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Gas Chromatography Analytical Methods for Teaching Laboratories

Analysis and comparison of different fuels/oils using gas chromatography with flame ionization detection.

Introduction

The analysis of Total Petroleum Hydrocarbons (TPH) is one of the key methodologies employed by Environmental Testing Laboratories around the world. The term TPH covers a wide range of petroleum-based products that are of concern due to the environmental pollution caused by these chemicals remaining in soil and water and potential adverse health effects.

This application utilizes a GC with a standard inlet and flame ionization detector to analyze one or more commercially available petroleum-based products. The sample data can then be divided into hydrocarbon bands from which conclusions may be drawn on the nature of the sample.

Reagents

Hexane – analytical grade

n-Alkane marker standard – Shimadzu p/n 980-01161 or suitable equivalent.

Sample material – any commercially petroleum-based product such as petrol, diesel, kerosene, lubricating oil.

Safety

Lab coats, safety glasses and gloves should be worn when handling reagents.

Follow all appropriate safe operating procedures and beware of any specific actions raised in the associated risk assessments.

Apparatus

- Gas chromatograph fitted with a split/splitless injection port and flame ionization detector (FID).
- Autoinjector and autosampler capable of handling liquid samples in 1.5 mL vials
- SH-I-1MS column, 12 m x 0.2 mm I.D. x 0.33 µm, p/n 227-36004-03 (or suitable equivalent).
- 1.5 mL autosampler vials and caps.
- Appropriate laboratory glassware for the preparation of the standard and samples.

Instrument Conditions

GC Parameters	
Injections Temperature	280°C
Injection Mode	Split
Split Ratio	1:5
Purge flow	3 mL/min
Column	SH-I-1MS 12 m x 0.2 mm I.D. x 0.33 µm film thickness
Oven Program	100°C initial temperature, hold 0.3 minutes 65°C/min to 175°C, 45°C/min to 300°C, 35°C/min to 340°C and hold for 0.5 minutes
Run Time	50.0 minutes
Carrier Gas	Hydrogen, constant flow 7.00 mL/min
Injection Volume	1.0 µL
FID Temperature	350°C
Hydrogen Flow rate	32 mL/min
Air flow rate	200 mL/min
Make up gas (N_2) flow rate	24 mL/min
Sampling Rate	40 msec

Preparation of n-Alkane standard

Dilute the stock standard 1 in 10 with hexane in a volumetric flask. Then transfer an aliquot to a 1.5 mL vial for analysis.

Preparation of test sample

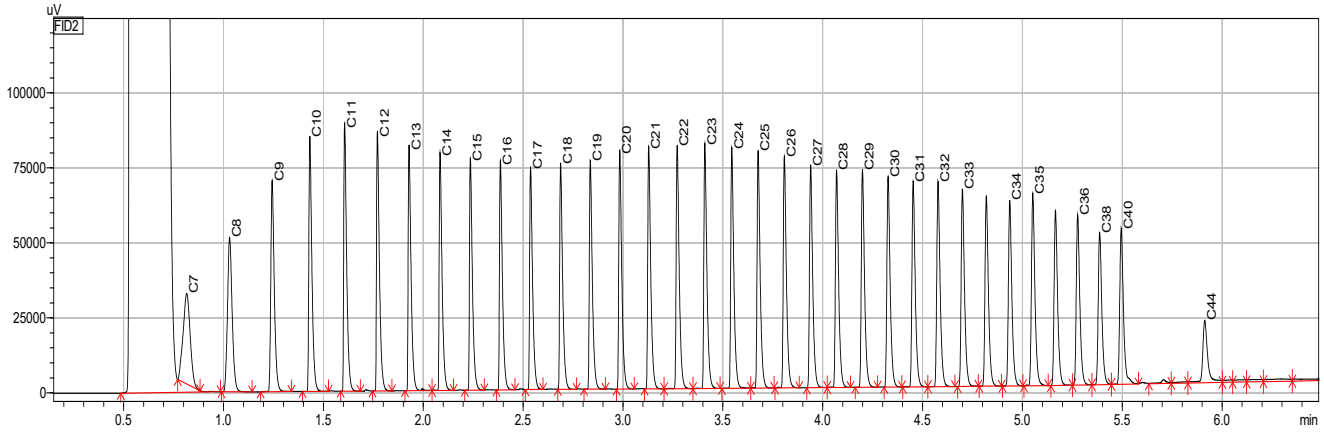
Dilute the sample in hexane to give a final concentration of approximately 50 mg/L.

