



SLOVENSKI STANDARD

oSIST prEN 17917:2023

01-januar-2023

Papir, karton in lepenka - Papier, karton in lepenka, namenjeni neposrednemu stiku z živilni - Ugotavljanje aluminija v vodnih ekstraktih

Paper and board - Paper and board intended to come into contact with foodstuffs - Determination of aluminium in aqueous extracts

Papier und Pappe - Papier und Pappe für den Kontakt mit Lebensmitteln - Bestimmung von Aluminium in wässrigen Extrakten

Papier et carton - Papiers et cartons destinés à entrer en contact avec les denrées alimentaires - Détermination de l'aluminium dans un extrait aqueux

Ta slovenski standard je istoveten z: prEN 17917

ICS:

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

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en,fr,de

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

DRAFT
prEN 17917

October 2022

ICS 67.250; 85.060

English Version

Paper and board - Paper and board intended to come into contact with foodstuffs - Determination of aluminium in aqueous extracts

Papier et carton - Papiers et cartons destinés à entrer en contact avec les denrées alimentaires - Détermination d'aluminium dans un extrait aqueux

Papier, Karton und Pappe - Papier, Karton und Pappe für den Kontakt mit Lebensmitteln - Bestimmung von Aluminium in wässrigen Extrakten

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

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 17917:2022) 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 CEN Enquiry.

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prEN 17917:2022 (E)**1 Scope**

This document is one in a series of standards for the determination of metals in aqueous extracts of paper and board intended for contact with food. This document specifies the test method for the determination of aluminium in aqueous extracts.

It is applicable to paper and board with extractable metal contents exceeding

— 0,2 mg aluminium per l extract.

Aluminium extract levels below those given can be measured using this document if sensitive equipment is available and if all other laboratory conditions fulfil the requirements for trace element analysis.

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*

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

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

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 <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

An aliquot portion from the cold water extract (see EN 645) or hot water extract (see EN 647) (see Clause 7) is analyzed by inductively coupled plasma optical emission spectrometry (ICP OES) or by inductively coupled plasma mass spectrometry (ICP MS).

NOTE AAS (atomic absorption spectrometry with graphite furnace) can be used, if validated.

5 Reagents

All reagents and the water used shall be suitable for trace element analysis. Store the solutions in high density polyethylene/polypropylene bottles.

- 5.1 Water grade 1** according to EN ISO 3696 ($\leq 0,01$ mS/m).
- 5.2 Nitric acid (HNO₃)**, a mass fraction of 65 % or similar quality.
- 5.3 Nitric acid (5.2)**, diluted to a volume fraction of 7 % with water (5.1).
- 5.4 Aluminium stock solution (AL)** with a concentration of 1 000 mg/l.

The stock solutions are reference materials such as defined in the ISO guide 30 [1]. The solution with a single element with their suited specifications indicating the acid used and the method of preparation is commercially available. The solutions of aluminium having different concentrations (for example 100 mg/l) are also permitted.

NOTE Solutions of multi-element standards are also permitted.

6 Apparatus

6.1 General laboratory equipment

NOTE It is possible to wash glassware with diluted nitric acid (5.3) before use to reduce blank values.

6.2 Micropipettes, with plastic tips polyethylene/polypropylene of high density.

6.3 Syringe filters 0,45 µm (e.g. Polytetrafluoroethylene, Polyamide)

6.4 ICP OES

Measuring instrument with an optical emission spectrometer with background correction.

6.5 ICP MS

A mass spectrometer with inductively coupled plasma (ICP) for multi-element determinations is required. The spectrometer shall offer a resolution of at least 1 m_r/z peak width at 5 % peak height (m_r = relative mass of the type of atom, z = valence).

7 Preparation of sample

Prepare a cold water extract or a hot water extract from the paper or board using the test methods described in EN 645 or EN 647 respectively using water (5.1).

The extracts shall be directly membrane filtered (6.3). The filtrate is stabilized by addition of nitric acid (5.2) in the ratio of 1,0 ml per 100 ml of extract.

8 Procedure

8.1 General

Detailed instructions depend on the equipment (6.4, 6.5) used. Follow the instructions of the manufacturer of the equipment.

8.2 Preparation of calibration solutions

The concentrations of the calibration solutions should cover the total range of concentrations being measured (e.g. 0,2 mg/l to 5 mg/l).

8.3 Determination of calibration blank

Prepare a calibration blank using all the reagents except for the metal stock solutions.

8.4 Determination of aluminium

8.4.1 General

Determine the concentration of the element by means of the calibration.

prEN 17917:2022 (E)**8.4.2 Suggested spectrometer settings****8.4.2.1 ICP OES**

The following wavelengths should be used: 396,152 nm, 308,215 nm or 237,312 nm.

8.4.2.2 ICP MS

The isotope ^{27}Al shall be used for the measurement.

8.4.3 Determination of blank value

Submit the water, the reagents and the equipment to the extraction procedure to provide a blank value to be deducted from the extract value.

9 Expression of results

Calculate the results under consideration of the moisture content of the sample. Take the blank value (8.4.3) into consideration in the evaluation. Express the results according to Formula (1) in mg/l of the extract.

$$C_l = (C_m - b) \times \frac{10}{G} \times \frac{100}{(100 - f)} \quad (1)$$

where

C_l amount of aluminium in mg/l extract;

C_m concentration of aluminium in mg/l measured;

b blank value;

f moisture content of the sample;

G sample weight in g per 250 ml.

Element determinations are sensitive to a number of sources of error. It is, therefore, recommended that the performance of the system be checked by running certified reference materials.

Special attention should be paid to factors such as high blank levels caused by impure reagents, contamination during handling of the solutions, adsorption on the walls of vessels, inadequate background correction or unmatched acid concentrations of sample and calibration solutions.

10 Test report

The test report shall refer to this document and state:

- a) a reference to this document, i.e. prEN 17917:2022;
- b) extraction method (EN 645 or EN 647);
- c) type, origin and designation of sample;
- d) date of receipt and date of analysis;
- e) analysis test result;
- f) any deviation from this document.

11 Precision

In the year 2020, an international round robin trial on this subject was arranged [2].

From this test ($n = 12$) with two samples of a cold water extract from one uncoated and one coated paper, the test method above gave the overall standard deviation shown in Tables 1 and 2.

Table 1 — Repeatability

Sample	Mean value of Al mg/l	Standard deviation s_r mg/l	Coefficient of variation $C_{v,r}$ %	Repeatability limit r mg/l
1	1,42	0,06	4,4	0,17
2	4,90	0,29	5,9	0,80

Table 2 — Reproducibility

Sample	Mean value of Al mg/l	Standard deviation s_R mg/l	Coefficient of variation C_{vR} %	Reproducibility limit R mg/l
1	1,42	0,21	15,0	0,59
2	4,90	1,32	26,9	3,96

Bibliography

- [1] ISO GUIDE 30, *Reference materials — Selected terms and definitions*
- [2] Report of the round robin trial

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