SLOVENSKI STANDARD
SIST-TS CEN/TS 17497:2020

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Vlaknine, papir, karton in lepenka - Ugotavljanje bisfenola A v ekstraktih papirja, kartona in lepenke

Pulp, paper and paperboard - Determination of bisphenol A in extracts from paper and paperboard

Zellstoff, Papier und Karton - Bestimmung von Bisphenol A in Papier- und Kartonextrakten

Cellulose, papier et canon - Détermination des bisphénol A dans des extraits de papier et canon

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ICS:

85.060	Papir, karton in lepenka	Paper and board

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TECHNICAL SPECIFICATION

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English Version

Pulp, paper and paperboard - Determination of bisphenol A in extracts from paper and paperboard

Cellulose, papier et carton - Détermination des bisphénol A dans des extraits de papier et carton

Zellstoff, Papier und Karton - Bestimmung von Bisphenol A in Papier- und Kartonextrakten

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (CEN/TS 17497:2020) has been prepared by Technical Committee CEN/TC 172 “Pulp, paper and board”, the secretariat of which is held by DIN.

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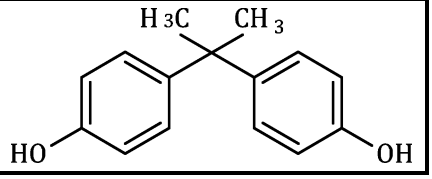
1 Scope

This document specifies an analytical test method for the determination of bisphenol A in solvent extracts of paper and board materials and articles intended to come into contact with foodstuffs using a high performance liquid chromatograph coupled to a fluorescence detector (HPLC-FLD).

This method can be applied to determine bisphenol A (see Table 1) in concentrations ranging from 0,025 mg/l to 2 mg/l in the solvent extracts, corresponding to 0,05 mg/kg to 4 mg/kg paper and board. The measurement range can easily be extended up to 40 mg/kg by adjusting the concentration factor of the solvent extract.

Table 1 — Bisphenol A

Name	Abbreviation	Formula	CAS N°	Structure
Bisphenol A	BPA	$C_{15}H_{16}O_2$	80–05–7	

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 186, *Paper and board - Sampling to determine average quality (ISO 186)*

EN ISO 536, *Paper and board - Determination of grammage (ISO 536)*

EN ISO 638, *Paper, board and pulps - Determination of dry matter content - Oven-drying method (ISO 638)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

4 Principle

To assess the content of bisphenol A in paper and board materials, solvent extracts of these are prepared and bisphenol A is determined in the concentrated extracts by HPLC-FLD.

Also, liquid chromatography-mass spectrometry (LC-MS) can be used as an alternative method.

5 Materials

5.1 Pasteur pipettes

5.2 Piston pipettes with a capacity adjustable from 10 µl to 25 µl, 20 µl to 50 µl, 50 µl to 250 µl and 100 µl to 1 000 µl.

5.3 Volumetric pipettes with a capacity of 10 ml and 100 ml.

5.4 Volumetric flasks with a capacity of 2 ml, 10 ml and 25 ml.

5.5 Glass-stoppered conical flask with a capacity of 250 ml.

5.6 Pear shaped flask with a capacity of 25 ml.

5.7 Disposable syringes with a capacity of 2 ml.

5.8 Polytetrafluoroethylene (PTFE) membrane filters, 0,2 µm.

5.9 Disposable screw thread glass vials with a capacity of 1,5 ml and the corresponding polypropylene (PP) caps with nitrile rubber/PTFE gaskets.

6 Apparatus

6.1 Balance capable of accurately weighing 0,000 1 g.

6.2 Thermostatically controlled oven, **incubator** or **refrigerator** capable of maintaining a temperature of (23 ± 1) °C.

6.3 Rotary evaporator.

6.4 Evaporator working with a stream of nitrogen.

6.5 High performance liquid chromatograph coupled to a fluorescence detector and optional to a diode array detector.

7 Reagents

7.1 General

All reagents should be of analytical grade or better. Solvents applied as mobile phase for the HPLC analysis should be of adequate quality.

