



Method Summary

Determination of PAH in soil samples by microwave extraction and GC-MS

Scope and Range

This method describes a procedure for the identification and quantification of sixteen polycyclic aromatic hydrocarbons (PAHs) plus coronene, using the CEM MARS microwave extraction unit and an Agilent Gas Chromatograph Mass Selective Detector (GC-MSD).

The method has been validated against sand, loam and clay matrices and is accredited to ISO 17025 and the MCERTS standard for the sixteen polycyclic aromatic hydrocarbons. Coronene is outside the scope of accreditation.

The limits of detection are detailed on the following page.

Principle

Soluble organic contaminants are extracted from soil samples with hexane:acetone:triethylamine 50:45:5%v/v using the CEM, Microwave Accelerated Reaction System (MARS).

A surrogate solution containing five deuterated PAHs is added to the samples prior to extraction; these are used to monitor the efficiency of the extraction procedure.

An internal standard solution is added to sample extracts. The sample components are separated and detected using a suitable GC-MS system.

Compound identification is performed by Selective Ion Monitoring (SIM) and quantification of the components is carried out by means of the Internal Standard (IS) calibration technique.

The recommended maximum holding time for the analysis of PAHs in soil is 14 days prior to extraction and 40 days after extraction. Samples and extracts are stored at 1 to 5°C.

Preparation and Extraction

5g of well homogenised 'as received' soil is extracted, using the microwave extraction system, with 20ml of the solvent mix to which the surrogate standards have been added. Following extraction, 1ml of the extract is transferred to an autosampler vial along with an internal standard, which is used in calculating concentrations.

Analysis

The vials are loaded onto the GC autosampler along with vials containing standards, blanks, and AQC samples. An AQC sample and a blank are extracted with and run with every twenty samples.

The standards used for quantitation contain the 16 USEPA PAHs and a six-point calibration is run. Any extracts with concentrations higher than the top standard are diluted and re-run until they fall within the calibration range.

Sample concentrations are calculated against the standard calibration. The surrogate recovery is reported but the results do not compensate for that recovery.

Interferences

Co-extracted materials 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 clean up by column chromatography.



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List of Compounds, Ions and Limits of Detection

Compound	Target Ions	Qualifier Ions	LOD (µg/kg)
Internal Standard			
Biphenyl-d10	164	80	-
Surrogates			
Naphthalene-d8	136	108	-
Acenaphthalene-d10	164	160	-
Phenanthrene-d10	188	160	-
Chrysene-d12	240	236	-
Perylene-d12	264	132	-
Target Compounds			
Naphthalene	128	102	9
Acenaphthylene	152	76	12
Acenaphthene	154	152	8
Fluorene	166	165	10
Phenanthrene	178	152	15
Anthracene	178	152	16
Fluoranthene	202	101	17
Pyrene	202	101	15
Benz(a)anthracene	228	226	14
Chrysene	228	226	10
Benzo(b)fluoranthene	252	126	15
Benzo(k)fluoranthene	252	126	14
Benzo(a)pyrene	252	125	15
Indeno(123,cd)pyrene	276	138	18
Dibenzo(ah)anthracene	278	139	23
Benzo(ghi)perylene	276	138	24

Determination of coronene in soil samples

The principle, preparation and extraction of coronene in soil samples is identical with the one described for the PAHs.

The analysis is been performed by GC-MSD using a four-point calibration. Any extracts with concentrations higher than the top standard are diluted and re-run until they fall within the calibration range.

Compound	Target Ions	Qualifier Ions	LOD (µg/kg)
Internal Standard			
Perylene-d12	264	132	-
Target Compounds			
Coronene	300	148	200