Method Summary

Determination of Total Petroleum Hydrocarbons (Oil and Grease) in Solids by Infra-red Spectroscopy

Scope and Range

This method is suitable for the determination of total petroleum hydrocarbons and mineral oil content of solids.

The detection limit for soils is 10mg/kg, with a maximum content of 2660mg/kg, without dilution.
The calibration range is 0-400mg/l.

References

The Determination of Hydrocarbon Oils in Waters by Solvent Extraction, Infra red Absorption and Gravimetry 1983,(MEWAM), HMSO, London

Analysis of Hydrocarbons in Waters – A Review, plus an Ultra Violet Fluorescence Spectrophotometric Method 1988, HMSO, London

Analysis of Hydrocarbons in Soils and Waters using Infra Red, USEPA Method 418.1.
DIN standard 38 409 Hartlepool 18 1981

Principle

Preparation and Extraction
Samples should be collected in a 1 litre plastic tub and kept at 1-8°C until ready for extraction.

3g of soil is mixed in a vial with sodium sulphate until it is a free moving powder before being sonicated in TCE (tetrachloroethylene) for 20 minutes and mixed at 60rpm for 15minutes. The sample is then filtered through a glass fibre filter into another labelled vial where it is stored until being transferred to a quartz cell for analysis.

Mineral oil (equivalent) preparation is done by adding activated florisil to the filtered sample and mixing end over end.

Analysis
An aliquot of the sample is placed in the spectrometer and scanned between 2800 and 3200 wavenumbers. Three absorbance peaks in the scanned spectrum are identified and measured automatically. These peaks are characteristic of the 3 Carbon-Hydrogen (C-H) bonds found in petroleum compounds, i.e. Aromatic, CH_2 and CH_3 C-H bonds. The heights of these peaks are compared to those found in standards of known concentration to give a result for the sample.

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The sample can be analysed for mineral oil content by adding activated florisil to remove the aromatic (grease) content and re-scanning. The difference in the TPH and mineral oil results is the grease content.

Interferences

The method is a basic scanning method for C-H bonds, and as such anything in the sample with C-H bonds will absorb at the wavenumbers used for the analysis. Any major interference seen in the scan will result in the sample being NDP'd for this analysis and another method such as GC-MS being recommended. Samples requiring more in-depth analysis should be analysed by GC.