Environmental Solid Matrices - Determination of polychlorinated biphenyls (PCB) by gas chromatography - mass selective detection (GC-MS) or electron-capture detection (GC-ECD)

EESTI STANDARDIKESKUS
ESTONIAN CENTRE FOR STANDARDISATION

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 17322:2020 sisaldab Euroopa standardi EN 17322:2020 ingliskeelset teksti.	This Estonian standard EVS-EN 17322:2020 consists of the English text of the European standard EN 17322:2020.
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 29.07.2020.	Date of Availability of the European standard is 29.07.2020.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 13.030.01, 13.030.10, 13.030.20, 13.080.10

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 17322

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ICS 13.030.01; 13.030.10; 13.030.20; 13.080.10

Supersedes EN 15308:2016, EN 16167:2018+AC:2019

English Version

Environmental Solid Matrices - Determination of polychlorinated biphenyls (PCB) by gas chromatography - mass selective detection (GC-MS) or electron-capture detection (GC-ECD)

Matrices solides environnementales - Dosage des polychlorobiphényles (PCB) par chromatographie en phase gazeuse-spectrométrie de masse (CG-SM) ou chromatographie en phase gazeuse avec détection par capture d'électrons (CG-ECD)

Feststoffe in der Umwelt - Bestimmung von polychlorierten Biphenylen (PCB) mittels Gaschromatographie und massenspektrometrischer Detektion (GC-MS) oder Elektronen-Einfang-Detektion (GC-ECD)

This European Standard was approved by CEN on 15 June 2020.

This European Standard was corrected and reissued by the CEN-CENELEC Management Centre on 9 September 2020.

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

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European foreword

This document (EN 17322:2020) 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 January 2021, and conflicting national standards shall be withdrawn at the latest by January 2021.

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 16167:2018+AC:2019 and EN 15308:2016.

This document is the result of the merging of EN 16167:2018+AC:2019 and EN 15308:2016, with minor technical modifications.

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

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 PCB 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, pre-treatment, extraction and 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 document was developed by merging of EN 16167:2018+AC:2019, 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 15308, 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. Detection with both MS-detection and ECD-detection is possible. Two different extraction procedures are described and 9 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 PAH (EN 16181:2018 and EN 15527:2008). 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 European Standard is applicable and validated

Matrix	Materials used for validation
Soil	Sandy soil Mix of soil from the vicinity of Berlin, Germany and PCB-free German reference soil
Sludge	Mix of municipal waste water treatment plant sludge from North Rhine Westphalia, Germany
Biowaste	Mix of compost from the vicinity of Berlin, Germany and sludge from North Rhine Westphalia, Germany
Waste	Contaminated soil, building debris, waste wood, sealant waste, electronic waste, shredder light fraction, cable shredder waste

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.

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

1 Scope

This document specifies methods for quantitative determination of seven selected polychlorinated biphenyls (PCB28, PCB52, PCB101, PCB118, PCB138, PCB153 and PCB180) in soil, sludge, sediment, treated biowaste, and waste 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 document, lower limit of application from 1 µg/kg (expressed as dry matter) for soils, sludge and biowaste to 10 µg/kg (expressed as dry matter) for solid waste can be achieved. For some specific samples the limit of 10 µg/kg cannot be reached.

Sludge, waste and treated biowaste may differ in properties, as well as in the expected contamination levels of PCB 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.

NOTE The analysis of PCB in insulating liquids, petroleum products, used oils and aqueous samples is referred to in EN 61619, EN 12766-1 and EN ISO 6468 respectively.

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

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 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*

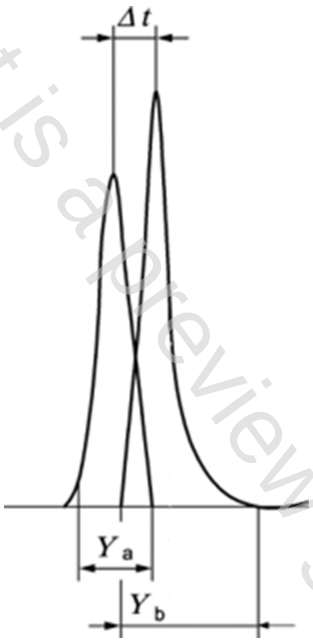
3 Terms and definitions

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 https://www.iso.org/obp/ui

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



$$R = 2 \times \frac{\Delta t}{Ya + Yb} \quad (x) \tag{1}$$

where

R	resolution
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