



Method Summary

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

Scope

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

The method is accredited to ISO17025 for TPH in surface water, treated sewage effluent and trade effluent. Mineral oil content using the florisil clean-up is not accredited.

The detection limit for waters is 1 mg/l, with a maximum content of 80mg/l, without dilution. The calibration range is 0-400mg/l.

Principle

Preparation and Extraction

Samples should be collected in glass bottles and kept at $5\pm 3^{\circ}\text{C}$ until ready for extraction.

125ml of sample is extracted into 25mls of tetrachloroethylene (PCE) using a shaker. This is then separated and dried with anhydrous sodium sulphate. The samples are kept in a rack with other samples in the extraction batch until being transferred to a quartz cell for analysis.

Mineral oil preparation is done by adding activated florisil to the 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.

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.