
Vlaknine, papir, karton in lepenka - Ugotavljanje koncentracije antrakinona v ekstraktih vlaknin, papirja, kartona in lepenke

Pulp, paper and paperboard - Determination of anthraquinone in extracts from pulp, paper and paperboard

Faserstoff, Papier und Karton - Bestimmung von Anthrachinon in Faserstoff-, Papier- und Kartonextraten

Pâte, papier et carton - Détermination d'antraquinone dans des extraits de pâte, papier et carton

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ICS:

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

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SPÉCIFICATION TECHNIQUE
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FprCEN/TS 17630

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ICS 67.250; 85.060

English Version

**Pulp, paper and paperboard - Determination of
anthraquinone in extracts from pulp, paper and
paperboard**

Pâte, papier et carton - Détermination d'anthraquinone
dans des extraits de pâte, papier et carton

Faserstoff, Papier und Karton - Bestimmung von
Anthrachinon in Faserstoff-, Papier- und
Kartonextraten

This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 172.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (FprCEN/TS 17630:2021) has been prepared by Technical Committee CEN/TC 172 “Pulp, paper and board”, the secretariat of which is held by DIN.

This document is currently submitted to the Vote on TS.

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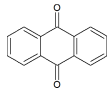
FprCEN/TS 17630:2021 (E)

1 Scope

This document specifies an analytical test method for the determination of anthraquinone (see Table 1) in water and 95 % ethanol extracts of pulp, paper and board materials and articles intended to come into contact with foodstuffs using a gas chromatograph coupled to a mass spectrometer (GC-MS). Moreover, acetone extracts of modified polyphenylene oxide (MPPO) that, according to EN 14338, can be used as a simulant to assess the possible transfer/migration of substances from paper and board into dry, non-fatty foodstuffs can be analysed with the method presented here.

This method can be applied to determine anthraquinone in concentrations ranging from 2 µg/l to 40 µg/l in the water and solvent extracts, corresponding to 0,05 mg/kg to 1 mg/kg pulp, paper and board or, respectively, 0,1 µg/dm² to 2 µg/dm² in the case of migration tests with MPPO. The measurement range can be lowered by enriching anthraquinone from the water and solvent extracts.

Table 1 — Anthraquinone

Name	Abbreviation	Formula	CAS N°	Structure
Anthraquinone	AQ	C ₁₄ H ₈ O ₂	84-65-1	

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

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

EN 14338, *Paper and board intended to come into contact with foodstuffs — Conditions for determination of migration from paper and board using modified polyphenylene oxide (MPPO) as a simulant*

EN 15519, *Paper and board intended to come into contact with foodstuffs — Preparation of an organic solvent extract*

EN 27213, *Pulps — Sampling for testing (ISO 7213)*

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)*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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/ui>

4 Principle

4.1 General

Anthraquinone is extracted from pulp, paper and board with water and/or 95 % ethanol according to EN 645, EN 647 and/or EN 15519, and with acetone from MPPPO which was brought into contact with the materials as a simulant for dry, non-fatty foodstuffs according to EN 14338. GC-MS is used to determine the anthraquinone content in the prepared extracts.

4.2 Interferences

Matrix related interferences can occur. In these cases, the quantification of anthraquinone may be performed by using one of the qualifier ions (12.1.2) or by standard addition.

5 Materials

5.1 Pasteur pipettes.

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5.2 Piston pipettes with a capacity adjustable from 3 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 2 ml, 5 ml and 10 ml.

5.4 Volumetric flasks with a capacity of 5 ml, 10 ml, 25 ml and 100 ml.

5.5 Pear shaped flasks with a capacity adapted to the volume of the extract, e.g. 25 ml.

5.6 Glass flasks with polytetrafluoroethylene (PTFE) lined screw caps with a capacity adapted to the volume of the extract, e.g. 25 ml.

5.7 Disposable screw thread glass vials with a capacity of 1,5 ml and the corresponding Polypropylene (PP) caps with silicone/PTFE gaskets.

6 Apparatus

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

6.2 Rotary evaporator.

6.3 Gas chromatograph coupled to a mass spectrometer (GC-MS).

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7 Reagents

All reagents should be of analytical grade or better.

7.1 Water, grade 1 according to ISO 3696.

7.2 Ethanol, a volume fraction of 95 %.

7.3 Toluene, ≥ 99 %.

7.4 Acetone, ≥ 99 %.

7.5 Anthracene-d10 (internal standard), CAS N° 1719-06-8.

Commercial ready-to-use solutions at 10 mg/l in cyclohexene are available.

7.5.1 Intermediate solution of the internal standard at 800 µg/l in ethanol.

2 ml of the internal standard solution (7.5) are pipetted into a 25 ml volumetric flask and made up to volume with ethanol (7.2).

7.6 Anthraquinone, $C_{14}H_8O_2$, CAS N° 84-65-1, $\geq 97\%$.

7.7 Solutions of anthraquinone

7.7.1 Stock solution of anthraquinone at 250 mg/l in toluene.

In a 100 ml volumetric flask, 25 mg anthraquinone (7.6) with an accuracy of 0,000 1 g are weighted and toluene (7.3) is added. The flask is then placed in an ultrasonic bath to dissolve the anthraquinone. After cooling down to room temperature, the flask is made up to volume with toluene.

7.7.2 Intermediate solution A1-95E of anthraquinone at 20 mg/l in 95 % ethanol.

800 µl of the stock solution (7.7.1) are pipetted into a 10 ml volumetric flask and made up to volume with 95 % ethanol (7.2).

