



SLOVENSKI STANDARD SIST EN ISO 15318:2000

01-december-2000

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SIST ENV 1798:1997

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Pulp, paper and board - Determination of 7 specified polychlorinated biphenyls (PCB)
(ISO 15318:1999)

Zellstoff, Papier und Pappe - Bestimmung von 7 ausgewählten Biphenylen (PCB) (ISO
15318:1999)

Pâtes, papiers et cartons - Détermination de 7 polychlorobiphényles (PCB) spécifiés
(ISO 15318:1999) <https://standards.iteh.ai/catalog/standards/sist/16210f3c-2f20-46d3-a036-a8e0802b3b11/sist-en-iso-15318-2000>

Ta slovenski standard je istoveten z: EN ISO 15318:1999

ICS:

| | | |
|--------|--|---|
| 67.250 | Materiali in predmeti v stiku z živili | Materials and articles in contact with foodstuffs |
| 85.040 | Vlaknine | Pulps |
| 85.060 | Papir, karton in lepenka | Paper and board |

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 15318

October 1999

ICS 67.250; 85.040; 85.060

English version

**Pulp, paper and board - Determination of 7 specified
polychlorinated biphenyls (PCB) (ISO 15318:1999)**

Pâtes, papiers et cartons - Détermination de 7
polychlorobiphényles (PCB) spécifiés (ISO 15318:1999)

Zellstoff, Papier und Pappe - Bestimmung von 7
ausgewählten Biphenylen (PCB) (ISO 15318:1999)

This European Standard was approved by CEN on 25 June 1999.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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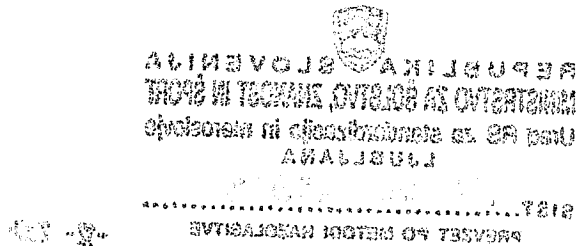
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Foreword

The text of EN ISO 15318:1999 has been prepared by Technical Committee CEN/TC 172 "Pulp, paper and board", the secretariat of which is held by DIN, in collaboration with Technical Committee ISO/TC 6 "Paper, board and pulps".

This European Standard supersedes ENV 1798:1995.

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 March 2000, and conflicting national standards shall be withdrawn at the latest by March 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

With regard to ENV 1798 : 1995-07 the following changes have been made:

- a) extension of the scope to "pulp";
- b) addition of detailed information on "precision";
- c) transformation from a European Prestandard (ENV) to a European Standard (EN);
- d) editorial updating.

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Introduction

CEN/TC 172 has decided to publish this test method as a European Standard (EN) because the validation of the test method on the level of the existing limit for PCB (2 ppm) was until now impossible due to the fact that there was no reference material with this level of PCB and all samples tested have a PCB content on the level of the detection limit (about 5 µg/kg of the congeners).

Prior to discontinuance of its use in 1971 a commercial PCB had been an ingredient in carbonless copy paper. The presence of these copy papers in waste paper can lead to contamination of recycled pulp, paper and board products with PCB.

The PCB contaminant has the same congener pattern as the PCB used earlier in carbonless copy papers and this identifies the source of PCB contamination.

In this method, seven specified PCB congeners (numbers 18, 28, 52, 101, 138, 153 and 180) are determined individually. Because the source of the PCB contamination can be identified from the congener pattern, the total PCB content of the paper may be estimated from these seven congeners.

For routine analyses, the spiking procedure of 5.10.5 and 7.4.4 may be omitted, provided that the result obtained from the analysis is less than 50 % of any present limit. This will require modification of clause 8 to take account of these changes. The use of this modification shall be stated in the test report. In the event of any dispute the full method shall be used.

Warning:

The use of this European Standard may involve hazardous materials, operations and equipment. It does not address all the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of safety regulations prior to use.

1 Scope

This European Standard gives guidance on a test method which permits the determination of seven specified PCBs in pulp, paper and board. Annex A gives a procedure for estimating the total content of PCB from the congener content.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN ISO 186

Paper and board - Sampling to determine average quality (ISO 186 : 1994)

EN 27213

Pulps – Sampling for testing (ISO 7213 : 1981)

3 Principle

The test material is extracted with boiling ethanolic or methanolic potassium hydroxide solution. An aliquot of the extract is mixed with water and subjected to liquid-solid partitioning on a disposable C₁₈ solid phase extraction cartridge followed by elution with hexane or ISO-octane.

The PCBs contained in the hexane phase are quantified by capillary gas chromatography using an electron capture detector (ECD) or a mass selective detector (MSD). The pattern of the seven congener peaks is compared with the pattern of a technical PCB.

If the patterns correspond, the level of total PCB can be estimated from the congener content by application of an appropriate factor.

