



Method Summary

Determination of Speciated Extractable Petroleum Hydrocarbons in Soils by GCxGC-FID

Scope and Range

Petroleum Hydrocarbons are a complex mixture of aliphatic and aromatic species. This method describes a procedure for the analysis of solvent (n-hexane/acetone) Extractable Petroleum Hydrocarbons (EPH), in the carbon number range >C10 to C40 by Comprehensive Gas Chromatography with flame ionisation detection (GCxGC-FID) from 'as received' samples. The extracts are speciated into aliphatic and aromatic species in the GC based on boiling point and polarity and banded in the ranges >C10-C12, >C12-C16, >C16-C21, >C21-C35 and >C35-40. The reporting limit for each band is <1mg/kg and <10mg/kg total based on discrete peak analysis. Reporting limits (LOR) for this method are based on 10g of soil being used for the extraction; however, the detection limits will vary if a reduced amount of sample is available for extraction. Any dilution factor (DF) required is taken into account in the calculation of results. Linear Range: The linear calibration range for the method is LOR to 8000 mg/kg.

Principle

Preparation and Extraction

Samples should be collected in glass containers and kept between 1-5°C until ready for extraction. 10g of as received sample is put into a 200ml bottle and spiked with surrogate. A hexane/acetone solvent mix is added and the samples are extracted using a reciprocating shaker. Analytical reagents are added and a portion of the sample extract is sent to the analysis laboratory.

Analysis

The vials are loaded onto the GCxGC-FID along with vials containing standards, blanks, and AQC samples. An AQC sample and a blank are extracted with and run with every twenty samples. GCxGC-FID instruments are calibrated using a mixture of common petroleum products fortified with PAHs and samples are quantified against a five-point calibration curve. Any extracts with concentrations higher than the top standard are diluted and re-run until they fall within the calibration range.

Interferences

Interferences co-extracted from the sample will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, it may be necessary to dilute the sample before GC analysis in order to reduce the effect of interferences.

Flame ionisation is a non-specific means of detection therefore any substance that is co-extracted and elutes from the chromatographic column within the region of interest will interfere with this determination.