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BSI Standards Publication

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 (GCECD)

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National foreword

This British Standard is the UK implementation of EN 16167:2018, incorporating corrigendum January 2019. It supersedes BS EN 16167:2012, which is withdrawn.

The start and finish of text introduced or altered by corrigendum is indicated in the text by tags. Text altered by CEN corrigendum January 2019 is indicated in the text by [AC⟩ ⟨AC].

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16167

August 2018

ICS 13.030.01; 13.080.10

Supersedes EN 16167:2012

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 (CG-SM) ou un détecteur par capture d'électrons (CG-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].

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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 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.

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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*

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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
$^{13}C_{12}$-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
$^{13}C_{12}$-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".]

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4 Principle

Due to the horizontal character of this European Standard, different procedures for different steps (modules) are allowed. Which modules should be used depends on the sample. A recommendation is given in this European Standard. Performance criteria are described and it is the responsibility of the laboratories applying this European Standard 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 upon the extensive extraction efficiency of the native PCB bonded to the matrix.

After pretreatment according to the methods referred to in 9.2, the test 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. The eluate is concentrated by evaporation.

The extract is analysed by gas chromatography with either mass spectrometric (GC-MS) or electron capture detection (GC-ECD). Applying GC-MS the various compounds are separated using a capillary column with a stationary phase of low polarity. In case of GC-ECD, extracts are analysed using two columns of different polarity (see 8.2.1).

PCBs 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 change 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.

During storage of the samples, losses of PCBs 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. The critical pair PCB28 and PCB31 is used for selection of the capillary column (see 8.2.2). 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.

— PCB28 – PCB31

— PCB52 – PCB73

— PCB101 – PCB89 / PCB90

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— PCB118 – PCB106

— PCB138 – PCB164 / PCB163

Presence of considerable amounts of mineral oil in the sample may interfere with the quantification of PCB in GC-MS. In presence of mineral oil, GC-ECD may be preferred or mineral oil can be removed using clean-up procedure G (see 10.4.8) using DMF/*n*-hexane.

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

6 Safety remarks

PCBs are highly toxic and shall be handled with extreme care. Contact between the body and solid materials, solvent extracts and solutions of standard PCB shall not be allowed to occur. 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 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 shall be followed with respect to all hazards associated with this method.

7 Reagents

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. The blank shall be less than 50 % of the lowest reporting limit.

7.1 Reagents for extraction

7.1.1 Propanone (Acetone), $(CH_3)_2$ CO.

7.1.2 *n*-heptane, C_7H_{16}.

7.1.3 Petroleum ether, boiling range 40 °C to 60 °C.

Hexane-like solvents with a boiling range between 40 °C and 89 °C are allowed.

7.1.4 Anhydrous sodium sulfate, Na_2SO_4:

The anhydrous sodium sulfate shall be kept carefully sealed.

7.1.5 Distilled water or water of equivalent quality, H_2O.

7.1.6 Sodium chloride, NaCl, anhydrous.

7.1.7 Keeper substance. High boiling compound, i.e. octane, nonane.

7.2 Reagents for clean-up:

7.2.1 Clean-up A using aluminium oxide:

7.2.1.1 Aluminium oxide, Al_2O_3.

Basic or neutral, specific surface 200 m^2/g, activity Super I according to Brockmann.

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7.2.1.2 Deactivated aluminium oxide

Deactivated with approximately 10 % water.

Add approximately 10 g of water (7.1.5) to 90 g of aluminium oxide (7.2.1.1). 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 The activity depends on the water content. It can be necessary to adjust the water content.

7.2.2 Clean-up B using silica gel 60 for column chromatography

7.2.2.1 Silica gel 60, particle size 63 µm to 200 µm.

7.2.2.2 Silica gel 60, water content: mass fraction $w(H_2O)$ = 10 %.

Silica gel 60 (7.2.2.1), 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) 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.

7.2.3 Clean-up C using gel permeation chromatography (GPC)

7.2.3.1 Bio-Beads® S-X3[1].

7.2.3.2 Ethyl acetate, $C_4H_8O_2$.

7.2.3.3 Cyclohexane, C_6H_{12}.

Preparation of GPC, for example: Put 50 g Bio-Beads® S-X3 (7.2.3.1) into a 500 ml Erlenmeyer flask and add 300 ml elution mixture made up of cyclohexane (7.2.3.3) and ethyl acetate (7.2.3.2) 1:1 (volume) 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^{-1} and push in the plungers to obtain a filling level of approximately 33 cm.

7.2.4 Clean-up D using Florisil®[2].

7.2.4.1 Florisil®, baked 2 h at 600 °C. Particle size 150 µm to 750 µm.

7.2.4.2 Iso-octane, C_8H_{18}.

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1) Bio-Beads® 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 CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

2) Florisil® 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 European Standard and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

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7.2.4.3 Toluene, C_7H_8.

