ILNAS

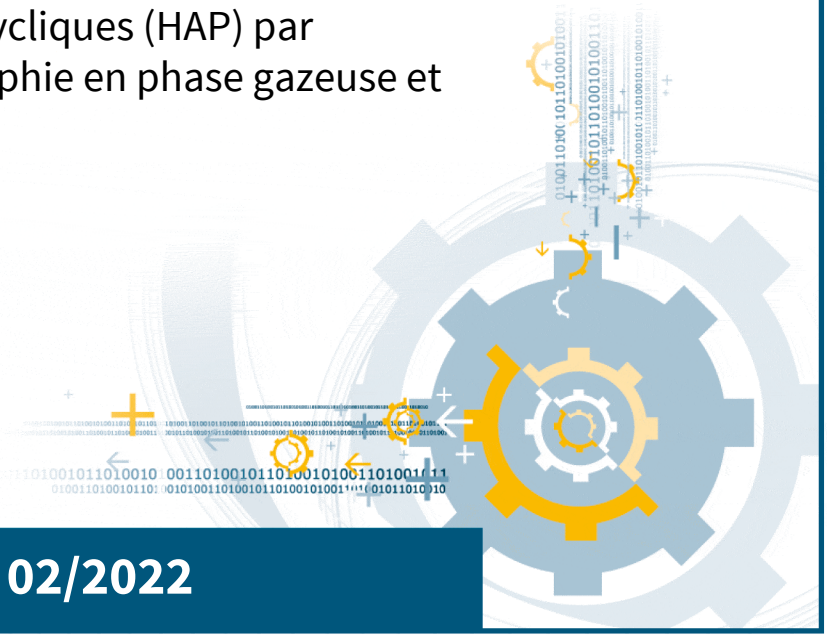
Institut luxembourgeois de la normalisation
de l'accréditation, de la sécurité et qualité
des produits et services

ILNAS-EN 17503:2022

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

Boden, Schlamm, behandelter Bioabfall und Abfall - Bestimmung von polycyclischen aromatischen Kohlenwasserstoffen (PAK) mittels

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



02/2022

National Foreword

This European Standard EN 17503:2022 was adopted as Luxembourgish Standard ILNAS-EN 17503:2022.

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ILNAS-EN 17503:2022

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 17503

February 2022

ICS 13.030.01; 13.080.10

Supersedes EN 15527:2008, EN 16181:2018

English Version

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

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

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

This European Standard was approved by CEN on 3 January 2022.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 17503:2022 E

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European foreword

This document (EN 17503:2022) has been prepared by Technical Committee CEN/TC 444 “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 August 2022, and conflicting national standards shall be withdrawn at the latest by August 2022.

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

This document supersedes EN 15527:2008 and EN 16181:2018.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, 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 prescribed steps specified 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 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.

This document was developed by the merging of EN 16181:2018, initially elaborated as a CEN Technical Specification in the European project 'HORIZONTAL' and validated by CEN/TC 400 with the support of BAM, with EN 15527, published by CEN/TC 292.

Considered the different matrices and possible interfering compounds, this document does not contain one single possible way of working. Several choices are possible, in particular relating to clean-up. Quantification with both GC-MS-detection and HPLC-DAD-UV/FLD is possible. Four different extraction procedures are described and three clean-up procedures. The use of internal and injection standards is described in order to have an internal check on choice of the extraction and clean-up procedure. The method is as far as possible in agreement with the method described for polychlorinated biphenyls (PCB) in EN 17322. It has been tested for ruggedness.

This document 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 document is applicable and validated

Matrix	Materials used for validation
Soil	Sandy soil Mix of soil from an industrial area in Brandenburg, Germany and PAH-free German reference soil
Sludge	Mix of municipal waste water treatment plant sludge from the vicinity of Berlin, Germany
Biowaste	Mix of compost from the vicinity of Berlin, Germany
Waste	Contaminated soil, building debris, waste wood, roofing tar, shredder light fraction

WARNING — Persons using this document should be familiar with usual laboratory practice. This document 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.

WARNING — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies different methods for quantitative determination of 16 polycyclic aromatic hydrocarbons (PAH) (see Table 2) in soil, sludge, treated biowaste, and waste, using GC-MS or HPLC-UV-DAD/FLD covering a wide range of PAH contamination levels (see Table 2).

NOTE The method can be applied to sediments provided that validity is demonstrated by the user.

When using fluorescence detection, acenaphthylene cannot be measured.

Table 2 —Target analytes of this document

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[*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

[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.

Under the conditions specified in this document, the lower limit of application from 10 µg/kg (expressed as dry matter) for soils, sludge and biowaste to 100 µg/kg (expressed as dry matter) for solid waste can be achieved. For some specific samples (e.g. bitumen) the limit of 100 µg/kg cannot be reached.

Sludge, waste and treated biowaste can differ in properties as well as in the expected contamination levels of PAH and presence of interfering substances. These differences make it impossible to describe one general procedure. This document contains decision tables based on the properties of the sample and the extraction and clean-up procedure to be used.

The method can be applied to the analysis of other PAH not specified in the scope, provided suitability is proven by proper in-house validation experiments.

Sampling is not part of this standard. In dependence of the materials, the following standards need to be considered, e.g. EN 14899, ISO 5667-12 and EN ISO 5667-13.

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

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 — Part 1: Linear calibration function*

3 Terms and definitions

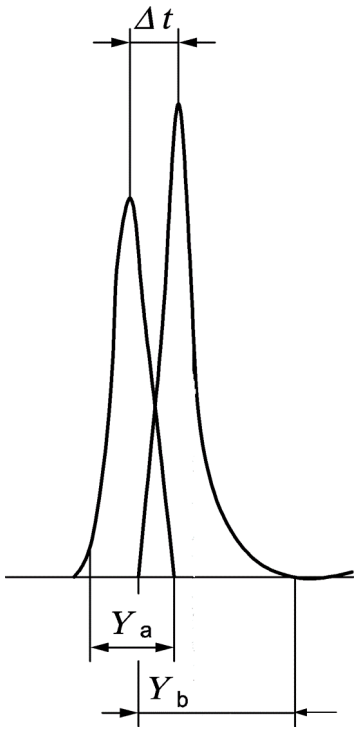
For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
critical pair
pair of PAH that are separated to a predefined degree (e.g. R = 0,5) to ensure chromatographic separation meets minimum quality criteria

EXAMPLE Figure 1 shows an example of a chromatogram of a critical pair.



$$R = 2 \times \frac{\Delta t}{Y_a + Y_b} \quad (1)$$

where:

R	peak separation
Δt	difference in retention times of the two peaks *a* and *b* in seconds (s)
Y_a	peak width at the base of peak a in seconds (s)
Y_b	peak width at the base of peak b in seconds (s)

Figure 1 — Example of a chromatogram of a critical pair

3.2
internal standard
compound added in a known amount to the sample from the beginning of the protocol and enabling analytical coverage throughout the procedure, and that is used to correct for losses during sample preparation and analysis by accounting for all-system matrix effects (recoveries, ionization effect, variability of the detector response of the instrument for example)

Note 1 to entry: isotopically labelled mostly deuterated PAH or native PAH unlikely to be present in the sample

[SOURCE: EN ISO 21253-2:2019, 3.10]

3.3
injection standard
standard mixture added to a sample before injection into the GC-MS apparatus, to monitor variability of instrument response and to calculate internal standard recovery

[SOURCE: ISO 28540:2011, 3.4]