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Characterization of waste - Determination of polycyclic aromatic hydrocarbons (PAH) in waste using gas chromatography mass spectrometry (GC/MS)

Charakterisierung von Abfällen - Bestimmung von polycyclischen aromatischen Kohlenwasserstoffen (PAK) in Abfall mittels Gaschromatographie-Massenspektrometrie (GC/MS)

Caractérisation des déchets - Dosage des hydrocarbures aromatiques polycycliques (HAP) dans les déchets par chromatographie en phase gazeuse/spectrométrie de masse (CG/SM)

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## Foreword

This document (EN 15527:2008) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2009, and conflicting national standards shall be withdrawn at the latest by January 2009.

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## Introduction

Polynuclear aromatic hydrocarbons (PAH) are ubiquitous because of the fact that they are released in appreciable quantities every year into the environment through the combustion of organic materials such as coal, fuel oils, petrol, wood, refuse and plant materials. Since some of these PAH compounds are carcinogenic or mutagenic, their presence in the environment (air, water, soil, sediment and waste) are regularly monitored and controlled. At present, the determination of PAH is carried out in these matrices in most of the routine laboratories following the preceding steps for sampling, pre-treatment, extraction, clean-up by measurement of specific PAH by means of gas chromatography in combination with mass spectrometric detection (GC-MS) or by HPLC in combination with UV-DAD- or Fluorescence-detection (HPLC-UV-DAD/FLD). However, the different matrices covered in this standard may contain a lot of contaminants. For this reason, the GC-MS method seems to be most appropriate for waste analysis.

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## 1 Scope

This European Standard specifies the quantitative determination of 16 polynuclear aromatic hydrocarbons (PAH) according to the priority list of the Environmental Protection Agency (EPA, 1982). This European Standard is applicable for wastes such as contaminated soil, sludge and rubble, bitumen or waste containing bitumen.

This European Standard describes a gas chromatographic method with mass spectrometric detection (GC-MS). Under the conditions specified in this document, a typical lower limit of application of 0,1 mg/kg for each individual PAH can be achieved.

NOTE 1 This method may be applied to the analysis of other PAH compounds not specified in the scope provided its applicability has been proven by proper in-house validation experiments.

NOTE 2 For some materials, e. g. bitumen, the lower limit of application of 0,1 mg/kg cannot be achieved due to interferences.

NOTE 3 Under certain circumstances the method may be applicable to PAH concentrations lower than 0,1 mg/kg but it is in the responsibility of the laboratory to provide proper validation data for such low concentrations.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14346, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

ISO 14507, *Soil quality — Pretreatment of samples for determination of organic contaminants*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1

#### **analyte**

selected polynuclear aromatic hydrocarbons (PAH) with 2 to 6 condensed aromatic rings (see Table 1)

### 3.2

#### **calibration standard**

solution of PAH prepared from a secondary standard and/or stock solutions of native PAH and deuterated PAH used to calibrate the response of the instrument

### 3.3

#### **extraction standard**

deuterated PAH or native (unlabeled) PAH of medium volatility that are unlikely to be present in waste added to the sample before extraction and used for control of the extraction efficiency

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**3.4 internal standard**  
deuterated PAH added to the sample after extraction and used for quantification of the concentrations of PAH in the sample

**3.5 injection standard**  
deuterated or native PAH that are unlikely to be present in waste samples added to the sample extract before injection into the GC, to monitor variability of instrument response and the recovery of the internal/extraction standards

**4 Principle**

The PAH are extracted from the sample by use of acetone and a hexane-like solvent. If appropriate, the obtained extract is purified by adsorption column chromatography. The extract is analysed by gas chromatography with mass spectrometric detection (GC-MS) using capillary columns of low polarity. Concentrations of PAH are quantified using an internal-standard-method.

**5 Reagents****5.1 General**

All reagents shall be of recognised analytical grade. The suitability of the reagents shall be checked by running a blank determination as described in 11.1.

**5.2 Reagents for extraction procedures****5.2.1 Reagents for extraction by shaking/sonication**

**5.2.1.1** Acetone.

**5.2.1.2** Petroleum ether (Boiling range 40 °C to 60 °C) or n-hexane or iso-hexane or cyclohexane (hexane-like solvents with a boiling range between 30 °C and 68 °C are allowed).

**5.2.1.3** Sodium chloride, anhydrous.

**5.2.1.4** Distilled water or water of equivalent quality.

**5.2.1.5** Anhydrous sodium sulphate, heated for at least 6 h to 550 °C ± 20 °C, cooled to about 200 °C in the furnace and then to ambient temperature in a desiccator containing magnesium perchlorate or a suitable alternative. The anhydrous sodium sulphate shall be kept carefully sealed. Commercial available anhydrous sodium sulphate is also applicable.

