

English Version

**Sludge, treated biowaste and soil - Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC)**

Boues, bio-déchets traités et sols - Dosage des hydrocarbures aromatiques polycycliques (HAP) par chromatographie en phase gazeuse et chromatographie liquide à haute performance

Schlamm, behandelter Bioabfall und Boden - Bestimmung von polycyclischen aromatischen Kohlenwasserstoffen (PAK) mittels Gaschromatographie (GC) und Hochleistungs-Flüssigkeitschromatographie (HPLC)

This Technical Specification (CEN/TS) was approved by CEN on 16 July 2012 for provisional application.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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# Contents

Page

Foreword.....	4
Introduction.....	5
1 Scope.....	6
2 Normative references.....	7
3 Terms and definitions .....	7
4 Principle.....	8
5 Interferences .....	8
5.1 Interference with sampling and extraction.....	8
5.2 Interference with GC-MS .....	9
5.3 Interferences with the HPLC .....	9
6 Safety remarks .....	9
7 Reagents.....	10
7.1 General .....	10
7.2 Reagents for extraction.....	10
7.3 Reagents for clean-up.....	10
7.3.1 Clean-up using aluminium oxide.....	10
7.3.2 Clean-up using silica gel 60 for column chromatography .....	10
7.3.3 Clean-up using gel permeation chromatography (GPC) .....	11
7.3.4 Clean-up using liquid-liquid partition/DMF/cyclohexane .....	11
7.4 Reagents for chromatographic analysis .....	11
7.4.1 GC-Analysis.....	11
7.4.2 HPLC-analysis.....	11
7.5 Standards .....	11
7.5.1 Reference substances, internal standards .....	11
7.5.2 Injection standard .....	12
7.6 Preparation of standard solutions.....	12
7.6.1 General .....	12
7.6.2 Standard solutions for HPLC-Analysis .....	13
7.6.3 Standard solutions for GC-MS analysis .....	13
7.6.4 Calibration standard solutions .....	13
7.7 Preparation of internal standard solutions .....	14
7.8 Preparation of injection standard solution .....	14
8 Apparatus .....	14
8.1 Extraction and clean-up procedures .....	14
8.2 Gas chromatograph .....	15
8.2.1 General .....	15
8.3 High-performance liquid chromatograph.....	15
9 Sample storage and preservation .....	15
9.1 Sample storage .....	15
9.2 Sample pretreatment.....	16
10 Procedure .....	16
10.1 Blank test.....	16
10.2 Extraction .....	16
10.2.1 General .....	16
10.2.2 Extraction procedure 1: acetone/petroleum ether and agitation .....	17
10.2.3 Extraction procedure 2: Soxhlet extraction (dry samples).....	18
10.2.4 Extraction procedure 3: acetone/petroleum ether/sodium chloride and agitation.....	18
10.3 Concentration or dilution.....	19

10.3.1	General .....	19
10.3.2	For HPLC analysis.....	19
10.4	Clean-up of the extract.....	19
10.4.1	General .....	19
10.4.2	Clean-up A – Aluminium oxide .....	20
10.4.3	Clean-up B – Silica gel .....	20
10.4.4	Clean-up C – Gel permeation chromatography (styrene divinylbenzene resin) .....	21
10.4.5	Clean-up D – DMF/cyclohexane partitioning for aliphatic hydrocarbons removal .....	21
10.5	Addition of the injection standard .....	21
10.6	Gas chromatographic analysis (GC) .....	21
10.6.1	Gas chromatographic analysis with mass spectrometric detection.....	21
10.6.2	Calibration of the method using an internal standard .....	22
10.6.3	Measurement .....	24
10.6.4	Identification.....	24
10.6.5	Check on method performance .....	24
10.6.6	Calculation.....	25
10.7	High-performance liquid chromatographic analysis (HPLC).....	25
10.7.1	General .....	25
10.7.2	Chromatographic separation .....	25
10.7.3	Detection.....	26
10.7.4	Calibration .....	27
10.7.5	Measurement of samples .....	27
10.7.6	Calculation.....	27
11	Performance characteristics.....	28
12	Precision.....	28
13	Test report .....	28
Annex A	(informative) Repeatability and reproducibility data.....	29
A.1	Materials used in the interlaboratory comparison study.....	29
A.2	Interlaboratory comparison results.....	30
Annex B	(informative) Examples of instrumental conditions and chromatograms.....	32
B.1	Measurement of PAH with GC-MS .....	32
B.2	Measurement of PAH with HPLC fluorescence.....	38
Bibliography	.....	42

