Method Summary

Determination of PAH 16, Total PAH 17 and coronene by Gas Chromatography with flame ionisation detection

Scope and Range

Polynuclear aromatic hydrocarbons (PAHs) are prevalent in the environment, occurring naturally in fossil fuel products or from incomplete combustion of organic material. PAHs are classified as carcinogenic. This method describes a procedure for detection, identification and quantification of 17 PAHs.
This method is applicable to soils and sediments. Detection limits for this method are 10 mg/kg for pah16 & pahs17 and 2mg/kg for coronene based on 5±1g of extracted soil/oil based cutting fluid samples, however the detection limits will vary if less sample is available for extraction. Any dilution factor (DF) required is taken into account in the calculation of results. The standards used for quantification are a mixture of pah17 spike (see below) and a six-point calibration is run covering the range from 2.5µl to 340µl. Any extracts with concentrations higher than the top standard are diluted and re-run until they fall within the calibration range.

References

none

Principle

Preparation and Extraction:

Using a calibrated balance, weigh 5±1g of into a 100ml bottle in Wet Prep, the samples are then delivered to the Rapid Organics Cell. Samples are extracted on 'as received' soil 2±1g of copper granules is added and approximately 8g of sodium sulphate. Then 10ml of acetone and 10ml of hexane is added. Every 20 samples an AQC and blank are extracted. AQC and blank soil has to be pre-wetted to a moisture content of 10% before extraction. AQC soil is spiked with 200 ± 5µl of 500 mg/l pah17 spike. Samples are then shaken for 60min at a speed of approximately 240rpm ±10rpm on a reciprocating shaker. After shaking using a calibrated 1ml pipette then transfer 1ml of extracted sample into a 2 ml vial and cap it.

Analysis:

Sample information including details necessary for calculation of results are entered into the software at the loading stage. The samples are loaded onto the carousel in a defined order with an initial heat-through programme to ensure the instrument is clean. Retention times are obtained by running a pah/coronene standard and drawing a baseline under the peaks of the chromatogram between these defined times performs integration of samples. Calculation of analyte concentration by the chromatographic software i.e. area under the chromatogram is converted to concentration using the pre-determined calibration curve. Final concentration of sample is calculated by the software and manually entered in to the LIMS.
A calibration check standard of differing levels is analysed every 20 samples so that the whole of the calibration range is re-assessed for linearity. The calibration check must fall within ±20% of the expected concentration.

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A 6-point calibration is run weekly. The calibration slopes must fall within the set criteria, having a correlation coefficient of greater than 0.998. The calibration curves are filed with the sequence. If the correlation coefficient is lower than 0.998 then the instrument must be recalibrated. If the calibration check standard does not fall within ±20% the GC must be re-calibrated and any samples analysed after the failed check standard should be re-analysed.

Interferences

Solvents, reagent glassware and other sample processing hardware may yield artefacts and/or interferences to sample analysis.
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.
Contamination by carry-over can occur whenever high-level and low-level samples are sequentially analysed. As part of the auto-sampling sequence of the GC-FID, the syringe is rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered the next analysis is a solvent blank to ensure that no cross contamination occurs.
Flame ionisation is a non-specific means of detection; therefore any substances that co elute from the column with any of the components of interest will interfere with this detection.