
Karakterizacija odpadkov - Določevanje izbranih polikloriranih bifenilov (PCB) v trdnih odpadkih s kapilarno plinsko kromatografijo z detektorjem z zajemom elektronov (ECD) ali z masno spektrometrično detekcijo

Characterization of waste - Determination of selected polychlorinated biphenyls (PCB) in solid waste by using capillary gas chromatography with electron capture or mass spectrometric detection

Charakterisierung von Abfällen - Bestimmung ausgewählter polychlorierter Biphenyle (PCB) in festem Abfall unter Anwendung der Kapillar-Gaschromatographie mit Elektroneneinfang-Detektion oder massenspektrometrischer Detektion

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Caractérisation des déchets - Détermination de polychlorobiphényles (PCB) sélectionnés dans les déchets solides par chromatographie en phase gazeuse capillaire avec détection par capture d'électrons ou spectrométrie de masse

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This European Standard was approved by CEN on 28 December 2007.

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Foreword

This document (EN 15308:2008) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, 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of an identical text or by endorsement, at the latest by August 2008, and conflicting national standards shall be withdrawn at the latest by August 2008.

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EN 15308:2008 (E)**1 Scope**

This document specifies a method for quantitative determination of seven polychlorinated biphenyl congeners (PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180) in solid waste using high-resolution gas chromatography with electron capture or mass spectrometric detection. The basic content of this standard is identical to that of the Horizontal PCB-standard and is therefore also applicable to soil, sludge and treated bio-waste. The detection and the quantification limits in this method are dependent on sample intake, the level of interferences as well as instrumental limitations. Under the conditions specified in this standard, minimum concentrations of individual PCB congeners equal or above 0,01 mg/kg dry matter can typically be determined with no interferences present.

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

NOTE 2 The method may be applied to the analysis of other PCB congeners not specified in the scope, but its suitability should be proven by proper in-house validation experiments.

2 Normative references

The following referenced documents are indispensable for the application 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 14346, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

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3 Terms and definitions

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

3.1

polychlorinated biphenyl

PCB

biphenyl substituted by one to ten chlorine atoms

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

The IUPAC congener numbers are for easy identification; they do not represent the order of chromatographic elution.

3.3

calibration standard

solution prepared from a secondary standard and/or stock solutions of native polychlorinated biphenyl congeners (PCB) and used to calibrate the response of the instrument with respect to analyte concentration

3.4

internal standard

¹³C₁₂-labelled PCB or other PCB that are unlikely to be present in waste samples added to the sample before extraction and used for quantification of PCB content

3.5**injection standard**

$^{13}\text{C}_{12}$ -labelled PCB or other PCB that is unlikely to be present in waste 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

3.6**critical pair**

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

3.7**resolution** R_s

difference in retention times between the maxima of the two peaks i and j , which constitute the critical pair, divided by the arithmetic mean of the peak widths of i and j at base, calculated as:

$$R_s = \frac{2(t_{Rj} - t_{Ri})}{(w_{bi} + w_{bj})} \quad (1)$$

where t_{Rj} and t_{Ri} are the retention times of the two peaks i and j (sec), and w_{bi} and w_{bj} are the peak widths of i and j (sec) at base

4 Principle**iTeh STANDARD PREVIEW****(standards.iteh.ai)**

A proper test portion is extracted with a mixture of organic solvents by an appropriate extraction technique (e. g. shaking, soxhlet, sonication), partitioned against water and the organic layer separated. The obtained extracts are concentrated and, if appropriate, cleaned up. Sample cleanup procedures may include sulphuric acid treatment, Dimethylsulfoxide/n-Hexan partitioning, column chromatography on alumina and silica. Tetrabutylammonium sulfite (TBA) or copper may be used to remove sulfur if required. The extract is analyzed by gas chromatography with either mass spectrometric (GC-MS) or electron capture detection (GC-ECD). In case of GC-MS analysis quantification is performed by the isotope dilution technique. In case of GC-ECD, extracts are analyzed using two columns of different polarity. Quantification is conducted by the internal standard method.

