Method Summary

Determination of Simulated Distillation Products from Soil and Water in the Carbon Range C15 – C90 by GC-FID

Scope and Range

This method covers the determination of petroleum distillate fractions. The method is applicable to petroleum distillate fractions having a boiling range of 270°C to 702°C (C15 to C90). This method describes a procedure for the analysis of solvent (DCM) extractable petroleum hydrocarbons, in the carbon number range C15 to C90 by Gas Chromatography (GC) with flame ionisation detection (FID). This method is applicable to soils and waters.

Limits of Detection (LOD)

Detection limits for this method are based on 5g of soil or 1000ml of water being used for the extraction; however, the detection limits will vary if less sample is available for extraction.

LOD soil – 100mg/kg.
LOD water – 1mg/l.

Linear range

The linear range for soils is 100mg/kg to 10^6mg/kg.
The linear range for waters is 1mg/l to 10^6mg/l.
This method is not accredited.

References

ASTM D6352 Standard Test Method for Boiling Range Distribution of Petroleum Distillates in Boiling Range from 174 to 700°C by Gas Chromatography

Principle

Samples are extracted using DCM, either by Soxtherm (soil) or liquid/liquid (water) extraction. The solvent extract is reduced to 1ml which is then analysed using GC-FID.

For water analysis

Water is extracted with DCM using a liquid/liquid extraction. The sample is shaken with DCM to extract any petroleum hydrocarbons into the solvent. The solvent extract is then separated from the sample and dried. The solvent extract is blown down to dryness to determine the Solvent Extractable Matter content (SEM). The extracted matter is then reconstituted and run on a GC-FID.

For soil analysis

Soil is extracted with DCM using a Soxtherm. The sample is extracted with DCM to remove any petroleum hydrocarbons into the solvent. The solvent extract is blown down to dryness to determine the SEM. The extracted matter is then reconstituted and run on a GC-FID.

Analysis and quantitation

Samples are injected using an auto sampler into a GC-FID system. A polywax standard and a

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Florida mix standard are analysed to determine carbon banding. These bands are then applied to the sample chromatogram to determine the extractable matter present in each band.

Interferences

External contamination of water, standards and glassware can be detected by analysis of extraction blanks. Solvent blanks are run on the GC and are used to evaluate the cleanliness of the system. Any source of interference must be determined and corrective action taken to eliminate the problem where appropriate.
Solvents, reagent glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis. This is undertaken by analysis of extraction blanks.
Contamination by carry over can occur whenever high-level and low-level samples are analysed sequentially. Any suspected highly concentrated samples should be diluted accordingly at the extraction stage.
Whenever a highly concentrated sample is encountered as part of a batch subsequent samples where carryover is suspected are repeated.
Any compound within the retention time bands that is not an EPH which produces a signal on an FID will interfere.