4 Apparatus and auxiliary aids

4.1 Ordinary laboratory apparatus

4.2 Extractor reservoir

An example is given in Annex B, where the reservoir comprises a glass tube approximately 200 mm long with an internal diameter of 30 mm.

The tube is tapered twice at the lower end to ensure that the connection to the disposable cartridge (4.3) is gas-tight and to allow drops to build up on the tip.

4.3 Disposable solid-phase extraction cartridge with a C₁₈ bonded phase (3,0 ml size and 200 mg).

4.4 Gas chromatograph with an electron capture detector (ECD) or a mass selective detector (MSD).

4.5 Capillary column suitable for the determination of PCB in accordance with the specification laid down in 8.1.

5 Reagents

Unless otherwise specified, reagents of a grade suitable for residue analysis shall be used. Water should be double-distilled or of equivalent quality. Methanol can be used in place of ethanol for all solutions if required and ISO-octane can be used in place of hexane.

5.1 Ethanol

(C₂H₅OH > 99,5 %)

5.2 Methanol

(CH₃OH > 99,8 %)

5.3 n-Hexane

(C₆H₁₄ > 98,0 %)

5.4 Sulphuric acid, concentrated

(d = 1,84)

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5.5 Reference substances

Ballschmit Nomenclature

| | | |
|-------|--------------------------------------|-----|
| 5.5.1 | 2,2',5'-Trichlorobiphenyl | 18 |
| 5.5.2 | 2,4,4'-Trichlorobiphenyl | 28 |
| 5.5.3 | 2,2',5,5'-Tetrachlorobiphenyl | 52 |
| 5.5.4 | 2,2',4,5,5'-Pentachlorobiphenyl | 101 |
| 5.5.5 | 2,2',3,4,4',5'-Hexachlorobiphenyl | 138 |
| 5.5.6 | 2,2',4,4',5,5'-Hexachlorobiphenyl | 153 |
| 5.5.7 | 2,2',3,4,4',5,5'-Heptachlorobiphenyl | 180 |

5.6 Comparison sample

Technical mixture of, for example, Chlophen¹⁾ A 30 to A 60® or Arochlor²⁾ 1242 to 1260.

5.7 Gas-Chromatography (GC) resolution sample

5.7.1 2,4',5-Trichlorobiphenyl (TCBP, PCB 31)

5.8 Internal standards

5.8.1 2,4,6-Trichlorobiphenyl (TCBP, PCB 30)

5.8.2 2,4,6-Tribromobiphenyl (TBBP)

¹⁾ Chlophen is the trade-name of a product supplied by Bayer. This information is given for the convenience of users of this Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

²⁾ Arochlor is an example of a suitable product available commercially. This information is given for the convenience of users of this Standard and does not constitute an endorsement by CEN of this product.

5.9 Ethanolic potassium hydroxide solution (2 % w/v)

Dissolve 30,0 g of potassium hydroxide in a solution 19:1 v/v ethanol/water (1500 ml). Allow to stand for 24 h, decant, and retain the clear solution.

5.10 Combined standard solutions

Prepare the following standard solutions using volumetric glassware throughout:

NOTE: Mixtures corresponding to 5.10.1 to 5.10.4 are commercially available.

5.10.1 Intermediate standard solutions A (200 µg/ml)

Take approximately 10,0 mg (to an accuracy of 0,1 mg) of reference congener substance 18 (5.5.1), transfer quantitatively to a 50,0 ml volumetric flask and make up to the mark with hexane. Shake to dissolve.

Repeat for congeners 28, 52, 101, 138, 153 and 180 (5.5), GC resolution sample 31 (5.7) and for TCBP or TBBP (5.8).

5.10.2 Intermediate standard solutions B (20 µg/ml)

Take 5,00 ml of solution A for congener 18 (5.10.1) and dilute to 50,0 ml using hexane.

Repeat for congeners 28, 52, 101, 138, 153 and 180, GC resolution sample 31, and for TCBP or TBBP (5.8).

5.10.3 Individual standard solutions for GC (0,1 µg/ml)

Take 1,00 ml of solution B for congener 18 (5.10.2) and dilute to 200,0 ml with hexane.

Repeat for congeners 28, 52, 101, 138, 153 and 180, GC resolution sample 31, and for TCBP or TBBP (5.8).

5.10.4 Combined standard solutions for GC (0,1 µg/ml)

Take 1,00 ml of solution B for each congener 18, 28, 52, 101, 138, 153 and 180, GC resolution sample 31, and TCBP or TBBP (5.8), and dilute to 200,0 ml with hexane.

5.10.5 Spiking solution (0,1 µg/ml)

Take approximately 10,0 mg (to an accuracy of 0,1 mg) of congener substances 18, 28, 52, 101, 138, 153 and 180 (5.5), transfer quantitatively to a 100,0 ml volumetric flask and make up to the mark with ethanol. Shake to dissolve.

Take 5,00 ml of this solution and dilute to 100,0 ml with ethanol.

