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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)**

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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 ~~documents~~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](http://www.iso.org/directives)).

Field Code Changed

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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](http://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;~~
- ~~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](http://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.

Considering 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](#) [Table 1](#) (see also [Annex A](#) [Annex A](#) for the results of the validation).

**Table 1 — Matrices for which this document ~~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.



# Environmental ~~Solid Matrices~~ solid matrices — Determination of polychlorinated biphenyls (PCB) by gas chromatography - Massmass 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~~).

Table 2 — Target analytes of this document

Target analyte	CAS-RN <sup>a</sup>
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

<sup>a</sup> 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.

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

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

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

where

$R$  resolution

$\Delta t$  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)

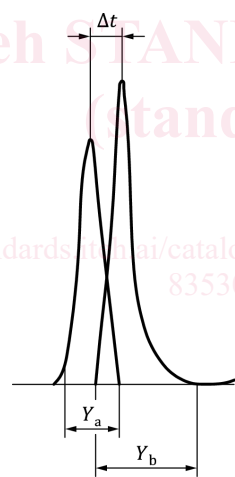
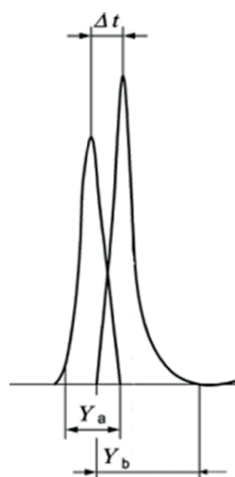


Figure 1 — Example of a chromatogram of a critical pair

**3.2 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.

### 3.3

#### 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

### 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

### 3.5

#### polychlorinated biphenyl

#### PCB

biphenyl substituted with one to ten chlorine atoms

### 3.6

#### sediment

solid material, both mineral and organic, deposited in the bottom of a water body

[SOURCE: ISO 5667-12:2017]

## 4 Principle

Due to the multi-matrix character of this document, different procedures for different steps (modules) are allowed. Which modules should be used depends on the sample. A recommendation is given in this document. Performance criteria are described and it is the responsibility of the laboratories applying this document to show that these criteria are met. Using of spiking standards (internal standards) allows an overall check on the efficiency of a specific combination of modules for a specific sample. But it does not necessarily give the information regarding the extensive extraction efficiency of the native PCB bonded to the matrix.

After pre-treatment, the sample is extracted with a suitable solvent.

The extract is concentrated by evaporation. If necessary, interfering compounds are removed by a clean-up method suitable for the specific matrix, before the concentration step.

The extract is analysed by gas chromatography. The various compounds are separated using a capillary column with a stationary phase of low polarity. Detection occurs by mass spectrometry (MS) or an electron capture detector (ECD).

PCB are identified and quantified by comparison of relative retention times and relative peak heights (or peak areas) with respect to internal standards added. The efficiency of the procedure depends on the composition of the matrix that is investigated.

## 5 Interferences

### 5.1 Interference with sampling and extraction

Use sampling containers of materials (preferably of steel, aluminium or glass) that do not affect the sample during the contact time. Avoid plastics and other organic materials during sampling, sample storage or extraction. Keep the samples from direct sunlight and prolonged exposure to light.

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During storage of the samples, losses of PCB may occur due to adsorption on the walls of the containers. The extent of the losses depends on the storage time.

## 5.2 Interference with GC

Substances that co-elute with the target PCB may interfere with the determination. These interferences may lead to incompletely resolved signals and may, depending on their magnitude, affect accuracy and precision of the analytical results. Peak overlap does not allow an interpretation of the result. Asymmetric peaks and peaks being broader than the corresponding peaks of the reference substance suggest interferences.

Chromatographic separation between the following pairs can be critical.

- ~~—~~PCB28 – PCB31
- ~~—~~PCB52 – PCB73
- ~~—~~PCB101 – PCB89 / PCB90
- ~~—~~PCB118 – PCB106
- ~~—~~PCB138 – PCB164 / PCB163

The critical pair PCB28 and PCB31 is used for selection of the capillary column (see [8.2.2.2.2](#)). If molecular mass differences are present, quantification can be made by mass selective detection. If not or using ECD, the specific PCB is reported as the sum of all PCBs present in the peak. Typically, the concentrations of the co-eluting congeners compared to those of the target congeners are low. When incomplete resolution is encountered, peak integration shall be checked and, when necessary, corrected.

Presence of tetrachlorobenzyltoluene (TCBT)-mixtures or sulfur can disturb the determination of the PCB with GC-ECD.

High mineral oil content can also disturb the determination of PCB with GC-MS.

## 6 Safety remarks

PCBs are highly toxic and shall be handled with extreme care. Avoid contact with solid materials, solvent extracts and solutions of standard PCB. Contact of solutions of standard with the body should be prevented. It is strongly advised that standard solutions are prepared centrally in suitably equipped laboratories or are purchased from suppliers specialized in their preparation.

Solvent solutions containing PCB and samples shall be disposed of in a manner approved for disposal of toxic wastes.

For the handling of hexane precautions shall be taken because of its neurotoxic properties.

National regulations enforcing locally stricter requirements are used with respect to all hazards associated with this method.

## 7 Reagents

### 7.1 General

All reagents shall be of recognized analytical grade. The purity of the reagents used shall be checked by running a blank test as described in 10.1.10.1. The blank shall be less than 50 % of the lowest reporting limit.