7.7.3 Intermediate solution A2-95E of anthraquinone at 1 mg/l in 95 % ethanol.

500 µl of the intermediate solution A1-95E (7.7.2) are pipetted into a 10 ml volumetric flask and made up to volume with 95 % ethanol (7.2).

7.7.4 Intermediate solution B1-T of anthraquinone at 20 mg/l in toluene.

800 µl of the stock solution (7.7.1) are pipetted into a 10 ml volumetric flask and made up to volume with toluene (7.3).

7.7.5 Intermediate solution B2-T of anthraquinone at 1 mg/l in toluene

500 µl of the intermediate solution B1-T (7.7.4) are pipetted into a 10 ml volumetric flask and made up to volume with toluene (7.3).

7.8 Calibration solutions of anthraquinone

7.8.1 Calibration solutions of anthraquinone in 95 % ethanol.

Five calibration solutions are prepared by pipetting 10 µl, 25 µl, 50 µl, 100 µl and 200 µl, respectively, of the intermediate solution A2-95E (7.7.3) into 5 ml volumetric flasks. Moreover, 125 µl of the internal standard intermediate solution (7.5.1) are added to each flask. These are made up to volume with

95 % ethanol (7.2). The anthraquinone concentrations in these calibration solutions are about 2 µg/l, 5 µg/l, 10 µg/l, 20 µg/l and 40 µg/l, and that of the internal standard anthracene-d10 is 20 µg/l in each solution.

7.8.2 Calibration solutions of anthraquinone in toluene.

Five calibration solutions are prepared by pipetting 10 µl, 25 µl, 50 µl, 100 µl and 200 µl, respectively, of the intermediate solution B2-T (7.7.5) into 5 ml volumetric flasks. Moreover, 125 µl of the internal standard intermediate solution (7.5.1) are added to each flask. These are made up to volume with toluene (7.3). The anthraquinone concentrations in these calibration solutions are about 2 µg/l, 5 µg/l, 10 µg/l, 20 µg/l and 40 µg/l, and that of the internal standard anthracene-d10 is 20 µg/l in each solution.

8 Sampling

If the analysis is performed to evaluate a batch of pulp, paper or board, the sample shall be selected in accordance with EN 27213 or 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 solutions from water extracts

The water extractions of pulp, paper and board samples shall be carried out in accordance with EN 645 or EN 647. In parallel to the sample extractions, blank solutions have to be prepared but without adding the internal standard solution (see below).

Of these water extracts, in each case 10 ml are transferred into a glass flask and 250 µl of the internal standard intermediate solution (7.5.1) as well as 10 ml toluene (7.3) are added. This mixture is intensively shaken for approximately 5 min. When the phases have separated, an aliquot of the organic phase is pipetted into a glass vial and directly subjected to GC-MS analysis (Clause 12). If needed, a concentrated solution can be obtained by using more of the water extract and/or by adding less toluene. The volume of the internal standard intermediate solution has to be adapted according to the concentration factor so that the amount of anthracene-d10 in the analysis and calibration solutions is the same, i.e. 20 µg/l.

10 Preparation of the analysis solutions from 95 % ethanol extracts

The 95 % ethanol extractions of pulp, paper and board samples shall be carried out in accordance with EN 15519 for 2 h at 60 °C. In parallel to the sample extractions, blank solutions have to be prepared but without adding the internal standard solution (see below).

Of these 95 % ethanol extracts, in each case 1 ml is pipetted into a glass vial and mixed with 25 µl of the internal standard intermediate solution (7.5.1). This solution is directly subjected to GC-MS analysis (Clause 12).

FprCEN/TS 17630:2021 (E)**11 Preparation of the analysis solutions from MPP0**

The migration tests of pulp, paper and board samples with MPP0 and its extraction shall be carried out in accordance with EN 14338. The solvent used for the extraction of the MPP0 is acetone (7.4). The pursuant to EN 14338 prepared MPP0 blank sample is extracted with acetone as well, but no internal standard is added (see below).

Of these acetone extracts, in each case 5 ml are transferred into a pear shaped flask and the solvent is evaporated to dryness using a rotary evaporator. The residue is redissolved in 5 ml toluene (7.3) containing 125 µl of the internal standard intermediate solution (7.5.1). An aliquot of the analysis solution is pipetted into a glass vial and directly subjected to GC-MS analysis (Clause 12). If needed, a concentrated solution can be obtained by using more of the acetone extract and/or by adding less toluene. The volume of the internal standard intermediate solution has to be adapted according to the concentration factor so that the amount of anthracene-d10 in the analysis and calibration solutions is the same, i.e. 20 µg/l.

12 Procedure**12.1 Example of suitable GC-MS conditions for the analysis of anthraquinone****12.1.1 GC conditions**

- Column: Phase: 95 % dimethylpolysiloxane, 5 % diphenylpolysiloxane; 30 m; 0,25 mm inner diameter; 0,5 µm film thickness;
- Carrier gas: Helium;
- Injector: 280 °C; high-pressure-injection (180 kPa, 1 min);
- Injection volume: 1 µl (splitless injection);
- Linear velocity: 40 cm/s;
- Temperature programme: 100 °C (1 min); 10 °C/min to 240 °C (0 min); 15 °C/min to 290 °C (15 min);
- Interface: 290 °C.

12.1.2 MS conditions

- Ionization: electron impact, negative mode;
- Source: 230 °C;
- Solvent delay: 4,5 min;
- Single ion chromatograms ± 0,75 min around the retention time of anthraquinone and anthracene-d10;
- Target ion for anthraquinone: 180 *m/z*;
- Qualifier ions for anthraquinone: 76 *m/z*, 151 *m/z*, 152 *m/z*;
- Target ion for the internal standard anthracene-d10: 188 *m/z*.