## **Foreword**

This document (CEN/TS 16181:2013) has been prepared by Technical Committee CEN/TC 400 "Project Committee - Horizontal standards in the fields of sludge, biowaste and soil", the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## Introduction

Polycyclic aromatic hydrocarbons (PAH) are ubiquitous because they are released in appreciable quantities every year into the environment through the combustion of organic matters 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) is regularly monitored and controlled. At present determination of PAH is carried out in these matrices in most of the routine laboratories following the preceding steps for sampling, pretreatment, extraction, clean-up by measurement of specific PAH by means of gas chromatography in combination with mass spectrometric detection (GC-MS) or by high performance liquid chromatography (HPLC) in combination with UV-DAD- or fluorescence-detection (HPLC-UV-DAD/FLD). Both the GC-MS and the HPLC methods are included in this horizontal standard.

It is to be underlined that the target contamination level of PAH can lie in the range of about 0,01 mg/kg per individual PAH (agricultural soil and sediment) to about 200 mg/kg and higher (e.g. contaminated soil at coking plant sites or waste). The use of internal and injection standards is described in order to have an internal check on execution of the extraction and clean-up procedure. The method is as far as possible in agreement with the method described for PCBs (see EN 16167).

This document is the result of a desk study "Horizontal Technical Specification for determination of PAH in sludge, soil and biowaste" in the project "Horizontal" and aims at evaluating the latest developments in assessing PAH in sludge, soil, treated biowaste and neighbouring fields. After an evaluation study, in which the ruggedness of the method was studied, a European-wide validation of the draft standard has taken place. The results of the desk studies as well as the evaluation and validation studies have been subject to discussions with all parties concerned in CEN.

This Technical Specification is applicable and validated for several types of matrices as indicated in Table 1 (see also Annex A for the results of the validation).

**Table 1 — Matrices for which this Technical Specification is applicable and validated**

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Fresh compost

**WARNING —** Persons using this Technical Specification should be familiar with usual laboratory practice. This Technical Specification does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

**IMPORTANT —** It is absolutely essential that tests conducted according to this Technical Specification be carried out by suitably trained staff.

## 1 Scope

This Technical Specification specifies the quantitative determination of 16 polycyclic aromatic hydrocarbons (PAH) (see Table 2) in sludge, soil and treated biowaste using GC-MS and HPLC-UV-DAD/FLD covering a wide range of PAH contamination levels (see also Annex B).

When using fluorescence detection, acenaphthylene cannot be measured.

**Table 2 — Polycyclic aromatic hydrocarbons  
which can be analysed using this Technical Specification**

Target analyte	CAS-RN <sup>a</sup>
Naphthalene	91-20-3
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Fluorene	86-73-7
Anthracene	120-12-7
Phenanthrene	85-01-8
Fluoranthene	206-44-0
Pyrene	129-00-0
Benz(a)anthracene	56-55-3
Chrysene	218-01-9
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(a)pyrene	50-32-8
Indeno(1,2,3-cd)pyrene	193-39-5
Dibenz(a,h)anthracene	53-70-3
Benzo(ghi)perylene	191-24-2
<sup>a</sup> CAS-RN Chemical Abstracts Service Registry Number.	

The limit of detection depends on the determinants, the equipment used, the quality of chemicals used for the extraction of the sample and the clean-up of the extract.

Typically, a lower limit of application of 0,01 mg/kg (expressed as dry matter) may be ensured for each individual PAH. This depends on instrument and sample.

Sludge, soil and treated biowaste may differ in properties and also in the expected contamination levels of PAHs and presence of interfering substances. These differences make it impossible to describe one general procedure. This Technical Specification contains decision tables based on the properties of the sample and the extraction and clean-up procedure to be used. Two general lines are followed, an agitation procedure (shaking) or use of soxhlet/pressurised liquid extraction.

NOTE Other PAH compounds can also be analysed with this method, provided suitability has been proven.

## 2 Normative references

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

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

EN ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples (ISO 5667-15)*

EN ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis (ISO 16720)*

EN ISO 22892, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry (ISO 22892)*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

## 3 Terms and definitions

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

### 3.1

#### **critical pair**

pair of congeners that will be separated to a predefined degree (e.g.  $R = 0,5$ ) to ensure chromatographic separation meets minimum quality criteria

[SOURCE: EN 15308:2008, 3.6]

Note 1 to entry: See Figure 1.