



NSAI
Standards

Irish Standard
S.R. CEN/TS 16181:2013

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

S.R. CEN/TS 16181:2013

Incorporating amendments/corrigenda/National Annexes issued since publication:

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SWIFT xxx: A rapidly developed recommendatory document based on the consensus of the participants of an NSAI workshop.

This document replaces:

This document is based on:
CEN/TS 16181:2013

Published:
11 October, 2013

This document was published under the authority of the NSAI and comes into effect on:
11 October, 2013

ICS number:

13.030.20

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ICS 13.030.20

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.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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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 ^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) 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.

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