
**Pulp, paper and board — Determination
of pentachlorophenol in an aqueous
extract**

*Pâtes, papier et carton — Détermination du pentachlorophénol dans
un extrait aqueux*

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ISO 15320:2003

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Foreword

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The main task of technical committees is to prepare International Standards. 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 the member bodies casting a vote.

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

ISO 15320 was prepared by the European Committee for Standardization (CEN) in collaboration with Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulps*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Throughout the text of this document, read “...this European Standard...” to mean “...this International Standard...”.

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Foreword

This document (EN ISO 15320:2003) 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 EN ISO 15320:2003 including the Amendment shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2004, and conflicting national standards shall be withdrawn at the latest by June 2004.

WARNING: The use of this European Standard may involve hazardous materials, e.g. methanol and pentachlorophenol, which are toxic substances, as well as acetic anhydride, which is corrosive. This European Standard does not address all the safety and environmental problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety, health and environmental practices and determine applicability of safety regulations prior to use.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

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1 Scope

This European Standard specifies a test method for the determination of pentachlorophenol (PCP) in an aqueous extract in pulp, paper and board. Though it is developed for paper and board intended to come into contact with foodstuffs, it is applicable to all kinds of pulp, paper and board.

NOTE 1 It was investigated that a hot water extract (acc. to EN 647) is sufficient for the determination of PCP in pulp, paper and board.

The working range for the method is 0,05 mg/kg to 0,5 mg/kg.

NOTE 2 The upper limit of the working range could be increased if the aqueous extract is diluted.

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 in this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 645 *Paper and board intended to come into contact with foodstuffs – Preparation of a cold water extract*

EN 647 *Paper and board intended to come into contact with foodstuffs – Preparation of a hot water extract*

EN ISO 3696:1995 *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*

3 Principle

A specimen of the material to be tested is extracted with cold water according to EN 645 or with hot water according to EN 647. The pentachlorophenol in the extract is concentrated using solid phase extraction on a phenyl column. The pentachlorophenol is then eluted from the phenyl column with hexane, acetylated using acetic anhydride and the amount of pentachlorophenol present determined using gas chromatography employing an ECD (electron capture detector) or MS (mass spectrometer) detector. The result is expressed as milligrams per kilogram of material.

4 Apparatus

4.1 Ordinary laboratory apparatus

4.2 Solid phase extraction (SPE) system and SPE columns

Propylphenyl columns, dimension 500 mg/3 ml.

4.3 Gas chromatograph

A conventional split/splitless injector gives suitable sensitivity when used in the splitless mode. Alternatively, an on-column injection mode may be used.

4.4 Capillary column

suitable for determination of acetylated derivatives of pentachlorophenol. A column with the following features is given as an example.

- stationary phase: polydimethylsiloxane with 5 % phenyl groups
- film thickness: 0,25 μm
- length: 30 m
- internal diameter: 0,32 mm.

4.5 Detector

— electron capture detector (ECD)

or

— mass spectrometer (MS) with the following features is given as an example:

- a) ionisation: EI 70 eV (electron impact)
- b) resolution: 1 amu (atomic mass unit)
- c) runability: SIM mode (selected ion monitoring) (SIM is called SIR, selected ion recording for some instruments)

5 Reagents

5.1 General

All reagents shall be of a grade "pro analysis" (p.a.) or equivalent quality, respectively water of grade 2 according to EN ISO 3696:1995.

5.2 Methanol,

CH₃OH.

5.3 Hydrochloric acid,

HCl (0,1 mol/l).

5.4 n-Hexane,

C₆H₁₄.

5.5 Sulphuric acid,

H₂SO₄ (diluted 1+1).

5.6 Acetic anhydride,

C₄H₆O₃ (99 %).

5.7 Potassium carbonate solution,

K₂CO₃ (0,1 mol/l). Weigh 13,8 g potassium carbonate with an accuracy of 0,1 g and dissolve it in a beaker with a small amount of water. Transfer the solution to a 1000-ml volumetric flask and dilute to volume with water.

5.8 Reference solutions (for ECD)**5.8.1 Pentachlorophenol in methanol reference stock solution,**

100 µg/ml. This solution is commercially available.

5.8.2 Pentachlorophenol in methanol dilute reference stock solution,

5 µg/ml. Pipette 1 ml of the stock solution (5.8.1) into a 20 ml volumetric flask and dilute to volume with methanol. Solution is stable more than 6 months in the refrigerator.

5.8.3 Pentachlorophenol in methanol reference solution,

0,5 µg/ml. Pipette 1 ml of the dilute stock solution (5.8.2) into a 10 ml volumetric flask and dilute to volume with methanol. Solution is stable more than 3 months in the refrigerator.

5.9 Internal standard solutions for ECD**5.9.1 2,3,6-trichlorophenol internal standard stock solution,**

10 µg/ml, internal standard stock solution. This solution is commercially available.