis of more than 400 samples per day.

1. Introduction

Shimadzu's Nexis GC-2030 can analyse over 400 samples in a single day, due to a highly optimised method that has an injection-to-injection time of less than 7 minutes per sample, or 3.5 minutes per sample, with dual injectors running simultaneously.

This method can resolve C₁₀ to C₄₀ alkanes, allowing it to be applied to almost all TPH/TRH applications. C₆ to C₄₀ can be resolved with a modified method (Figure 1).

During the production and use of petroleum hydrocarbon products, the environment, including waters and soils can be contaminated.

Petroleum hydrocarbons do not, in general, biodegrade readily. Furthermore, some pose risks to health, consequently, the control of contamination is important. The analysis of petroleum products in waters and soils is commonly referred to as TPH (Total Petroleum Hydrocarbon) or TRH (Total Recoverable Hydrocarbon), which covers a range of standardised method variants.

Some classes of petroleum products pose greater risks than others. Hence, reporting the total concentration of TPH does not provide a reliable basis for evaluating the potential risks to the environment and human health.

For this reason, the chromatographic separation is divided into bands and resolves the analytes based on carbon atom (C) numbers.

Table 1: Common TPH/TRH bands

Hydrocarbon bands
C ₁₀ -C ₁₄
C ₁₅ -C ₂₈
C ₂₉ -C ₃₆
>C ₁₀ -C ₁₆
>C ₁₆ -C ₃₄
>C ₃₄ -C ₄₀

2. Sample Introduction

The analysis of volatile hydrocarbons below C₁₀ is typically performed using Headspace or Purge & Trap techniques, as these are often difficult to resolve from the solvent. The higher molecular weight hydrocarbons are analysed using liquid injection. Most testing laboratories analyse samples up to C₄₀, which covers a wide range of boiling points. Conventionally, this would require an on-column injector (OCI) to ensure non-discriminatory sample introduction. However, a recent application note (SCA_180_021) demonstrated that Shimadzu's

split/splitless inlet (SPL-2030) has negligible discrimination. For instance, the area ratio of C₄₀ to C₂₀ is better than 0.96 (but less than 1).

3. Optimisation of method speed

Traditionally, TPH columns are selected with a thinner film thickness to reduce elution times of the high-boiling components. This often impacts the resolution of the low boilers from the solvent. Consequently, the GC oven starting temperature is typically low (30-35 °C), resulting in extremely long cool-down times that greatly limit sample throughput.

The Rxi-1ms column from Restek, with non-conventional dimensions (Table 2), was selected to improve sample throughput. This allows for separation of the volatile components from the solvent at a higher starting temperature and reduces retention of the high-boiling hydrocarbons up to C₄₀ (Figure 2).

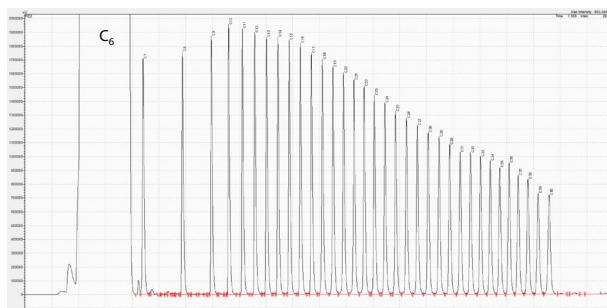


Figure 1: n-Alkane mix from C₆-C₄₀

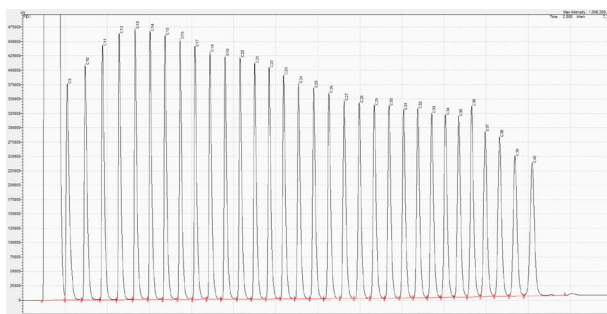


Figure 2: n-Alkane mix from C₉-C₄₀

The use of hydrogen as the carrier gas also makes it possible to achieve faster analysis and maintain good chromatographic resolution. Thus, run-times can be significantly reduced. Furthermore, the recent increase in helium prices due to its availability, make methods with hydrogen carrier gas more popular and commercially viable.

The throughput can be further increased by using Shimadzu's Nexis GC-2030 dual injection mode, with two AOC-30 liquid autosamplers.

Table 2. Method parameters for ultra-fast GC

Components	Settings
SPL temperature	320 °C
Flow rate	7 mL/min ramped to 10 mL/min
Column	Rxi-1ms (Restek) 12 m x 0.2 mm x 0.33 µm
Column oven program	100 °C, 0.3 min, then maximum ramp rate to 340 °C.
FID temperature	350 °C

A column oven volume reducing pillow from Restek was used to improve both oven heating and cooling rates.