Interpretation of results

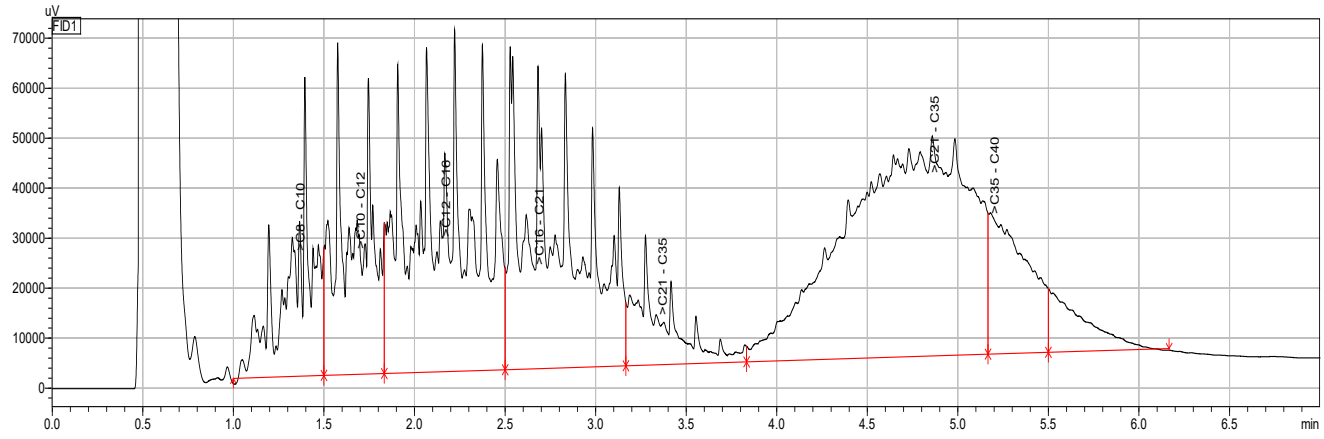
Petroleum based products can contain thousands of individual compounds, to identify and report all of these separately would involve a lot of work. Instead, the results from the TPH analysis are divided into carbon bands, e.g. >C10 to C14. The distribution of compounds in these bands will give information on the type of product in the sample.

The n-Alkane marker standard contains alkanes of known carbon number from C7 to C44, see image below. Note: the standard and sample are diluted in hexane (C6), and this will appear as a large peak at the start of the analysis.

From these the retention times for the start and end of each carbon band can be determined.



A processing method can now be made to divide the test sample into the required carbon bands. The whole chromatogram should be treated as one ‘peak’ and the split peak function used to divide it at the required retention times as determined from the n-Alkane standard, see example below.



The type of product can be determined from the distribution of compounds in each band. The table below gives some typical carbon ranges for petroleum-based products.

Product	Carbon Range
Petrol	>C6 to C10
Kerosene	>C9 to C16
Diesel	>C11 to C20
Mineral/lubricating oil	>C15 to C44

The chromatogram for each petroleum-based product is like a fingerprint, unique to that product. A library of product chromatograms could be created to aid identification of test samples through direct comparison.

Key learning outcomes

- How complex sample data can be processed.
- How to interpret data and draw conclusions on the type of sample analyzed.

Notes

The instrument conditions are based on the Shimadzu application note ‘High-Speed Analysis of Total Petroleum Hydrocarbons (TPH) using Brevis GC-2050’. If an alternative column or carrier gas is used, these conditions will need to be adapted appropriately.