Take 5,00 ml of this second solution and dilute to 250,0 ml with ethanolic potassium hydroxide solution (5.9).

5.10.6 Internal standard solution (0,1 µg/ml)

Take approximately 10,0 mg (to an accuracy of 0,1 mg) of internal standard substance TCBP or TBBP (5.8), transfer quantitatively to a 100,0 ml volumetric flask and make up to the mark with ethanol. Shake to dissolve.

Take 5,00 ml of this solution and dilute to 100,0 ml with ethanol.

Take 5,00 ml of this second solution and dilute to 250,0 ml with ethanolic potassium hydroxide solution (5.9).

6 Sampling

Sampling shall be carried out in accordance with EN ISO 186 or EN 27213. The sample shall be wrapped in aluminium foil between acquisition and testing, to prevent any change before the test.

7 Procedure

7.1 Suitable safety measures shall be taken when the work laid down in this test method is carried out.

7.2 Attach the solid-phase disposable cartridge (4.3) to the extractor reservoir (4.2) to give a gas-tight seal and condition with two charges of methanol (5.2) (typically 2 x 3,0 ml) followed by water (typically 2 x 3,0 ml) in accordance with the manufacturer's instructions.

7.3 Take a representative sample of the pulp, paper or board sample (about 100 g as received), cut into pieces of about 1 cm² and randomise by shaking in a large glass beaker.

7.4 Take nine 100 ml round-bottomed flasks. Add (2,00 ± 0,02) g portions of the paper or board, or for pulp (1,80 ± 0,02) g as oven dry (7.3) to eight of these flasks. Treat the nine flasks as follows:

7.4.1 Sample MB (method blank)

Add 2,00 ml internal standard solution (5.10.6) to the flask with no pulp, paper or board.

7.4.2 Sample SB (sample blank)

No further additions.

7.4.3 Samples S1 to S3 (triplicate samples)

Add 2,00 ml of the internal standard solution (5.10.6).

7.4.4 Samples C1 to C4 (calibration samples)

Add 2,00 ml of the internal standard solution (5.10.6) together with 1,00 ml, 2,00 ml, 3,00 ml or 4,00 ml of the spiking solution (5.10.5).

7.5 Add ethanolic potassium hydroxide solution (5.9) to each of the flasks to give a total liquid volume of 50,0 ml.

Reflux for 60 min, cool to room temperature and pipette 25,0 ml of the extract into a conical flask containing 50,0 ml water. Mix immediately for 5 s.

7.6 Pour the mixture immediately into the extractor reservoir with the conditioned cartridge attached (7.2). Apply pressure or vacuum to give a flow rate of 50 to 100 drops per min. Discard the eluent.

Remove the reservoir and dry the cartridge for approximately 10 min with the pressure or vacuum maintained. Place a separating funnel or a test tube (10,0 ml) at the cartridge outlet and pipette 0,5 ml n-hexane (5.3) into the cartridge. Allow the n-hexane to soak into the cartridge packing. Set the cartridge aside for 5 min to 10 min to allow the n-hexane to permeate the packing, and then add a further 0,5 ml n-hexane and elute carefully into the separating funnel using pressure or vacuum. Finally, add a further 1,0 ml n-hexane and elute with pressure.

7.7 Wash the hexane extract successively with 2,0-ml portions of sulphuric acid (5.4) until no further colour is extracted. Transfer the hexane extract into a 2,0 ml volumetric flask and make up to the 2,0 ml mark with hexane.

The hexane phase is now ready for GC analysis.

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8 Determination**8.1 Working conditions for gas chromatography**

The gas chromatograph and electron capture detector or mass selective detector should be optimised with regard to sensitivity, linearity and reproducibility, according to the manufacturer's instructions.

The following are examples of columns and conditions that have proved suitable for this analysis.

| | |
|-------------------------|---|
| Capillary column (4.5): | cross-linked 5 % phenylmethyl silicone on fused silica. 50 m to 60 m length. 0,2 mm to 0,35 mm internal diameter. 0,1 μ m to 0,3 μ m phase. |
| Injector: | 250° C to 270° C split/splitless (vaporising) mode. |
| ECD detector: | 300° C to 350° C. |
| Oven: | temperature programmed from 100° C (2 min) at 25° C/min to 160° C and hold 10 min, then at 5° C/min to 280° C and hold for 10 min, or, 130° C (zero min) rising at 2,5° C/min to 290° C and hold 5 min. |

8.2 Establishing GC retention times

Analyse sequentially the individual standard solutions (5.10.3) to establish retention times for the internal standards, PCB congeners and GC resolution sample PCB 31.

8.3 Criteria for retention time reproducibility

Make three injections of the combined standards GC solution (5.10.4) and establish the variability (standard deviation) of the retention time for each component. This should typically not exceed 3 s or 0,5 % of the retention time, whichever is the greater.

8.4 Criteria for GC resolution

The resolution between congeners 28 and 31 serves for evaluation of the gas chromatograph. A resolution of $R > 0,5$ should be achieved.