NOTE In case of plastic shredder a pure non-polar solvent should be used for extraction to prevent dissolving of the polymer matrix as far as possible.

5 Reagents**5.1 General**

All reagents shall be of recognized analytical grade. Running a blank determination as described in 11.1 shall check the purity of the reagents used.

5.2 Reagents for extraction and drying**5.2.1 Acetone.****5.2.2 Hexane like solvents with a boiling range of 36 °C to 98 °C, e. g. petroleum ether, n-hexane, n-heptane.**

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5.2.3 Anhydrous sodium sulfate, heated for at least 6 h to $550\text{ °C} \pm 20\text{ °C}$, cooled to about 200 °C in the furnace and then to ambient temperature in a desiccator containing magnesium perchlorate or another suitable drying reagent. The anhydrous sodium sulfate shall be kept carefully sealed.

5.3 Reagents for clean-up procedures

5.3.1 Aluminium oxide clean-up

5.3.1.1 One of two types of alumina, acidic or basic, can be used in the clean-up of sample extracts:

- acidic alumina, activate by heating to 130 °C for a minimum of 12 h;
- basic alumina, activate by heating to 600 °C for a minimum of 24 h.

Preparation of deactivated aluminium oxide, the aluminium oxide is deactivated with 10 % water. To 90 g of aluminium oxide (5.3.1.1) add 10 g of water. Shake until all lumps have disappeared. Allow the aluminium oxide to condition before use for some 16 h, sealed from the air.

NOTE ICN Alumina Super I or an equivalent may be used without activation.

5.3.2 TBA sulfite reagent for sulfur removal

5.3.2.1 Tetrabutylammonium reagent (TBA sulfite reagent); saturate a solution of tetrabutylammonium hydrogen sulfate in a mixture of equal volumina of water and 2-propanol, $c((\text{C}_4\text{H}_9)_4\text{NHSO}_4) = 0,1\text{ mol/l}$, with sodium sulfite.

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

5.3.3 Pyrogenic copper for sulfur removal SIST EN 15308:2008

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

5.3.3.1 Copper-(II)sulfate pentahydrate, $\text{Cu}_5\text{SO}_4 \cdot 5\text{ H}_2\text{O}$.

5.3.3.2 Hydrochloric acid, HCl, $c = 2\text{ mol/l}$.

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

5.3.3.4 Anionic detergent aqueous solution (e. g. 35 % *m/V* n-dodecane-1-sulfonic acid sodium salt ($\text{CH}_3(\text{CH}_2)_{11}\text{SO}_3\text{Na}$)).

5.3.3.5 Deoxygenated water.

5.3.4 Silica – silver nitrate clean up

5.3.4.1 Silica gel (60 μm to 200 μm).

5.3.4.2 Silver nitrate, AgNO_3 .

5.3.4.3 Preparation of silica gel impregnated with silver nitrate.

Dissolve 10 g of AgNO_3 in 40 ml of water and give this mixture in portions to 90 g silica. Shake the mixture until it is homogenous and leave standing it for 30 min. Put the mixture into a drying oven at 70 °C . Within 5 h increase the temperature from 70 °C up to 120 °C . Activate the mixture for 15 h at 125 °C . Store the mixture in brown glass bottles.

5.3.5 Commercially available benzenesulfonic acid/silica gel cartridges, 3 ml**5.3.6 Dimethylsulfoxide/n-hexane partitioning**

5.3.6.1 Dimethylsulfoxide (DMSO).

5.3.6.2 n-hexane.

5.3.7 Sulphuric acid clean-up

5.3.7.1 Sulphuric acid, 95 % to 97 %.

5.3.8 Silica gel/sulphuric acid clean-up

5.3.8.1 Silica/H₂SO₄ 44 %: Pour 28 g of activated silica and 22 g of sulfuric acid in a flask, stopper air tight and shake thoroughly until disappearance of all agglomerates.

