

Tuhé environmentálne matrice Stanovenie polychlórovaných bifenylov (PCB) plynovou chromatografiou – hmotnostným spektrometrom (GC-MS) alebo s detektorom elektrónového záchytu (GC-ECD) (ISO 18475: 2023)

**STN EN ISO 18475** 

83 8265

Environmental solid matrices - Determination of polychlorinated biphenyls (PCB) by gas chromatography - mass selective detection (GC-MS) or electron-capture detection (GC-ECD) (ISO 18475:2023)

Táto norma obsahuje anglickú verziu európskej normy. This standard includes the English version of the European Standard.

Táto norma bola oznámená vo Vestníku ÚNMS SR č. 10/25

Obsahuje: EN ISO 18475:2025, ISO 18475:2023

Oznámením tejto normy sa ruší STN EN 17322 (83 8265) z decembra 2020

### EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

**EN ISO 18475** 

July 2025

ICS 13.080.10

Supersedes EN 17322:2020

#### **English Version**

## Environmental solid matrices - Determination of polychlorinated biphenyls (PCB) by gas chromatography - mass selective detection (GC-MS) or electron-capture detection (GC-ECD) (ISO 18475:2023)

Matrices solides environnementales - Dosage des polychlorobiphenyles (PCB) par chromatographie en phase gazeuse-spectrometrie de masse (CG-SM) ou chromatographie en phase gazeuse avec detection par capture d'electrons (CG-ECD) (ISO 18475:2023) Feststoffe in der Umwelt - Bestimmung von polychlorierten Biphenylen (PCB) mittels Gaschromatographie und massenspektrometrischer Detektion (GC-MS) oder Elektronen-Einfang-Detektion (GC-ECD) (ISO 18475:2023)

This European Standard was approved by CEN on 13 July 2025.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of 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, Türkiye and United Kingdom.



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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

#### EN ISO 18475:2025 (E)

Contents	Page
European foreword	2
EUTOPEAN 10TEWOTU	

EN ISO 18475:2025 (E)

#### **European foreword**

The text of ISO 18475:2023 has been prepared by Technical Committee ISO/TC 190 "Soil quality" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 18475:2025 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 2026, and conflicting national standards shall be withdrawn at the latest by January 2026.

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 17322:2020.

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organizations 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, Türkiye and the United Kingdom.

#### **Endorsement notice**

The text of ISO 18475:2023 has been approved by CEN as EN ISO 18475:2025 without any modification.

### 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)



#### **COPYRIGHT PROTECTED DOCUMENT**

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office CP 401 • Ch. de Blandonnet 8 CH-1214 Vernier, Geneva Phone: +41 22 749 01 11 Email: copyright@iso.org Website: www.iso.org Published in Switzerland

Coı	Contents					
Fore	word	rences 4 Interference with sampling and extraction 4 Interference with GC 4 remarks 5				
Intr	oductio	n	vii			
1	Scone	Δ	1			
	-					
2						
3	Term	is and definitions	2			
4	Princ	Principle				
5	Inter	Interferences				
	5.1					
	5.2	Interference with GC	4			
6	Safet	y remarks	5			
7	Reag	ents	5			
	7.1	General	5			
	7.2	Reagents for extraction				
	7.3	Reagents for clean-up				
		<ul><li>7.3.1 Clean-up A using aluminium oxide</li><li>7.3.2 Clean-up B using silica gel 60 for column chromatography</li></ul>				
		7.3.3 Clean-up C using gel permeation chromatography (GPC)	0 6			
		7.3.4 Clean-up D using Florisil®				
		7.3.5 Clean-up E using silica H <sub>2</sub> SO <sub>4</sub> /silica NaOH	7			
		7.3.6 Clean-up F using benzenesulfonic acid/sulfuric acid	7			
		7.3.7 Clean-up G using TBA sulfite reagent				
		7.3.8 Clean-up H using pyrogenic copper				
	7.4	7.3.9 Clean-up I using silica/silver nitrate Gas chromatographic analysis				
	7. <del>4</del> 7.5	Standards				
	7.5	7.5.1 General				
		7.5.2 Calibration standards				
		7.5.3 Internal and injection standards				
	7.6	Preparation of standard solutions				
		7.6.1 Preparation of calibration standard solutions of PCBs				
		<ul><li>7.6.2 Preparation of internal standard solution</li><li>7.6.3 Preparation of injection standard solution</li></ul>				
8	<b>Appa</b> 8.1	ratus Extraction and clean-up procedures				
	8.2	Gas chromatograph				
0						
9	<b>Sam</b> p 9.1	ole storage and preservation Sample storage				
	9.1	Sample pre-treatment				
10						
10	10.1	edure Blank test				
	10.1	Extraction				
	10.2	10.2.1 General				
		10.2.2 Extraction procedure 1: Samples using acetone/petroleum ether or				
		hexane-like solvent and agitation or sonication	14			
		10.2.3 Extraction procedure 2: Samples using Soxhlet or pressurized liquid	4 =			
		extraction extraction procedure 3: Samples using acetone/petroleum ether or	15			
		hexane-like solvent/sodium chloride and agitation	15			
	10.3	Concentration				
	10.4					

		10.4.1	General	16
			Clean-up A – Aluminium oxide	
			Clean-up B – Silica gel	
		10.4.4	Clean-up C – Gel permeation chromatography	17
		10.4.5	Clean-up D – Florisil®	18
		10.4.6	Clean-up E – Silica H <sub>2</sub> SO <sub>4</sub> /silica NaOH	18
		10.4.7	Clean-up F – Benzenesulfonic acid/sulfuric acid	18
		10.4.8	Clean-up G – TBA sulfite reagent	
		10.4.9	Clean-up H – Clean-up using pyrogenic copper to remove elemental sulfur	
			and some other organic sulfur compounds	
			Clean up I – AgNO <sub>3</sub> /silica	
	10.5		on of the injection standard	
	10.6		romatographic analysis (GC)	
			General	
			Setting the gas chromatograph	
	10.7		spectrometry (MS)	
			Mass spectrometric conditions	
		10.7.2	Calibration of the method using an internal standard	20
			Measurement	
			Identification	
			Check on method performance	
			Calculation	
	10.8		on capture detection (ECD)	
			General	
			ECD conditions	
11	Perfo	rmance	e characteristics	25
12	Preci	ision		25
13	Test	report		26
Anne	<b>x A</b> (in	- formativ	ve) Repeatability and reproducibility data	27
	-		ve) Examples for gas chromatographic conditions and retention times	le
AIIII C			e Examples for gas chromatographic conditions and retention times	32
Anne	<b>x C</b> (inf	formativ	re) Calculation method for the estimation of total PCB content	33
Sibliography4				40
	iography40			

#### **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 <a href="www.iso.org/directives">www.iso.org/directives</a>).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at <a href="www.iso.org/patents">www.iso.org/patents</a>. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

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 <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

#### 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 <u>Table 1</u> (see also <u>Annex A</u> for the results of the validation).

 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

Table 1 — Matrices for which this document is applicable and validated

**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.

# 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 <u>Table 2</u>).

	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.			

Table 2 — Target analytes of this document

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  $\mu$ g/kg (expressed as dry matter) for soils, sludge and biowaste to 10  $\mu$ g/kg (expressed as dry matter) for solid waste can be achieved. For some specific samples the limit of 10  $\mu$ g/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

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

koniec náhľadu – text ďalej pokračuje v platenej verzii STN