**5.2.2 Reagents for Soxhlet extraction**

1:1 (v/v) mixtures of acetone/petroleum ether (boiling range 40 °C to 60 °C) or acetone/ n-hexane or acetone/iso-hexane or acetone/cyclohexane.

**5.3 Reagents for clean-up procedures****5.3.1 Clean-up A – Aluminium oxide**

**5.3.1.1** Aluminium oxide basic or neutral, specific surface 200 m<sup>2</sup>/g, activity Super I.



**5.3.1.2** Preparation of deactivated aluminium oxide. Add 10 g of water to 90 g of aluminium oxide (5.3.1.1). Shake until all lumps have disappeared. Allow the aluminium oxide to equilibrate before use for some 16 h, sealed from air. Use it for a maximum of 2 weeks.

### 5.3.2 Clean up B- Silica gel 60

**5.3.2.1** Silica gel 60, particle size 63 µm to 200 µm.

**5.3.2.2** Preparation of silica gel 60 with a water content of 10 % (w/w). Silica gel 60, heated for at least 3 h at 450 °C, cooled down and stored in a desiccator containing magnesium perchlorate or another suitable drying agent. Before use, heat it once again at least for 5 h at 130 °C in a drying oven. Then allow to cool in a desiccator to ambient temperature. Transfer the silica gel to a flask and add 10 % water (w/w). Shake for 5 min intensively by hand until all lumps have disappeared and then for 2 h in a shaking machine. Store the deactivated silica gel sealed from air and use it for maximum of one week.

## 5.4 Reagents for gas chromatographic analysis

**Carrier gas** for gas chromatography, e. g. helium of suitable purity.

## 5.5 Standards

### 5.5.1 Calibration standards, extraction standards and internal standards

Choose internal/extraction standard substances whose physical and chemical properties (such as extraction behaviour, retention time) are similar to those of the compounds to be analysed. Deuterated PAH should be used as internal standards for the GC-MS method for evaluation of results. Verify the stability of the internal/extraction standards regularly. Table 1 contains native and a minimum number of deuterated PAH to be used for calibration of specific analytes. The use of isotope dilution (each native PAH is quantified with respect to his labelled analogue) is recommended.

NOTE Certified solutions of PAH and single solid PAH substances with certified purity are commercially available.

Table 1 — Native PAH and deuterated PAH

PAH reference substances for calibration	Internal/extraction standard substances: (deuterated PAH)
Naphthalene (CAS No. 91-20-3)	Naphthalene- D <sub>8</sub>
Acenaphthene (CAS No.83-32-9)	Acenaphthene-D <sub>10</sub>
Acenaphthylene (CAS No.208-96-8)	
Fluorene (CAS No.86-73-7)	
Anthracene (CAS No.120-12-7)	
Phenanthrene (CAS No. 85-01-8)	Phenanthrene-D <sub>10</sub> (extraction standard)
Fluoranthene (CAS No.206-44-0)	
Pyrene (CAS No.129-00-0)	
Benzo(a)anthracene (CAS No.56-55-3)	Benzo(a)anthracene-D <sub>12</sub>
Chrysene (CAS.No.218-01-9)	
Benzo(b)fluoranthene (CAS No. 205-99-2)	
Benzo(k)fluoranthene (CAS No.207-08-9)	
Benzo(a)pyrene (CAS No.50-32-8)	Benzo(a)pyrene-D <sub>12</sub>
Indeno(1,2,3-cd)pyrene (CAS No.193-39-5)	
Dibenzo(ah)anthracene (CAS No.53-70-3)	
Benzo(ghi)perylene (CAS No.191-24-2)	Perylene-D <sub>12</sub>

### 5.5.2 Injection standards

A deuterated PAH is added to the final extract before GC-MS injection to check the recovery of the deuterated internal/extraction standards. Suitable injection standards are 1-methylnaphthalene-D<sub>10</sub>, benzo(e)pyrene-D<sub>10</sub> and triphenylene-D<sub>12</sub>.

## 6 Apparatus

### 6.1 Extraction and clean-up procedures

Customary laboratory glassware.

All glassware and material that comes into contact with the sample or extract shall be free of PAH and any interfering compounds.

#### 6.1.1 Extraction procedures

**6.1.1.1** Glass sample bottles of appropriate size with glass stopper or screw top and polytetrafluorethene seal (PTFE).

