

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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English Version

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

Sols, biodéchets traités et boues - Dosage des hydrocarbures aromatiques polycycliques (HAP) par chromatographie en phase gazeuse et chromatographie liquide à haute performance

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

This European Standard was approved by CEN on 4 April 2018.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 16181:2018) has been prepared by Technical Committee CEN/TC 444 "Test methods for environmental characterization of solid matrices", 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of an identical text or by endorsement, at the latest by December 2018, and conflicting national standards shall be withdrawn at the latest by December 2018.

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

This document supersedes CEN/TS 16181:2013.

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 organizations of the following countries are bound to implement this European Standard: 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, Serbia, 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.

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 European Standard 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 European Standard 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 European Standard is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Fresh compost
Soil	Sandy soil

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard 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 European Standard be carried out by suitably trained staff.

1 Scope

This European Standard 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 European Standard

Target analyte	CAS-RN ^a
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[<i>a</i>]anthracene	56-55-3
Chrysene	218-01-9
Benzo[<i>b</i>]fluoranthene	205-99-2
Benzo[<i>k</i>]fluoranthene	207-08-9
Benzo[<i>a</i>]pyrene	50-32-8
Indeno[1,2,3- <i>cd</i>]pyrene	193-39-5
Dibenz[<i>a,h</i>]anthracene	53-70-3
Benzo[<i>ghi</i>]perylene	191-24-2
^a 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) can be ensured for each individual PAH. This depends on instrument and sample.

Sludge, soil and treated biowaste can 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 European Standard 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/pressurized liquid extraction.

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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 PAH that will be separated to a predefined degree (e.g. $R = 0,5$) to ensure chromatographic separation meets minimum quality criteria

[SOURCE: EN 15308:2016, 3.6]

Note 1 to entry: See Figure 1.