

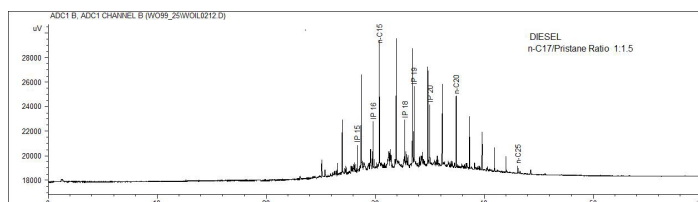
Environmental Total Petroleum Hydrocarbon (TPH) Analyser

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Abstract

Total petroleum hydrocarbons (TPH) is a term used to describe a large family of several hundred chemical compounds that originally come from crude oil. The typical range from C5 – C40 consist of fractions and classes. The fractions range from petrol, through kerosene, diesel to oils, jet fuel and crude. The main classes of compounds present are saturated and unsaturated aliphatics and aromatics containing carbon and hydrogen. However there are lower concentrations of other classes containing oxygen such as FAMES (fatty acid methyl esters) from biofuels. From an environmental perspective not only total petroleum measurement is a requirement, but the compound classes and sometimes the individual components need reporting. This is due to the toxicity differences between the classes and individual compounds. In addition it is also very useful to know what fraction of petroleum is present, in the case of land contamination and how long it has been present.

Typical Diesel Chromatogram



The current methodology to analyse for waters and soils for TPH involves extraction processes such as solvent/ Soxhlet or microwave digestion with various clean up steps possible, such as SPE. Subsequent analysis is by GC/FID with headspace, GC/FID, GC/MS or even GC/MS/MS to analyse individual PAH's. Then a manual assessment of the chromatograms is performed or an Excel macro used to distinguish between petrol, kerosene and differences between weathered or biodegraded products such as diesel. The whole process is a time consuming exercise with numerous sources of errors.

Introduction

JSB's challenge was to minimise the sample clean up and provide more accurate and reliable TPH results. The five critical challenging areas were:

- Reduce sample preparation steps
- Minimise the number of methods for TPH analysis
- Visually display and report more accurate TPH data
- Visually display and report of banding, fractions, classes of compounds and individual compounds
- **UKAS & MCERTS** Accreditation

Instrumentation & Software

JSB's customised gas chromatograph (based on Agilent 7890) fitted with 7693A auto sampler, split/splitless injector and Flame Ionisation Detector (FID). An Agilent Flow Modulator was used to control the instrumentation and GC image software to analyse and display the 2D images and data.



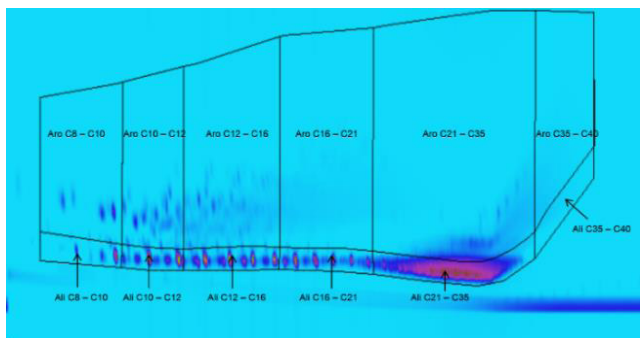
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Experimental

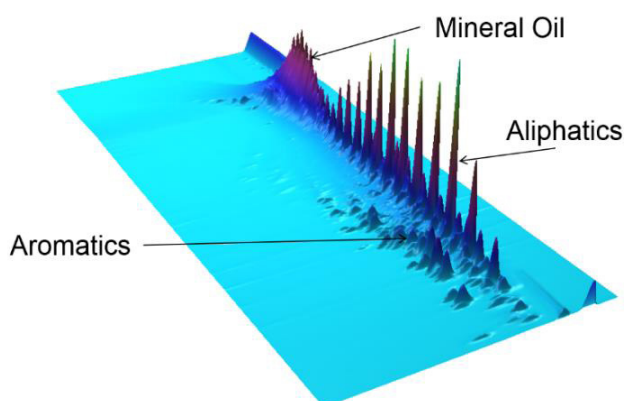
A series of standards from 40µg/ml to 4000µg/ml TPH standards were made up in pentane/DCM. Samples were extracted in pentane/DCM. Blanks, calibrations and samples were injected using the 7693A sampler and low split flows/ratios with hydrogen as a carrier. Column 1 and column 2 flows were optimised along with modulation times. The data was then imported in GC image software and a TPH template applied to generate images and results for banding, fractions, compound classes and individual components.

GCxGC 2D Plot



2D plot showing aliphatic/aromatic separations and banding

GCxGC 3D Plot – Mineral Oil



Results Summary

- Clear Separation of Aliphatics from Aromatics
- Unambiguous Banding
- Quantitation 5-5000 ppm TPH linearity >0.995 - RT %RSD's Column 1 <0.01%
- Result repeatability <1%
- Recovery 98 -101% (500 – 2500ppm doped soil)

A Turn-key solution

The JSB TPH Environmental analyzer is configured and fully tuned, tested and performance checked at the customers site ensuring trouble free operation. Agilent's hardware and JSB's application solution form a powerful combination, ensuring easy-of-use and fast and reliable results. In addition, training is given, however this system does not require a high degree of operator skill. This minimises the time spent from installation to obtain reliable accurate results. **UKAS & MCERTS** Accredited for C8 - C40 Hydrocarbons.

Flexibility

The analyser can be tuned to analyse a variety of samples in the C5 - C44 hydrocarbon range. The auto sampler can be utilised on a second injector/detector.

Conclusions

GCxGC can be used to remove the time consuming, costly and inaccurate aliphatic/aromatic splitting required for traditional TPH analysis. A second instrument and method may not be required. The savings and associated cost reductions will vary between laboratories dependent on numbers of samples being analysed. Data processing is a simple and easy process, the complex looking chromatograms are processed automatically using predefined templates. The Agilent flow modulator is a reliable and robust technology and has made GCxGC a viable and cost effective technology for use in a routine environment. An existing Agilent 7890B GC fitted with a Split/Splitless injector and FID can simply be upgraded to a TPH Analyser.

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References

[TPH Analysis Overview](#)

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