4. Results

A series of six calibration standards were analysed using the optimised method parameters (Table 2). The standards were made to cover a total hydrocarbon content concentration range from 32 ppm to 3200 ppm.

Calibration curves were acquired for $C_{10} - C_{14}$, $C_{15} - C_{28}$,

$C_{29} - C_{36}$, $>C_{10} - C_{16}$, $>C_{16} - C_{34}$, $>C_{34} - C_{40}$.

All bands gave a linear regression coefficient greater than 0.998 (Figure 3).

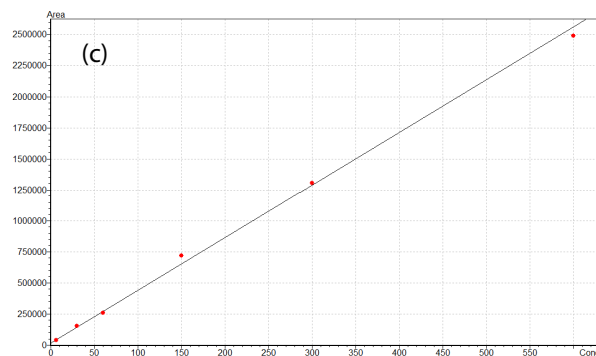
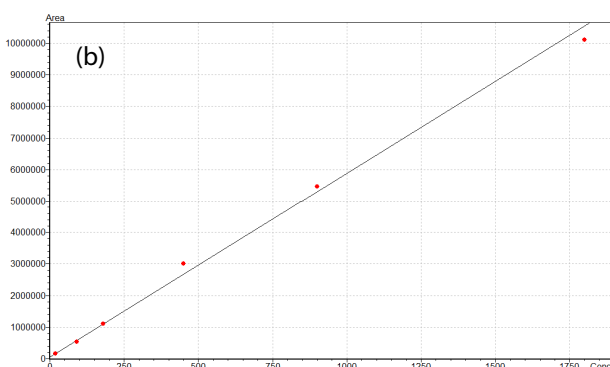
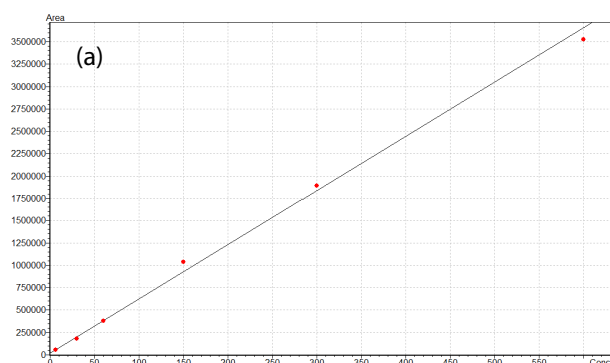


Figure 3: Calibration curves for the total TPH group ($>C_{10} - C_{16}$) [$R^2 = 0.998$] (a), $>C_{16} - C_{34}$ [$R^2 = 0.998$] (b), and $>C_{34} - C_{40}$ [$R^2 = 0.998$] (c).

The GC oven program was set to 3.5 minutes to allow for any heavy components $>C_{40}$ (retention time = 2.9 min) to elute. With a single line system, this gives an injection-to-injection time of 7 minutes.

Samples from various matrices, including water and soil were measured. The sample chromatogram shown in Figure 4 was obtained from the analysis of 1% w/v diesel + 1% w/v motor oil solution.

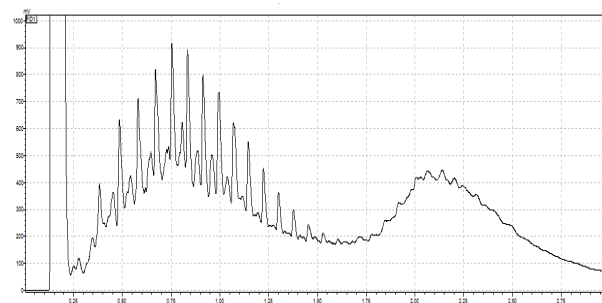


Figure 4: Chromatogram of a 1% w/v diesel + 1% w/v motor oil solution

5. Conclusion

Shimadzu's GC-2030 with split/splitless inlet and FID offers an ideal solution for TPH and TRH analysis across the widest boiling point range.

The GC-2030NX's high-performance oven and the use of hydrogen carrier gas facilitate a very short run time. The combination of this, with a dual line configuration, results in injection-to-injection run times of below 3.5 minutes per sample.

Over a 24-hour period, a single GC-2030 instrument is able to analyse over 200 samples.

With dual FID-2030s fitted, a single GC-2030 can analyse over 400 samples in a 24-hour period

The LabSolutions™ GC software's highly flexible integration parameters allow various baselines and bandings to be performed automatically. This offers easy conversion from existing Chromatography data systems (CDS).

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