5.3.8.2 Silica/NaOH 33% 1N: pour 33,5 g of activated silica and 16,5 g 1N NaOH in a flask, stopper air tight and shake thoroughly until disappearance of all agglomerates.

5.3.9 Commercially available silica cartridges, 3 ml or 6 ml**5.3.10 Florisil clean up**

5.3.10.1 Florisil¹⁾ 100 mesh to 200 mesh, activated by heating to 600 °C for a minimum of 2 h.

5.3.10.2 Iso-octane.

5.3.10.3 Iso-octane/Toluene 95/5. [SIST EN 15308:2008
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5.4 Reagents for gas chromatographic analysis

5.4.1 Operating gases for gas chromatography, of high purity and in accordance with manufacturer's specifications.

5.5 Standard compounds**5.5.1 Calibration standards**

Use the following PCB for the calibration:

- PCB-28: 2,4,4'-trichlorobiphenyl (CAS number: 7012-37-5);
- PCB-52: 2,2',5,5'-tetrachlorobiphenyl (CAS number: 35693-99-3);
- PCB-101: 2,2',4,5,5'-pentachlorobiphenyl (CAS number: 37680-37-2);
- PCB-118: 2,3',4,4',5-pentachlorobiphenyl (CAS number: 31508-00-6);
- PCB-138: 2,2',3,4,4',5'-hexachlorobiphenyl (CAS number: 35056-28-2);

1) 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 it can be shown to lead to comparable results.

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- PCB-153: 2,2',4,4',5,5'-hexachlorobiphenyl (CAS number: 35065-27-1);
- PCB-180: 2,2',3,4,4',5,5'-heptachlorobiphenyl (CAS number: 35065-29-3).

5.5.2 Internal and injection standards**5.5.2.1 MS detection**

The labelled PCB congeners to be considered as internal standards are listed below.

- ¹³C-2,4,4'-trichlorobiphenyl (PCB-28);
- ¹³C-2,2',5,5'-tetrachlorobiphenyl (PCB-52);
- ¹³C-2,2',4,5,5'-pentachlorobiphenyl (PCB-101);
- ¹³C-2,3',4,4',5-pentachlorobiphenyl (PCB-118);
- ¹³C-2,2',3,4,4',5'-hexachlorobiphenyl (PCB-138);
- ¹³C-2,2',4,4',5,5'-hexachlorobiphenyl (PCB-153);
- ¹³C-2,2',3,4,4',5,5'-heptachlorobiphenyl (PCB-180).

¹³C-2,2',3,3',5,5',6-heptachlorobiphenyl (PCB-178), other ¹³C-labelled PCB or PCB that are unlikely to be present in waste samples may be used as internal and injection standards as well.

NOTE The application of isotopic dilution mass spectrometry is recommended but adding ¹³C-labelled internal standards to the test portion before extraction is associated with high costs. Hence, if only an aliquot of the extract is subjected to the clean-up adding ¹³C-labelled internal standards to this aliquot might be a good option to ensure high quality of the analysis and to reduce costs.

5.5.2.2 ECD detection

Also for ECD-detection internal and injection standards shall be added. Use at least one of the following standards unlikely to be present in waste samples and not interfering with the analytes as internal standard

- PCB-29 – 2,4,5-trichlorobiphenyl (CAS number: 15862-07-4);
- PCB-30 – 2,4,6-trichlorobiphenyl (CAS number: 35693-92-6);
- PCB-143 - 2,2',3,4,5,6'-hexachlorobiphenyl (CAS number: 68194-15-0);
- PCB-155 - 2,2',4,4',6,6'-hexachlorobiphenyl (CAS number: 33979-03-2);
- PCB-198 - 2,2',3,3',4,5,5',6,-octachlorobiphenyl (CAS number: 68194-17-2);
- PCB-207 - 2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl (CAS number: 52663-79-3);
- PCB-209 - 2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl (CAS number: 2051-24-3).

