
**Pulp, paper and board — Determination of
7 specified polychlorinated biphenyls (PCB)**

*Pâtes, papiers et cartons — Détermination de 7 polychlorobiphényles
(PCB) spécifiés*

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ISO 15318:1999

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 734 10 79
E-mail copyright@iso.ch
Web www.iso.ch

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15318 was prepared by the European Committee for Standardization (CEN) in collaboration with ISO Technical Committee TC 6, *Paper, board for pulp*, Subcommittee SC 5, *Test methods and quality specifications for pulp*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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Throughout the text of this standard, read "(...this European Standard...)" to mean "...this International Standard...".

Annexes A, B and C form a normative part of this International Standard.

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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.

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1 Scope

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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.

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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.

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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.

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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)

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)

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.

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.