

Gas Chromatography

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Analysis of Petroleum Hydrocarbons C10-C40 in Water, Soil and Waste with GC-FID

Introduction

Total Petroleum Hydrocarbons (TPH) is a term used to describe a large family of compounds

originating from crude oil and refined petroleum products, whose composition depends on the source and the process in which they are involved.

For the determination of Petroleum Hydrocarbons in the range of C10-C40 in the different matrices (water, soil, waste), three standards methods are taken as a reference. Each one specifies a method for the determination of the hydrocarbon by means of solvent extraction followed by the analysis by Gas Chromatography (GC) with Flame Ionization Detector (FID).

EN ISO 9377-2:2000 (Water, conc. > 0.1 mg/l)¹

EN ISO 14039:2004 (Waste, conc. between 100 mg/Kg and 10.000 mg/Kg)²

ISO 16703:2011 (Soil, conc. between 100 mg/Kg and 10.000 mg/Kg)³

The Hydrocarbon oil index (water) or Hydrocarbon content (waste and soils) is the sum of the concentrations of long-chain and branched aliphatic, alicyclic, aromatic and alkyl-substituted aromatic hydrocarbons present in the sample. Hydrocarbons are defined as all the compounds extractable with a nonpolar solvent (boiling point between 36 °C and 69 °C), not adsorbed on Florisil and having GC retention times between those of n-decane (C10) and n-tetracontane (C40).

Experimental

As a chromatographic system, the PerkinElmer Clarus® GC was used, equipped with a Programmable Split Splitless (PSS) injector that can be programmed in temperature, flow, pressure and velocity. The injector liner was a 2 mm Siltek™ liner with deactivated wool (P/N N6502001). The detector used was a Flame Ionization Detector (FID). The GC was equipped with a built-in liquid autosampler with a 50 µl syringe that provide the possibility to use the Large Volume Injection (LVI) technique. An Elite-5HT column 15 m x 0.32 mm x 0.1 µm (P/N N9316274) was used obtaining a good chromatographic profile for Hydrocarbons. Helium was used as carrier gas.

The experimental conditions for this analysis are given in Table 1 below:

Table 1. Experimental conditions.

Injection Volume	20 µl	
Oven	50 °C for 3.5 min, 30 °C/min ramp to 325 °C then isotherm for 7.33 min (total run time 20 min)	
Injector	50 °C for 0.5 min, 999 °C/min ramp to 325 °C for 999 min	
Carrier He	1 mL/min	Split 120 mL/min
Detector	320 °C	Air 450 mL/min; Hydrogen 45 mL/min; Range 1; Attenuation -6
Timed Event	SPL1 value 0 mL/min at 0.5 min SPL1 value 120 mL/min at 3 min	

Standard and sample preparation were made accordingly to EN and ISO methods.

Results

Figure 1 shows the linear calibration curve for Hydrocarbons C10-C40 starting from a standard concentration of 0.0177 mg/mL to 0.354 mg/mL (three replicates for each calibration point).

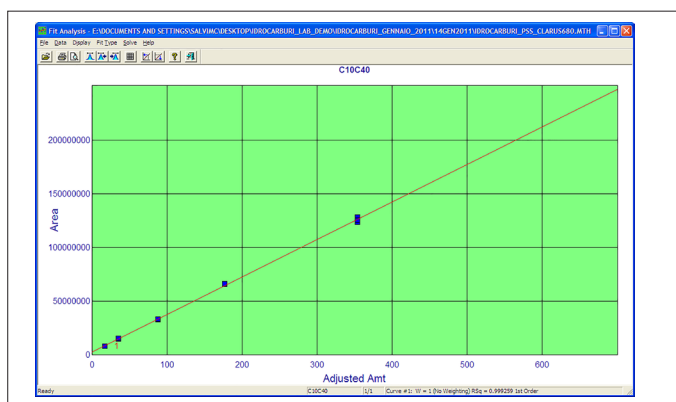


Figure 1. Five-level linear calibration curve for Hydrocarbons C10-C40.

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Figure 2 Shows a chromatogram of a standard solution made by Oil Type A (diesel fuel) plus Oil Type B (Mineral oil) diluted with Retention-time window standard solution (RTW, C10 and C40).

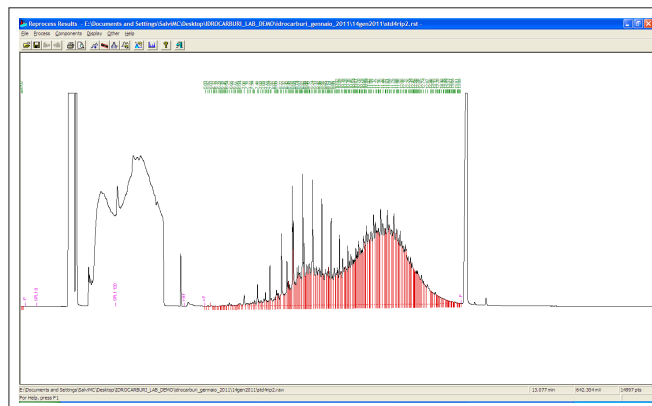


Figure 2. Chromatogram of a standard solution.

Conclusion

Method EN ISO 9377-2:2000 for water establishes a minimum limit of 0.1 mg/L using a sample more or less 1000 times concentrated during sample preparation. The lowest concentration standard reported in EN ISO 9377 method is 0.2 mg/mL.

With the PSS injector and Large Volume Injection (LVI) technique used in this Application Note, it is possible to reach an instrumental limit of quantitation lower than 0.010 mg/mL. The sensitivity of the analysis using LVI, permits us to concentrate the sample only 50-100 times instead of 1000 times. It is therefore possible to reduce the analysis time and the volume of the sample extracted, concentrating 50-100 mL to 1 mL.

References

1. Water quality - Determination of Hydrocarbon oil index. Method using solvent extraction and gas chromatography.
EN ISO 9377-2:2000
2. Characterization of waste - Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography.
EN ISO 14039:2004
3. Soil quality - Determination of content of hydrocarbon in the range C10 to C40 by gas chromatography.
ISO 16703:2011



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