NOTE For quantification (internal standard) PCB198 and PCB209 are recommended because of little interferences found in practice. Use of PCB 30 and PCB 209 for relative retention is recommended see EN 12766-1.

5.5.2.3 PCB congeners for resolution check

- PCB-28 – 2,4,4'-trichlorobiphenyl (CAS number: 7012-37-5);

— PCB-31 – 2,4',5-trichlorobiphenyl (CAS number: 16862-07-4).

6 Apparatus

6.1 Extraction and clean-up procedures

6.1.1 General

Customary laboratory glassware.

All glassware and material that comes into contact with the sample or extract shall be free of PCB and interfering compounds.

6.1.2 Extraction procedures

6.1.2.1 Glass sample bottles of proper size according to the amount of sample taken with glass stopper or screw top and polytetrafluorethene seal (PTFE).

6.1.2.2 Shaking device.

With horizontal movement (200 min⁻¹ to 300 min⁻¹).

6.1.2.3 Water bath, adjustable up to 100 °C.

6.1.2.4 Separatory funnels of appropriate volume.

6.1.2.5 Conical flasks of appropriate volume.

6.1.2.6 Soxhlet extraction apparatus.

Consisting of round bottom flask e. g. 100 ml, Soxhlet extractors and soxhlet thimbles e. g. 27 mm × 100 mm, vertical condensers e. g. 300 mm, with water-bath or heating mantle as heating device.

6.1.2.7 Evaporator.

Kuderna Danish or other evaporators, e. g. a rotary evaporator.

6.1.2.8 Analytical balance.

6.1.3 Clean-up procedures

6.1.3.1 Quartz wool or silanized glass wool.

WARNING — Working with quartz wool imposes a risk to health through the release of fine quartz particles. Inhalation of these should be prevented by using a fume cupboard and wearing a dust mask.

6.1.3.2 Boiling chips, Glass or porcelain beads.

6.1.3.3 Calibrated test tubes with a capacity of 10 ml to 15 ml and ground glass stopper.

6.1.3.4 Glass chromatography columns of e. g. 600 mm length and 5 mm internal diameter.

6.1.3.5 Empty SPE-cartridge of e. g. 6 ml volume.

EN 15308:2008 (E)**6.2 Gas chromatograph**

Equipped with a capillary column, mass spectrometric detector (MS) or electron capture detector (ECD) based on ^{63}Ni . The injector shall be either an on-column injector, a split/splitless injector or a programmable temperature vaporiser injector.

NOTE 1 Working with an encapsulated radioactive source, as present in an ECD requires a license according to the appropriate national radiation protection regulations.

NOTE 2 Using ECD, gas chromatographs equipped with two detectors and with facilities for connecting two capillary columns to the same injection system are very well suited for this analysis; with such apparatus the confirmatory analysis can be performed simultaneously.

6.3 Capillary columns

Comprising a non-polar stationary phase, e. g. 5 % phenyl-methyl silicone, coated onto a fused silica capillary column or an equivalent chemically bonded phase column. Use a chromatogram of a standard solution containing PCB-28 and PCB-31 (5.5.2.3) at equal concentrations run under identical conditions as samples for the determination of the resolution of the critical pair PCB-28/PC-B31. The resolution of this pair shall be better than 0,5. In general column lengths should be 25 m to 60 m, internal diameter 0,18 mm to 0,32 mm and film thickness 0,1 μm to 0,5 μm .

Using ECD-detection, a column coated with a moderate polar phase, e. g. CP-Sil 19, OV 1701 etc., shall be used to confirm the result obtained on the non-polar column. Confirmation analysis using a second column is not necessary in case the analytical result is much below any regulatory level.

