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<p><a href="#">Method name in non-abbreviated full form.</a></p> <p><b>Grimmer method</b> – the Grimmer method is based on the stable isotope dilution principle using GC-MS with selected ion monitoring (SIM mode) and allows the quantification of the PAH content in the sub-ppb range. The Grimmer method has been validated for various matrices during the work of BIU for the Environmental Specimen Bank of the German Federal Environment Agency (UBA) and is published.</p>	
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<p><a href="#">Method description in brief.</a></p> <p>The GC-MS analysis is performed using an Agilent 6890N instrument connected to an Agilent 5973N quadrupole mass spectrometer operated in the selected-ion monitoring (SIM) mode and an Agilent 7683 Autosampler from Agilent Technologies (Santa Clara, CA, USA). Separation of the PAH profile is performed on an Agilent DB-35MS capillary (Agilent Technologies, 30 m × 0.25 mm i.d. × 0.25 µm film thickness, virtually equivalent to a (35%-phenyl)methylpolysiloxane) using helium (purity 99.999%) as carrier gas (flow rate 1 mL/min). Separation of PAH is achieved using the following conditions: splitless injection of 1.0 µL sample; the GC oven temperature was programmed from 100 °C, hold for 5 min, following by different optimized temperature ramps to 340 °C (held for 5 min).</p> <p>A 3-point calibration is performed for each PAH compound with linear curve fitting in a working range of 0.03 to 10 ng/µL. The limits of quantification (LOQ) and limits of detection (LOD) are determined using the signal-to-noise ratios (S/N; LOQ is determined by a S/N ratio of 10:1). Identification of PAH is conducted based on relative retention times and molecular ions compared to reference materials. Quantification is achieved via the PAH applied as internal standards (stable isotope dilution method). The system is operated by Agilent Enhanced ChemStation Software (G1701DA Version D.00.00.38).</p>	
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<p><a href="#">Applicability of method.</a></p> <p>Hydrocarbon class: polycyclic aromatic hydrocarbons (PAH) with a carbon number range from C10 (eg Naphthalene) to C24 such as coronene (molecular weight range from 128 to 302). Using a 10 g aliquot of the sample, the Grimmer method shows LOD values in the range of 0.002 to 0.356 ppb, whereas LOQ values lying in the range of 0.006 to 1.068 ppb (different for individual PAH compounds). Resolution of critical PAH isomers such as the benzofluoranthenes, triphenylene/chrysene, dibenzanthracenes etc. is achieved.</p>	
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#### Sample preparation required.

The sample preparation depends on the matrix to be analysed and is adjusted accordingly. If the matrix is soluble in cyclohexane, sample preparation is started with dissolution of a sample aliquot in this solvent. If not, an extract is first prepared in hot toluene (Soxhlet technique). In order to remove almost all matrix components, the solution of the crude material (or extract) is subjected to LLE (liquid-liquid extraction) and subsequent SPE steps using silica gel. Before subjected to instrumental analysis the sample volume is reduced via evaporation under vacuum or diluted if needed.

#### Method strengths.

A significant technical strength of the Grimmer method is the excellent removal of most matrix components allowing to use a large sample size for analysis. Thus, individual PAH can be determined with high sensitivity (sub-ppb range). Also due to the high specificity and the excellent gaschromatographic resolution of single peaks the method allows the determination of a wide range of PAH and alkylated PAH (methyl-PAH). The method has been validated by various collaborative studies and is recommended for the determination of PAH in several matrices.

#### Estimated time for analysis.

Time required for sample preparation can vary substantially depending on the type of matrix, thus, here an average time for preparation can be reported only.

- Preparation of two samples (in parallel) takes approximately 4 hours
- Instrumental analysis needs 1,5 hours per sample
- Data processing and interpretation is possible in roughly 30 minutes per sample
- Quality control of all data requires about 30 minutes

#### Method weaknesses.

No real weaknesses are known, but because of their relative volatility the PAH compounds naphthalene, acenaphthylene, acenaphthene and fluorene are difficult to be determined especially at low levels. The results obtained for these four PAH therefore always reflect only the current situation of the samples at the time of their investigation.

#### Result interpretation / visualisation / presentation.

- Data are GC selected-ion monitoring (SIM) chromatograms reflecting the signal areas of the analytes and internal standards
- Using the internal standards and calibration, the signal areas are converted to concentrations reported as  $\text{mg kg}^{-1}$  or  $\mu\text{g kg}^{-1}$ .
- Software generated custom reports are used to automatically generate test reports covering the requested PAH compounds.

#### Relevant publications.

Grimmer, G., Jacob, J., and Naujack, K.-W. (1983). Profile of the polycyclic aromatic compounds from crude oils. Inventory by GCGC/MS. - PAH in environmental materials, Part 3. *Fres. Z. Anal. Chem.*, 314, 29-36.

Grimmer, G. (1985). Occurrence, analytics and significance of PAH as environmental carcinogens. (in German: Vorkommen, Analytik und Bedeutung der PAH als Umweltcarcinogene), *Erdöl und Kohle, Erdgas, Petrochemie*, 38, 310-314.

Grimmer, G., Jacob, J. and Naujack, K.-W. (1997) Atmospheric emission of polycyclic aromatic hydrocarbons in sampling areas of the German environmental specimen bank. Method for the precise measurement of gaseous and particle-associated polycyclic aromatic hydrocarbons in the sub-nanogram range using deuterated internal standards. *Chemosphere*, 34, 2213-2226.