### 7.2 Reagents for extraction

7.2.1 **Acetone (2-propanone)**,  $(\text{CH}_3)_2\text{CO}$ .

7.2.2 ***n*-heptane**,  $\text{C}_7\text{H}_{16}$ .

7.2.3 **Petroleum ether**, boiling range 40 °C to 60 °C.

Hexane-like solvents with a boiling range between 30 °C and 98 °C are allowed.

7.2.4 **Sodium sulfate**,  $\text{Na}_2\text{SO}_4$ . The anhydrous sodium sulfate shall be kept carefully sealed.

7.2.5 **Distilled water** or water of equivalent quality,  $\text{H}_2\text{O}$ .

7.2.6 **Sodium chloride**,  $\text{NaCl}$ .

7.2.7 **Keeper substance**. High boiling compound, i.e. octane, nonane.

### 7.3 Reagents for clean-up

#### 7.3.1 Clean-up A using aluminium oxide

7.3.1.1 **Aluminium oxide**,  $\text{Al}_2\text{O}_3$

Basic or neutral, specific surface 200  $\text{m}^2/\text{g}$ , activity Super I (3.3.1.1).  
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7.3.1.2 **Deactivated aluminium oxide**

Deactivated with approximately 10 % water.

Add approximately 10 g of water (7.2.5(7.2.5)) to 90 g of aluminium oxide (7.3.1.1(7.3.1.1)). Shake until all lumps have disappeared. Allow the aluminium oxide to condition before use for some 16 h, sealed from the air, use it for maximum two weeks.

NOTE 1 The activity depends on the water content. It can be necessary to adjust the water content.

NOTE 2 Commercially available aluminium oxides with 10 % mass fraction water can also be used.

#### 7.3.2 Clean-up B using silica gel 60 for column chromatography

7.3.2.1 **Silica gel 60**, particle size 63  $\mu\text{m}$  to 200  $\mu\text{m}$ .

7.3.2.2 **Silica gel 60**, water content: mass fraction  $w(\text{H}_2\text{O}) = 10\%$ .

Silica gel 60 (7.3.2.1(7.3.2.1)), heated for at least 3 h at 450 °C, cooled down and stored in a desiccator containing magnesium perchlorate or a suitable drying agent. Before use heat at least for 5 h at 130 °C in

a drying oven. Then allow cooling in a desiccator and add 10 % water (mass fraction) (7.2.5(7.2.5)) in a flask. Shake for 5 min intensively by hand until all lumps have disappeared and then for 2 h in a shaking device. Store the deactivated silica gel in the absence of air, use it for maximum of two weeks.

### 7.3.3 Clean-up C using gel permeation chromatography (GPC)<sup>1</sup>

7.3.3.1 **Bio-Beads**<sup>®2</sup> S-X3.

7.3.3.2 **Ethyl acetate**, C<sub>4</sub>H<sub>8</sub>O<sub>2</sub>.

7.3.3.3 **Cyclohexane**, C<sub>6</sub>H<sub>12</sub>.

Preparation of GPC, for example: put 50 g Bio-Beads<sup>®</sup> S-X3 (7.3.3.1(7.3.3.1)) into a 500 ml Erlenmeyer flask and add 300 ml elution mixture made up of cyclohexane (7.3.3.3(7.3.3.3)) and ethyl acetate (7.3.3.2(7.3.3.2)) 1:1 (volume fraction) in order to allow the beads to swell; after swirling for a short time until no lumps are left, maintain the flask closed for 24 h. Drain the slurry into the chromatography tube for GPC. After approximately three days, push in the plungers of the column so that a filling level of approximately 35 cm is obtained. To further compress the gel, pump approximately 2 l of elution mixture through the column at a flow rate of 5 ml · min<sup>-1</sup> and push in the plungers to obtain a filling level of approximately 33 cm.

### 7.3.4 Clean-up D using Florisil<sup>®3</sup>

7.3.4.1 **Florisil**<sup>®</sup>, baked 2 h at 600 °C. Particle size 150 µm to 750 µm.

7.3.4.2 **Iso-octane**, C<sub>8</sub>H<sub>18</sub>.

7.3.4.3 **Toluene**, C<sub>7</sub>H<sub>8</sub>.

7.3.4.4 **Iso-octane/Toluene** 95/5 volumetric fraction

### 7.3.5 Clean-up E using silica H<sub>2</sub>SO<sub>4</sub>/silica NaOH

7.3.5.1 **Silica**, SiO<sub>2</sub>, particle size 70 µm to 230 µm, baked at 180 °C for a minimum of 1 h, and stored in a pre-cleaned glass bottle with screw cap that prevents moisture from entering.

7.3.5.2 **Sulfuric acid H<sub>2</sub>SO<sub>4</sub>** 95 – 97 % percent mass fraction

7.3.5.3 **Silica**, treated with sulfuric acid.

Mix 56 g silica (7.3.5.1(7.3.5.1)) and 44 g sulfuric acid (7.3.5.2(7.3.5.2)).

7.3.5.4 **Sodium hydroxide solution**, c(NaOH) = 1 mol/l.

<sup>1</sup> GPC is also known as SEC (size exclusion chromatography).

<sup>2</sup> Bio-Beads<sup>®</sup> is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products can be used if they can be shown to lead to the same results.

<sup>3</sup> Florisil<sup>®</sup> is a trade name for a prepared diatomaceous substance, mainly consisting of anhydrous magnesium silicate. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products can be used if they can be shown to lead to the same results.