7.2.4.4 Iso-octane/Toluene 95/5 (v/v).

7.2.5 Clean-up E using silica H_2SO_4/silica NaOH.

7.2.5.1 Silica, SiO_2, 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.

7.2.5.2 Silica, treated with sulfuric acid.

Mix 56 g silica (7.2.5.1) and 44 g sulfuric acid (7.2.8.1).

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

7.2.5.4 Silica, treated with sodium hydroxide.

Mix 33 g silica (7.2.5.1) and 17 g sodium hydroxide (7.2.5.3).

7.2.5.5 *n*-hexane, C_6H_{14}.

7.2.6 Clean-up F using benzenesulfonic acid/sulfuric acid

7.2.6.1 3 ml silica gel column, of adsorbent mass 500 mg, particle size 40 µm.

7.2.6.2 3 ml benzenesulfonic acid/sulfuric acid column, of adsorbent mass 500 mg, particle size 40 µm.

7.2.7 Clean-up G using DMF/hexane partitioning

7.2.7.1 Dimethylformamide(DMF), C_3H_7NO.

7.2.8 Clean-up H using concentrated sulfuric acid

7.2.8.1 Sulfuric acid, H_2SO_4 of purity 96 % to 98 % (mass fraction).

7.2.9 Clean-up I using TBA sulfite reagent

7.2.9.1 Tetrabutylammonium reagent (TBA sulfite reagent)

Saturate a solution of tetrabutylammonium hydrogen sulfate in a mixture of equal volume of water and 2-propanol, $c((C_4H_9)_4NHSO_4)$ = 0,1 mol/l, with sodium sulfite.

25 g of sodium sulfite should be sufficient for 100 ml of solution.

7.2.9.2 2-Propanol, C_3H_8O.

7.2.9.3 Sodium sulfite, Na_2SO_3.

7.2.10 Clean-up J using pyrogenic copper

WARNING — Pyrogenic copper is spontaneously inflammable. Suitable precautions shall be taken.

7.2.10.1 Copper(II)-sulfate pentahydrate, $CuSO_4 \cdot 5\ H_2O$.

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7.2.10.2 Hydrochloric acid, *c*(HCl) = 2 mol/l.

7.2.10.3 Zinc granules, Zn, particle size 0,3 mm to 1,4 mm.

7.2.10.4 Anionic detergent aqueous solution (e.g. 35 g/100 ml, n-dodecane-1-sulfonic acid sodium salt ($CH_3(CH_2)_{11}SO_3Na$).

Other commercially available detergents may also be suitable.

7.2.10.5 Deoxygenated water

Water from which dissolved O_2 has been removed, e.g. by stripping with an inert gas, or by adequate membrane filtration.

7.2.10.6 Pyrogenic copper

Dissolve 45 g copper(II)-sulphate pentahydrate (7.2.10.1) in 480 ml water containing 20 ml hydrochloric acid (7.2.10.2) in a 1 000 ml beaker.

Take 15 g of zinc granules size (7.2.10.3), add 25 ml water and one drop of anionic detergent solution (7.2.10.4) in another 1 000 ml beaker.

Stir with a magnetic stirrer at a high speed to form a slurry. Then whilst stirring at this high speed, carefully add the copper(II)-sulphate solution drop by drop using a glass rod.

Hydrogen is liberated and elemental pyrogenic copper is precipitated (red coloured precipitate).

Stirring is continued until the hydrogen generation almost ceases. Then the precipitated copper is allowed to settle. The supernatant water is carefully removed and the product washed with deoxygenated water (7.2.10.5) three times, to eliminate residual salts.

Then the water is carefully replaced with 250 ml acetone (7.1.1) (whilst continuously stirring the mixture). This operation is repeated twice more to ensure elimination of water.

Then the above procedure is repeated three times with 250 ml hexane (7.2.5.5), to ensure elimination of the acetone.

Carefully transfer the copper with hexane into an Erlenmeyer flask and store under hexane. The flask shall be sealed to prevent ingress of air and stored in an explosion-proof refrigerator 2 °C to 8 °C.

The shelf life of the pyrogenic copper is at least two months. The clean-up efficiency then declines. The copper changes colour as the clean-up efficiency decreases.

7.2.11 Clean-up K using silica/silver nitrate

7.2.11.1 Silver nitrate, $AgNO_3$.

7.2.11.2 Silver nitrate/silica adsorbent

Dissolve 10 g of $AgNO_3$ (7.2.11.1) in 40 ml water and add this mixture in portions to 90 g of silica (7.2.5.1). Shake the mixture until it is homogenous and leave it for 30 min. Put the mixture into a drying oven at (70 ± 5) °C. Within 5 h regular increase the temperature from 70 °C to 125 °C. Activate the mixture for 15 h at 125 °C. Store the mixture in brown glass bottles.

7.3 Gas chromatographic analysis

Operating gases for gas chromatography/ECD or MS, of high purity and in accordance with the manufacturer's specifications.

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