



SLOVENSKI STANDARD

SIST EN 15308:2017

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Nadomešča:
SIST EN 15308:2008

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 gas chromatography with electron capture or mass spectrometric detection

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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 avec détection par capture d'électrons ou spectrométrie de masse

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ICS:

13.030.10	Trdni odpadki	Solid wastes
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

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

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This European Standard was approved by CEN on 21 September 2016.

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Contents	Page
European foreword.....	4
Introduction	5
1 Scope	6
2 Normative references	6
3 Terms and definitions	6
4 Principle	7
5 Reagents	8
5.1 General.....	8
5.2 Reagents for extraction and drying.....	8
5.3 Reagents for clean-up procedures.....	8
5.4 Reagents for gas chromatographic analysis.....	10
5.5 Standard compounds.....	10
5.5.1 Calibration standards.....	10
5.5.2 Internal and injection standards.....	10
6 Apparatus.....	11
6.1 Extraction and clean-up procedures.....	11
6.1.1 General.....	11
6.1.2 Extraction procedures.....	11
6.1.3 Clean-up procedures.....	12
6.2 Gas chromatograph	12
6.3 Capillary columns	12
7 Safety precautions	12
8 Interferences	13
9 Sample storage.....	13
10 Pretreatment	13
10.1 General.....	13
10.2 Drying.....	13
10.3 Particle size reduction.....	13
11 Procedure.....	14
11.1 Blank.....	14
11.2 Extraction.....	14
11.2.1 Extraction by shaking or sonification.....	14
11.2.2 Soxhlet extraction	14
11.3 Clean-up.....	15
11.3.1 General.....	15
11.3.2 Aluminium oxide clean-up.....	15
11.3.3 Silica cartridge clean-up	15
11.3.4 Silica H ₂ SO ₄ clean-up	15
11.3.5 Florisil® clean-up.....	15
11.3.6 TBA clean-up.....	16
11.3.7 Pyrogenic copper clean-up	16

11.3.8	AgNO ₃ /silica clean-up	16
11.3.9	DMSO/n-hexane partitioning clean-up.....	17
11.3.10	Benzenesulfonic acid/silica cartridge clean-up.....	17
11.4	Gas chromatographic analysis.....	17
11.4.1	General	17
11.4.2	Optimizing the gas chromatograph.....	17
11.4.3	Detectors.....	17
11.4.4	Check on method performance	18
11.4.5	GC-MS determination	19
11.4.6	GC-ECD determination	21
12	Calculation	22
13	Test report	22
Annex A	(informative) Performance characteristics.....	23
A.1	General	23
A.2	Type of samples and sample preparation	23
A.3	Homogeneity and stability	24
A.4	Extraction	25
A.5	Clean-up	25
A.6	Detection.....	25
A.7	PCB standard solution	25
Annex B	(informative) Examples of GC-MS chromatograms of a calibration standard solution and a contaminated soil sample	29
Annex C	(informative) Examples of GC-ECD chromatograms of a calibration standard solution and a cable shredder sample	36
Annex D	(normative) Calculation method for the estimation of total PCB content.....	39
Annex E	(informative) Summary of general requirements and recommendations.....	48
Bibliography	49

EN 15308:2016 (E)

European foreword

This document (EN 15308:2016) has been prepared by Technical Committee CEN/TC 444 “Test methods for the 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 April 2017, and conflicting national standards shall be withdrawn at the latest by April 2017.

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 15308:2008.

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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).

Taking 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 Mass Spectrometry and Electron Capture is possible. Two different extraction procedures and nine clean-up procedures are described. 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. This European Standard has been validated on seven solid waste which are typically contaminated with PCB (building debris, cable shredder, contaminated soil, electronic waste, sealant waste, shredder light fraction and waste wood). Validation data are given in Annex A (informative).

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1 Scope

This European Standard 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 amounts of individual PCB congeners equal or above 0,01 mg/kg dry matter can typically be determined with no interferences present.