7.2 Water, grade 1 according to ISO 3696.

7.3 Acetonitrile, for HPLC analysis, ≥ 99,9 %.

7.4 Bisphenol A, ≥ 99 %.

7.5 Solutions of bisphenol A in acetonitrile

7.5.1 Stock solution at 1 000 mg/l

In a 25 ml volumetric flask, 25 mg bisphenol A (7.4) with an accuracy of 0,000 1 g are weighted and made up to volume with acetonitrile (7.3).

7.5.2 Intermediate solution at 5 mg/l

50 µl of the stock solution (7.5.1) are pipetted into a 10 ml volumetric flask and made up to volume with acetonitrile (7.3).

7.6 Calibration solutions of bisphenol A in acetonitrile

Seven calibration solutions are prepared by pipetting 10 µl, 20 µl, 40 µl, 100 µl, 200 µl, 400 µl and 800 µl of the intermediate solution (7.5.2), respectively, into 2 ml volumetric flasks. These are made up to volume with acetonitrile (7.3). The bisphenol A concentrations in these calibration solutions are about 0,025 mg/l, 0,05 mg/l, 0,1 mg/l, 0,25 mg/l, 0,5 mg/l, 1 mg/l and 2 mg/l.

8 Sampling

If the analysis is performed to evaluate a batch of paper or board, the sample shall be selected in accordance with EN ISO 186. If other types of samples shall be analysed, the source of the sample, and, if possible, the sampling procedure have to be reported. The selected test specimens shall be representative for the sample received.

If required, separate samples for the determination of the grammage in accordance with EN ISO 536 and/or of the dry matter content in accordance with EN ISO 638 shall be taken.

The sample shall be protected from contamination during transport and/or storage between sampling and analysis by wrapping it with aluminium foil.

9 Preparation of the analysis samples

The paper or board samples taken are cut into pieces of at most 1 cm^2. Then (5,0 ± 0,05) g of each sample are weighted to an accuracy of 0,000 1 g into a conical flask and 100 ml acetonitrile (7.3) are added. The extractions of the samples are carried out for (24 +1) hours at (23 ± 1) °C in a thermostatically controlled oven.

Of these extracts, in each case 10 ml are transferred to a pear shaped flask and the solvent is reduced to a volume of approximately 1 ml using a rotary evaporator. The residual solvent is evaporated to dryness using a gentle stream of nitrogen. The residue is redissolved in 1 ml acetonitrile (7.3) and after filtration through a 0,2 µm membrane filter, the concentrated extracts are directly subjected to HPLC-FLD analysis.

In parallel to the sample extractions, blank samples have to be prepared using a portion of the same batch of organic solvent that was used to extract the paper or board sample.

10 Procedure

10.1 Example of suitable HPLC conditions for the analysis of bisphenol A

— column: RP 18, (150 × 3) mm, 5 µm;

— eluent A: water (7.2), eluent B: acetonitrile (7.3);

— column temperature: 40 °C;

— injection volume: 20 µl;

— flow rate: 0,8 ml/min;

— gradient: 0 min – 30 % B; 8 min – 70 % B; 9 min – 100 % B; 11 min – 100 % B; 13 min – 30 % B; 15 min – stop;

— detection FLD: excitation 275 nm, emission 305 nm;

— optional: DAD detection at 230 nm.

The identification and quantification of bisphenol A is performed by FLD, while the DAD signal can be used additionally for identification in case of doubt.

10.2 Calibration

By analysis of the calibration solutions (7.6) with the HPLC method given under point 10.1, the retention time of bisphenol A as well as its spectrum are recorded. From these analyses, also linear calibration curves are constructed by plotting the peak areas against the corresponding concentrations of bisphenol A in the calibration solutions.

10.3 Analysis of the sample extracts

Aliquots of each analysis solution including the blank solutions (9) are injected into the HPLC device (10.1). Using the retention time established from the analysis of the calibration solutions (10.2), bisphenol A is located in the chromatograms received.

Each signal at the retention time of bisphenol A is integrated to determine its peak area. For identification of bisphenol A, the FLD spectra of the peaks detected in the analysis and blank solutions are compared with those obtained from the analysis of the calibration solutions. In case of an equivocal FLD spectra, also the DAD spectra can be used for identification.

The bisphenol A concentrations are calculated by the HPLC software in mg/l using the calibration curve obtained from the calibration solutions in acetonitrile (10.2).