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7 Safety precautions

Anyone dealing with waste and sludge analysis has to be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples may contain hazardous (e. g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction. Consequently it is recommended that these samples should be handled with special care. The gases, which may be produced by microbiological or chemical activity, are potentially flammable and will pressurise sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

8 Interferences

Some organic chlorinated compounds (e. g. tetrachlorobenzyltoluenes, polychlorinated naphthalenes, polychlorinated terphenyls, toxaphenes, also sulphur) give rise to interferences since their behaviour is very similar to that of PCB during sample clean-up and gas chromatographic separation. It is possible that several PCB congeners elute within one peak. On many capillary columns PCB-138 coelutes with PCB-160, PCB-163 and PCB-164. Hence PCB-138 concentration may represent the sum of those congeners (also in case of mass spectrometric detection) The same is true for PCB-101 and PCB-90. Typically the concentrations of the co-eluting congeners compared to those of the target congeners are low.

Presence of considerable amounts of mineral oil in the sample may interfere with the quantification of PCB in GC-MS analysis. In such cases, GC-ECD may be the preferred method or clean-up of the sample extract according to 11.3.8 using DMSO/n-hexane partitioning to remove the mineral oil from sample extract is recommended.

9 Sample storage

In principle, the samples shall be analyzed as soon as possible after sampling. This applies in particular to the examination of microbiologically active solids. Field moist samples can be stored at a temperature of

4 °C ± 2 °C in sample containers in a dark place for a maximum of one week. If the sample cannot be processed within seven days, it has to be stored at temperatures below –18 °C. Dried samples shall be stored at room temperature in a dark place.

10 Pre-treatment

10.1 General

The goal of a pre-treatment procedure is to prepare a test sample in which the content of the PCB congeners is not significantly changed compared to the laboratory sample. Due to the different properties of the various kinds of materials there is no general procedure available. Recommendations for sample pre-treatment are given in EN 15002 and ISO 14507.

10.2 Drying

Depending on the nature of the sample material and the extraction solvent to be used a drying step might be needed. If it is necessary, air-dry the complete sample or dry it in a ventilated drying oven at 40 °C or in a freeze dryer. The drying time depends on the technique chosen and the nature of the sample.

For sludge, freeze-drying is strictly applied.

10.3 Particle size reduction

In order to achieve a homogeneous and representative test portion, one or more particle-size reduction steps might be needed. The choice of the technique depends on the nature of the sample and on the particle size needed. Typically, particle-size reduction is a multi-step operation that implies the use of a sequence of different techniques like crushing, cutting or grinding.

Grinding of samples which have a plastic or paste-like consistency requires embrittlement with liquid nitrogen and particle size reduction to less than 0,5 mm, e. g. by using an ultra-centrifugal mill.

NOTE In case of plastic materials (e. g. cable shredder) the material to be granulated is poured in portions into liquid nitrogen and ground in a centrifugal mill (10 000 rounds/min to 15 000 rounds/min) cooled with liquid nitrogen. It should be noted that the mill is constantly kept cool by addition of liquid nitrogen in short intervals. The plastic material should be filled into the mill constantly in small portions without causing any significant slowing down of the grinder.

11 Procedure

11.1 Blank

Perform a blank determination following the paragraphs of the procedure applied to samples (selected extraction and clean-up). Use the same amounts of reagents that are used for pre-treatment, extraction, clean up and analysis of samples. Analyse the blank immediately prior to analysis of the samples to demonstrate freedom from contamination.

11.2 Extraction

11.2.1 Extraction by shaking or sonification

Weigh a test portion of 10 g to 25 g to the nearest 0,1 g in a stoppered flask. Add a definite volume of the internal standard solution. Isotopically labelled internal standards may be added after extraction to an aliquot of the extract where appropriate. Typically the concentration of the individual internal standards in the final extract will be 0,1 µg/ml. Add 50 ml of acetone to the test sample. Extract by shaking thoroughly for 30 min on a shaking device or by sonification. Then add 50 ml of hexane-like solvent (5.2.2) and extract again thoroughly