

Soil, treated biowaste and sludge - Determination of polychlorinated biphenyls (PCB) by gas chromatography with mass selective detection (GC-MS) and gas chromatography with electron-capture detection (GC-ECD) (Corrected version 01.2019)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 16167:2018 sisaldab Euroopa standardi EN 16167:2018+AC:2019 ingliskeelset teksti.	This Estonian standard EVS-EN 16167:2018 consists of the English text of the European standard EN 16167:2018+AC:2019.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 15.08.2018, parandusega konsolideeritud väljaanne 30.01.2019.	Date of Availability of the European standard is 15.08.2018, for consolidated version 30.01.2019.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

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English Version

Soil, treated biowaste and sludge - Determination of polychlorinated biphenyls (PCB) by gas chromatography with mass selective detection (GC-MS) and gas chromatography with electron-capture detection (GC-ECD)

Sols, biodéchets traités et boues - Dosage des polychlorobiphényles (PCBs) par chromatographie en phase gazeuse-spectrométrie gazeuse couplée avec un détecteur de masse (GC-SM) ou un détecteur par capture d'électrons (GC-ECD)

Boden, behandelter Bioabfall und Schlamm - Bestimmung von polychlorierten Biphenylen (PCB) mittels Gaschromatographie mit Massenspektrometrie-Kopplung (GC-MS) und Gaschromatographie mit Elektroneneinfangdetektion (GC-ECD)

This European Standard was approved by CEN on 20 March 2018 and includes the Corrigendum issued by CEN on 30 January 2019.

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European foreword

This document (EN 16167:2018+AC:2019) 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of an identical text or by endorsement, at the latest by July 2019, and conflicting national standards shall be withdrawn at the latest by July 2019.

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 AC EN 16167:2018 AC.

This document includes the corrigendum 1 which deletes the mandate information.

The start and finish of text introduced or altered by corrigendum is indicated in the text by tags AC AC

AC *deleted text* AC.

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, 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

Polychlorinated biphenyls (PCB) have been widely used as additives in industrial applications where chemical stability has been required. This stability on the other hand creates environmental problems when PCBs are eventually released into the environment. Since some of these PCB compounds are highly toxic, their presence in the environment (air, water, soil, sediment and waste) is regularly monitored and controlled. At present determination of PCB 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 PCB by means of gas chromatography in combination with mass spectrometric detection (GC-MS) or gas chromatography with electron capture detector (GC-ECD).

This European Standard was developed in the European project 'HORIZONTAL'. It is the result of a desk study "3-12 PCB" and aims at evaluation of the latest developments in assessing PCBs in sludge, soil, treated biowaste and neighbouring fields. Taken into account the different matrices and possible interfering compounds, this European Standard does not contain one single possible way of working. Several choices are possible, in particular relating to clean-up. Detection with both MS-detection and ECD-detection is possible. Three different extraction procedures are described and 11 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 PAHs (see EN 16181). It has been tested for ruggedness.

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 sewage sludge
Biowaste	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 draft European Standard specifies a method for quantitative determination of seven selected polychlorinated biphenyls (PCB28, PCB52, PCB101, PCB118, PCB138, PCB153 and PCB180) in sludge, treated biowaste and soil using GC-MS and GC-ECD (see Table 2).

Table 2 — Target analytes of this European Standard

Target analyte		CAS-RN ^a
PCB28	2,4,4'-trichlorobiphenyl	7012-37-5
PCB52	2,2',5,5'-tetrachlorobiphenyl	35693-99-3
PCB101	2,2',4,5,5'-pentachlorobiphenyl	37680-73-2
PCB118	2,3',4,4',5-pentachlorobiphenyl	31508-00-6
PCB138	2,2',3,4,4',5'-hexachlorobiphenyl	35065-28-2
PCB153	2,2',4,4',5,5'-hexachlorobiphenyl	35065-27-1
PCB180	2,2',3,4,4',5,5'-heptachlorobiphenyl	35065-29-3
^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 European Standard, limit of application of 1 µg/kg (expressed as dry matter) can be achieved.

Sludge and treated biowaste may differ in properties and also in the expected contamination levels of PCBs 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.

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.

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

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

3.1

polychlorinated biphenyl

PCB

biphenyl substituted by one to ten chlorine atoms

[SOURCE: EN 15308:2016, 3.1]

3.2

congener

member of the same kind, class or group of chemicals, e.g. anyone of the two hundred and nine individual PCB

Note 1 to entry: The IUPAC congener numbers are for easy identification; they do not represent the order of chromatographic elution.

[SOURCE: EN 15308:2016, 3.2]

3.3

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 15308:2016, 3.6]

3.4

internal standard

$^{13}\text{C}_{12}$ -labelled PCB or other PCB that are unlikely to be present in samples added to the sample before extraction and used for quantification of PCB content

[SOURCE: EN 15308:2016, 3.4, modified – "waste samples" is replaced here with "samples".]

3.5

injection standard

$^{13}\text{C}_{12}$ -labelled PCB or other PCB that is unlikely to be present in samples added to the sample extract before injection into the gas chromatograph, to monitor variability of instrument response and the recovery of the internal standards

[SOURCE: EN 15308:2016, 3.5, modified – "waste samples" is replaced here with "samples".]