INTERNATIONAL STANDARD

ISO 18475

First edition
2023-10

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

Reference number
ISO 18475:2023(E)

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by the European Committee for Standardization (CEN) (as EN 17322:2020) and was adopted, without modification other than those given below, by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization.*

— change of EN ISO 5667-15 reference to ISO 5667-15 reference;

— change of EN ISO 16720 reference to ISO 16720 reference;

— change of EN ISO 22892 reference to ISO 22892 reference;

— change of EN ISO 5667-13 reference to ISO 5667-13 reference;

— change of EN ISO 6468 reference to ISO 6468 reference;

— uniform spelling of sulfate and sulfite;

— editorially revised.

This first edition cancels and replaces ISO 10382:2002 and ISO 13876:2013, which have been technically revised.

The main changes are as follows:

— deletion of OCP analysis (this document specifies methods for quantitative determination of polychlorinated biphenyls);

— addition of GC-MS as a detection method;

— extension of the scope to sludge, sediment, treated biowaste and waste;

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— addition of modern extraction techniques and commonly used methods with optimized extraction time, proven clean-up methods and state of the art quantification methods;

— update of normative references.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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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, 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 document 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.

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

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 document

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 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.

ISO 5667-15, *Water quality — Sampling — Part 15: Guidance on the preservation and handling of sludge and sediment samples*

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ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods — Part 1: Linear calibration function*

ISO 16720, *Soil quality — Pretreatment of samples by freeze-drying for subsequent analysis*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

ISO 22892, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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)

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