- 6.1.1.2 Shaking device, with horizontal movement (200 to 300 strokes per minute).
- 6.1.1.3 Ultrasonic bath.
- 6.1.1.4 Water bath, adjustable up to 100 °C.
- 6.1.1.5 Separatory funnels with a capacity of 1 l.
- 6.1.1.6 Conical flasks with a capacity of 500 ml.
- 6.1.1.7 Soxhlet extraction apparatus, consisting of: round bottom flask, e. g. 100 ml, Soxhlet extractors and soxhlet thimbles, e. g. 27 mm x 100 mm, vertical condensers, e. g. 300 mm, water-bath or heating mantle as heating apparatus.
- 6.1.1.8 Evaporator, Kuderna Danish or other evaporators, e. g. a rotary evaporator, if found to be equally suitable.
- 6.1.1.9 5 ml volumetric flask.

## 6.1.2 Clean-up procedures

- 6.1.2.1 Quartz wool or silanized glass wool

NOTE Working with quartz wool imposes a risk to health through the release of fine quartz particles. Inhalation of these should be prevented.

- 6.1.2.2 Boiling chips glass or porcelain beads
- 6.1.2.3 Calibrated test tubes with a capacity of 10 ml to 15 ml and ground glass stopper.
- 6.1.2.4 Glass chromatography column, 5 mm to 10 mm internal diameter, length e. g. 600 mm.

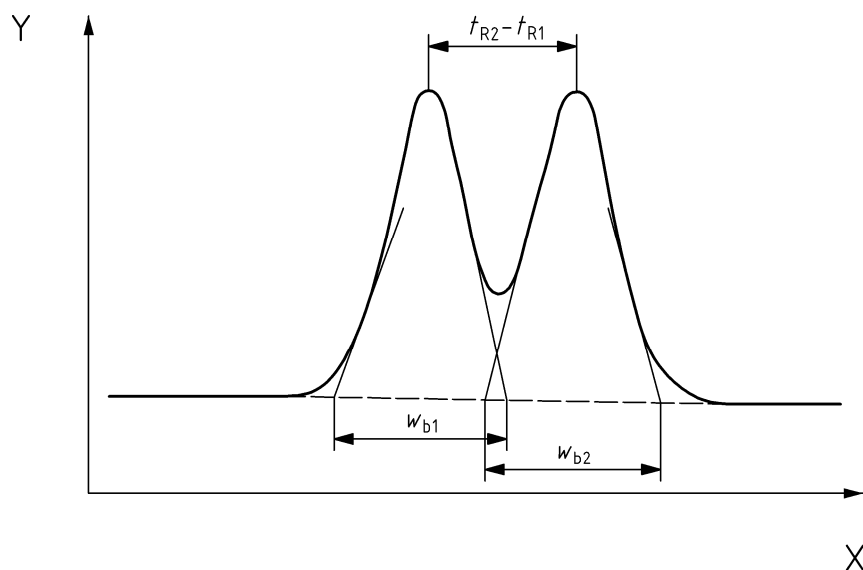
## 6.2 Gas chromatograph

Gas chromatograph equipped with an on-column, split/splitless or programmable temperature vaporizer (PTV) injection system, capillary column (6.3) and a mass spectrometric detector (GC-MS).

## 6.3 Capillary columns

Low to medium polar capillary columns such as 5 % phenyl-methyl silicone stationary phase coated onto fused silica capillary column or an equivalent chemically bonded phase column may be used. Choose a capillary column that allows for sufficient resolution of the critical pairs mentioned below.

The resolution between the chromatographic peaks of benzo(b)fluoranthene/ benzo(k)fluoranthene as well as of benzo(a)pyrene/benzo(e)pyrene shall be better than 0,5. The resolution is defined according to Figure 1 and Equation (1):

**Key**

- X** Time  
**Y** Intensity  
 $t_{R1}, t_{R2}$  Retention times of each eluted component 1 and 2 in s  
 $w_{b1}, w_{b2}$  Peak widths of each peak at its base in s

**Figure 1 — Resolution of chromatographic peaks**  
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$$R = 2 \frac{(t_{R2} - t_{R1})}{w_{b1} + w_{b2}}$$

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(1)

where

- $R$  is the resolution;  
 $t_{R1}, t_{R2}$  are the retention times of each eluted component 1 and 2 in s;  
 $w_{b1}, w_{b2}$  are the peak widths of each peak at its base in s.

## 6.4 Preparation of standard solutions

### 6.4.1 Single substance stock solution

Solutions of the individual substances of native and deuterated PAH (see Table 1) in an appropriate solvent with a mass concentration of e. g. 10 µg/ml.

Single substance stock solutions can be prepared from pure solid PAH or purchased. These solutions are used for confirmation and identification of single PAH in the chromatogram. The single substance stock solutions and diluted standard solutions are to be stored in a dark place at about 4 °C (or less). They are stable for about 1 year.

### 6.4.2 Multiple substance stock solution of native PAH

Dilute the multiple substance stock solution of the native PAH in an appropriate solvent to a mass concentration of e. g. 10 µg/ml for each individual PAH.