

SLOVENSKI STANDARD

SIST EN ISO 15320:2004

01-februar-2004

Določitev vsebnosti pentachlorophenola v vodni ekstrakti (ISO 15320:2003)

Pulp, paper and board - Determination of pentachlorophenol in an aqueous extract (ISO 15320:2003)

Halbstoff, Papier und Pappe - Bestimmung von Pentachlorphenol in einem wässrigen Extrakt (ISO 15320:2003)

Pâtes, papier et carton - Détermination du pentachlorophénol dans un extrait aqueux (ISO 15320:2003)

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Ta slovenski standard je istoveten z: **EN ISO 15320:2003**

ICS:

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 15320

December 2003

ICS 85.040; 85.060

English version

**Pulp, paper and board - Determination of pentachlorophenol in
an aqueous extract (ISO 15320:2003)**

Pâtes, papier et carton - Détermination du
pentachlorophénol dans un extrait aqueux (ISO
15320:2003)

Halbstoff, Papier und Pappe - Bestimmung von
Pentachlorphenol in einem wässrigen Extrakt (ISO
15320:2003)

This European Standard was approved by CEN on 20 February 2003.

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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of an identical text or by endorsement, at the latest by June 2004, and conflicting national standards shall be withdrawn at the latest by June 2004.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)*

EN ISO 15320:2003 (E)

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.

EN ISO 15320:2003 (E)**5.9.2 2,3,6-trichlorophenol internal standard solution,**

2 µg/ml, internal standard solution. Pipette 2 ml of the internal standard stock solution (5.9.1) into a 10 ml volumetric flask and dilute to volume with methanol. Solution is stable more than 3 months in the refrigerator.

5.10 Internal standard solutions for MS**5.10.1 ¹³C6 labelled pentachlorophenol (labelled at all six carbons) internal standard stock solution,**

10 µg/ml, internal standard stock solution. This solution is commercially available. It is an alternative to the 2, 3, 6-trichlorophenol methanol solution mentioned in 5.9.1 and can be applied only when a mass spectrometer is used as a detector.

5.10.2 ¹³C6 labelled pentachlorophenol internal standard solution,

1 µg/ml, internal standard solution. Pipette 1 ml of the internal standard stock solution (5.10.1) into a 10 ml volumetric flask and dilute to volume with methanol. Solution is stable more than 3 months in the refrigerator.

6 Sampling and extraction

Sampling, preparation of sample and extraction shall be carried out in accordance with EN 645 (cold water extract) or EN 647 (hot water extract).

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7 Procedure

The water extract shall be analysed. Filter according to EN 645 or EN 647.

7.1 Preparation

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Take out 50 ml from the water extract. Add 200 µl of the internal standard solution (5.9.2 or 5.10.2) and acidify with 1 ml sulphuric acid (5.5).

7.2 Solid phase concentration**7.2.1 Conditioning**

Pre-rinse a phenyl column (4.2) with 2 ml methanol and 5 ml 0,1 M HCl (5.3). Do not allow the column to run dry.

7.2.2 Concentration

Pour the acidified extract onto the column, elute at 2 ml/min to 3 ml/min. Subsequently, rinse the column with 5 ml pure analytical water (5.1) and suck it dry (approx. 5 min to 10 min).

7.2.3 Elution

For the receiver use a 50 ml conical flask or a separatory funnel, with 35 ml 0,1 M K₂CO₃ solution (5.7). Pour 2,5 ml hexane onto the phenyl column and wait until the first drop is seen in the lower end. Close the valve and elute after 2 min. Rinse with another 2,5 ml hexane.

7.3 Acetylation

Add 1 ml acetic anhydride (5.6) to the receiver and stir vigorously for approximately 3 min to allow the release of all carbon dioxide. Allow the two phases to separate. Fill the receiver with water until the hexane phase reach the neck of the receiver. Transfer the organic layer into a vial in order to avoid a reverse reaction. This extract has to be analysed within 24 h.

NOTE If the hexane solution is dried with e.g. sodium sulphate the solution can be stored for at least two weeks.

7.4 Gas chromatographic analysis

Choose the suitable conditions for determination of pentachlorophenol. Follow the instructions provided by the manufacturer of the instrument. The following conditions are given as an example.

- splitless injection: 1 µl;
- injector temperature: 250 °C;
- electron capture detector temperature: 350 °C;
- carrier gas, flow: 1 ml/min.
- temperature programme: 50 °C (1 min) at 20 °C/min to 180 °C and hold 2 min, then at 2 °C/min to 200 °C and hold for 1 min or 20 °C (1 min) to 280 °C and hold 5 min.

Carry out the detection either by the electron capture detector or by the use of a mass spectrometer. In both cases, follow the instructions provided by the manufacturer of the instrument.

If a mass spectrometer is used, with $^{13}\text{C}_6$ -labelled pentachlorophenol as internal standard, the base peak, 266, is used for quantification of PCP, ($266 = M + 2$ minus ketene, $\text{CH}_2=\text{C}=\text{O}$, from the acetate). The corresponding ion from the $^{13}\text{C}_6$ -labelled pentachlorophenol is 272 ($266 + 6$). Verification of PCP should be made from the retention time and the ratio between 264 and 266, which shall be close to 61/100 due to the chlorine isotope ratio.

If the value of PCP obtained exceeds the range covered by the calibration solutions, repeat the analysis from 7.1 with a more dilute sample solution.

8 Calibration

8.1 Calibration for electron capture detector, ECD

8.1.1 Preparation of calibration solutions

Prepare pentachlorophenol standard solutions for the calibration as follows:

Take 35 ml potassium carbonate solution (5.7) in a 50 ml conical flask or a separatory funnel (see 7.2.3). Add 200 µl of the internal standard solution (5.9.2). Make four solutions by adding various amounts of PCP (use the reference solutions (5.8.2 and 5.8.3). Shake for 3 min. Add 5 ml hexane (5.4) and 1 ml acetic anhydride and shake until the gas evolution has stopped (approximately 3 min). Fill up with pure analytical water until the organic phase is in the neck of the conical flask and can be removed with a pipette.

NOTE If the hexane solution is dried with e. g. sodium sulphate the solution can be stored for at least four weeks.

8.1.2 Gas chromatographic determination

Proceed to gas chromatographic determination as described in 7.4.

Carry out the gas chromatographic determinations in duplicate.