

MINERAL OIL/TPH

Technology Advantage: Agilent Intuvo 9000 GC with FID



Introduction

The analysis of hydrocarbon contaminants in environmental samples is commonly called TPH, or total petroleum hydrocarbon determination. For TPH analysis, chromatographic separation of individual compounds is not required. Instead the entire sample can elute as a largely unresolved mass for quantification. This fact permits one to use Ultra-Fast GC techniques; namely, short columns, fast temperature program ramps, and high carrier gas flow rates. The short run times typical of Ultra-Fast GC allows many more analyses in a typical workday, an important consideration for labs struggling to keep up with large sample backlogs. In addition to the ability to perform ultra-fast GC, the instrument must provide a very high degree of reliability.

The Agilent Intuvo 9000 GC is designed to enable Ultra-Fast GC separations with a high degree of precision and reliability

- An innovative direct column heating technology allows fast heating and cool down.
- A new electronic pneumatics controller (EPC) provides consistent high column flow during fast heating.

Experimental

Sample Preparation

For this work a Certified Reference Material (BAM-U021) composed of TPH contaminated soil was obtained from the Bundesanstalt für Materialforschung und prüfung in Germany. The sample was certified by consensus analysis to contain 3,560 mg/kg of mineral oil hydrocarbons with an uncertainty of 260 mg/kg. Prior to GC analysis, the sample was prepared in duplicate using the extraction and cleanup procedure described in ISO Method 16703¹.

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GC Instrument Conditions

Parameter	Value
Inlet	Splitless 350 °C
Injection volume	0.5 µL
Column	Agilent Intuvo DB-1HT, 5 m × 0.32 mm, 0.1 µm
Constant column flow	10 mL/min helium
Column temperature	40 °C for 0.5 minutes 250 °C/min to 260 °C 6 °C/min to 350 °C hold 1.3 minutes
Detector	Flame ionization 350 °C

Results and Discussion

Five injections of each BAM-U021 duplicate sample were run on the Agilent Intuvo 9000 GC. The figure below shows an overlay of a single run from each duplicate sample. Each analysis is completed in about 3 minutes using Ultra-Fast GC conditions. The C10 and C44 peaks are added to the sample to serve as integration markers for the total TPH sample response. Each run has nearly identical chromatographic response and retention time. Table 2 shows the quantitative results.

Three analysis performance measures are illustrated with this data. First, the method accuracy is shown by comparing the mean results to the BAM certified values. Each duplicate analysis agrees with the 3,560 mg/kg certified value. Next, the instrument precision is shown by the RSDs calculated for each duplicate. Five runs of each duplicate sample have a quantitate RSD of less than 1 %. Finally, the single lab method precision is measured using the single lab repeatability (r) test described in the ISO 16703 method. The experimental repeatability of 59 mg/kg is well below the required value of 139 mg/kg.

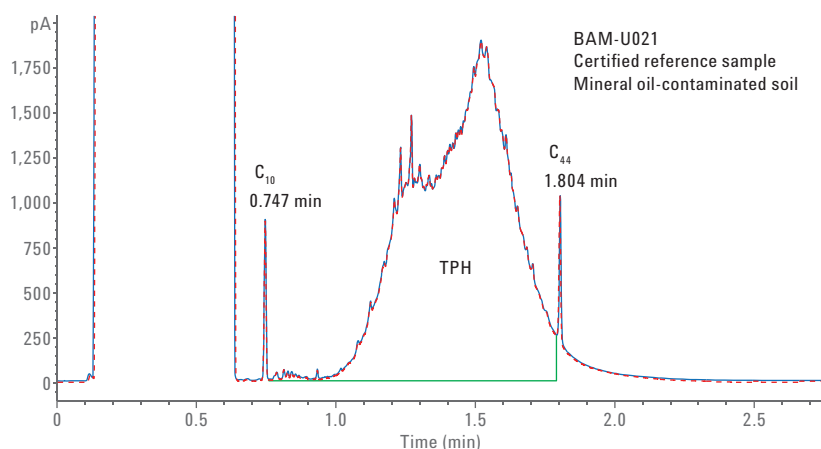


Figure 1. Overlay of duplicate TPH soil extracts.

Table 2. Repeatability for duplicate analyses of TPH soil extracts.

Run	U021 A (mg/kg)	U021 B (mg/kg)
1	3,462	3,480
2	3,487	3,485
3	3,502	3,482
4	3,513	3,479
5	3,538	3,492
Mean	3,500	3,484
Cert. value	3,560 ± 260	
Std Dev	28.547	5.234
RSD	0.82 %	0.15 %
r (exp)	59	
r* (ref)	136	

Conclusion

An Ultrafast GC method was developed for the analysis of TPH in environmental samples. The Agilent Intuvo 9000 GC has the instrument performance to deliver rapid column heating, rapid cool down, and high flow rates using Agilent Intuvo GC columns to run this method with a high degree of method accuracy and precision.

Reference

1. Soil Quality-Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography. ISO 16703:2004(E). Geneva, Switzerland: ISO.

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