NOTE 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.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12766-1, *Petroleum products and used oils - Determination of PCBs and related products - Part 1: Separation and determination of selected PCB congeners by gas chromatography (GC) using an electron capture detector (ECD)*

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EN 12766-2, *Petroleum products and used oils - Determination of PCBs and related products - Part 2: Calculation of polychlorinated biphenyl (PCB) content*

EN 14346, *Characterization of waste - Calculation of dry matter by determination of dry residue or water content*

3 Terms and definitions

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

Note 1 to entry: Any one of the two hundred and nine individual PCB.

Note 2 to entry: 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**

¹³C₁₂-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 needs to 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

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 clean-up 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 analysed 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 analysed using two columns of different polarity. Quantification is conducted by the internal standard method.

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

5.2.2 Hexane like solvents with a boiling range of 36 °C to 98 °C, e.g. petroleum ether, n-hexane, n-heptane.

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, which 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.

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 volumes of water and 2-propanol, $c((\text{C}_4\text{H}_9)_4\text{NHSO}_4) = 0,1\text{ mol/l}$, with sodium sulfite.

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

5.3.3 Pyrogenic copper for sulfur removal:

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

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

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

5.3.3.3 Zinc granules, Zn, 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, particle size 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 pour 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, C_6H_{14} .

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5.3.7 Sulphuric acid clean-up:

5.3.7.1 Sulphuric acid, H_2SO_4 , 95 % to 97 %.

5.3.8 Silica gel/sulphuric acid clean-up:

5.3.8.1 Silica/ H_2SO_4 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 % 1 mol/l: pour 33,5 g of activated silica and 16,5 g 1 mol/l 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 Florisil[®] 1) 100 mesh to 200 mesh, activated by heating to 600 °C for a minimum of 2 h.

5.3.10.2 Iso-octane, C_8H_{18} .

5.3.10.3 Iso-octane/Toluene 95/5.

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.

EN 15308:2016 (E)

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);
- 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

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

- ^{13}C -2,4,4'-trichlorobiphenyl (PCB-28); [SIST EN 15308:2017](https://standards.iteh.ai/catalog/standards/sist/cc154cda-f71e-421f-8ad8-574e3370c2d/sist-en-15308-2017)
- ^{13}C -2,2',5,5'-tetrachlorobiphenyl (PCB-52); <https://standards.iteh.ai/catalog/standards/sist/cc154cda-f71e-421f-8ad8-574e3370c2d/sist-en-15308-2017>
- ^{13}C -2,2',4,5,5'-pentachlorobiphenyl (PCB-101);
- ^{13}C -2,3',4,4',5-pentachlorobiphenyl (PCB-118);
- ^{13}C -2,2',3,4,4',5'-hexachlorobiphenyl (PCB-138);
- ^{13}C -2,2',4,4',5,5'-hexachlorobiphenyl (PCB-153);
- ^{13}C -2,2',3,4,4',5,5'-heptachlorobiphenyl (PCB-180).

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

The application of isotopic dilution mass spectrometry is recommended but adding ^{13}C -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 ^{13}C -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).

For quantification (internal standard) PCB-198 and PCB-209 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).

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6 Apparatus

6.1 Extraction and clean-up procedures

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6.1.1 General <https://standards.iteh.ai/catalog/standards/sist/cc154cda-f71e-421f-8ad8-57ff31370c2d/sist-en-15308-2017>

6.1.1.1 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.

EN 15308:2016 (E)

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.**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 vaporizer 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

The columns consist of 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 the test under identical conditions as samples for the determination of the resolution of the critical pair PCB-28/PCB-31. 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 or equivalent, 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.

7 Safety precautions

Anyone dealing with waste and sludge analysis shall 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 pressurize 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 sulfur) 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 analysed 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 $4\text{ °C} \pm 2\text{ °C}$ in sample containers in a dark place for a maximum of one week. If the sample cannot be processed within seven days, it shall be stored at temperatures below -18 °C . Dried samples shall be stored at room temperature in a dark place.

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10 Pretreatment

<https://standards.iteh.ai/catalog/standards/sist/cc154cda-f71e-421f-8ad8-57ff31370c2d/sist-en-15308-2017>

10.1 General

The goal of a pretreatment 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 pretreatment 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 $0,5\text{ mm}$, e.g. by using an ultra-centrifugal mill.