SLOVENSKI STANDARD
SIST ENV 12498:2000

01-april-2000

Papir, karton in lepenka - Papir, karton in lepenka v neposrednem stiku z živili - Določevanje kadmija, svinca in kroma v vodnem ekstraktu

Paper and board - Paper and board intended to come into contact with foodstuffs - Determination of cadmium, lead and chromium in an aqueous extract

Papier und Pappe - Papier und Pappe für den Kontakt mit Lebensmitteln - Bestimmung von Cadmium, Blei und Chrom in einem wäßrigen Extrakt

Papier et carton - Papiers et cartons destinés à entrer en contact avec les denrées alimentaires - Détermination du cadmium, du plomb et du chrome dans un extrait aqueux

Ta slovenski standard je istoveten z: **ENV 12498:1997**

ICS:

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

SIST ENV 12498:2000 **en**

EUROPEAN PRESTANDARD
PRÉNORME EUROPÉENNE
EUROPÄISCHE VORNORM

ENV 12498

October 1997

ICS 67.250; 85.060

Descriptors: paper, paperboards, food products, food-container contact, chemical analysis, determination of content, cadmium, lead, chromium, aqueous extract, atomic absorption spectrometry

English version

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This European Prestandard (ENV) was approved by CEN on 6 March 1997 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

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

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Ref. No. ENV 12498:1997 E

Content

Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 172 "Pulp, paper and board", the secretariat of which is held by DIN.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Prestandard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

CEN/TC 172 has decided to publish this test method as a European Prestandard (ENV) because the validation of the test method could not be carried out due to the fact that until now the levels found were below the limit of determination.

1 Scope

This European Prestandard is one in a series of Prestandards for the determination of heavy metals in an aqueous extract of paper or board intended for contact with food. This European Prestandard specifies the test method for the determination of cadmium, lead and chromium in an aqueous extract.

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

- 0,1 mg per kg for cadmium;
- 0,6 mg per kg for lead;
- 2,0 mg per kg for chromium.

NOTE 1: The above limits of determination are achieved by multiplying the actual limits existing today or proposed in Europe by 0,2.

NOTE 2: Metal content levels below those given can be measured by this European Prestandard if very sensitive equipment is available and if all other laboratory conditions fulfill the requirements for trace element analysis.

2 Normative references

This European Prestandard incorporates by dated and 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 and revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to 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

3 Principle

An aliquot portion from the stabilized cold water (see EN 645) or stabilized hot water extract (see EN 647) (see clause 6) is analysed by atomic absorption spectrometry using a graphite tube furnace.

4 Reagents

All reagents and the water used shall be suitable for trace element analysis.

Store the solutions in high densitive Polyethylene/Polypropylene bottles.

4.1 Nitric acid (HNO_3), 65% (d = 1,42)

4.1.1 Nitric acid, (4.1), diluted 1 : 1 (*V*/*V*) with water

4.1.2 Nitric acid, (4.1), diluted 1% (*V*/*V*) with water

4.2 Hydrochloric acid (HCl), 36% (d = 1,19)

4.3 Hydrochloric acid (HCl), 0,3 mol/l solution

4.4 Cadmium - stock solution, (Cd) = 1000 mg/l

Dissolve 1,142 g of cadmium oxide (CdO) in the minimum volume of nitric acid (4.1.1). Make up to 1000 ml with nitric acid (4.1.2).

4.5 Lead - stock solution, (Pb) = 1000 mg/l

Dissolve 1,598 g of lead nitrate ($Pb(NO_3)_2$) in the minimum volume of nitric acid (4.1.1). Make up to 1000 ml with nitric acid (4.1.2).

4.6 Chromium - stock solution, (Cr) = 1000 mg/l

Dissolve 1,923 g of chromium (VI) oxide in 15,0 ml of nitric acid (4.1.1) Make up to 1000 ml with nitric acid (4.1.2).

4.7 Matrix modifiers

4.7.1 Ammonium dihydrogen phosphate ($NH_4H_2PO_4$) solution 100,0 g/l

4.7.2 Palladium nitrate ($Pd(NO_3)_2$) solution, 21,7 g/l (= 1% Pd)

4.7.3 Magnesium nitrate ($Mg(NO_3)_2$) solution, 61,0 g/l (= 1% Mg)

NOTE: Commercially available standard solutions and matrix modifiers may be used if preferred.

4.8 Gases for atomic absorption spectrometry

- Nitrogen
- Argon

as appropriate

5 Apparatus

5.1 General laboratory equipment

5.2 Volumetric flasks, 1000 ml

5.3 Analytical balance, accuracy 0,1 mg

5.4 Micropipettes from 5,0 µl to 20,0 µl with plastique tips (high densitive Polyethylene/Polypropylene bottles)

5.5 Atomic absorption spectrometer with graphite tube furnace, and with background correction

NOTE: Wash all flasks, pipettes etc. with nitric acid before use and stored in dilute nitric acid (4.1.2) until required. Rinse with demineralized water before use.

6 Preparation of sample

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

Stabilize the extract by the addition of nitric acid (4.1) in the ratio of 1,0 ml per 100,0 ml of sample.

Mix the aqueous extract well and take an aliquot portion for investigation.

Use the appropriate matrix modifier(s) (4.7) according to the particular instrument in use and the analytical matrix of the extract.

7 Procedure

7.1 General

Detailed instructions depend on the form of the equipment used. Follow the instructions of the manufacturer of the equipment.

Correct the background absorption by use of a suitable system.

7.2 Preparation of reference solutions

Prepare the reference solutions daily by diluting the single element solutions with nitric acid (4.1.2). The concentration to be selected will depend on the instrument used and the expected concentrations in the extract. However, reference solutions containing 10,0 µg/l are usually appropriate. Except for cadmium a reference solution where a cadmium concentration of 1,0 µg/l is appropriate.

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

7.3 Determination of the elements

7.3.1 General

Two parallel extractions shall be carried out. From each extract at least two parallel determinations shall be carried out.

Determine the concentration of the element by means of the calibration graph (7.3.2) or alternatively, by use of the method of standard addition.

7.3.2 Calibration curve

The calibration curve shall contain at least 3 points and cover the total range of concentrations being measured. Calculate the concentration of the element from the measured absorption.

7.3.3 Suggested spectrometer settings

- Cadmium 228,8 nm
- Lead 217,0 nm or
  283,3 nm (preferred)
- Chromium 357,9 nm

7.3.4 Determination of blank value

Submit the water and reagents used for the extraction to the test procedure to provide a blank value to be deducted from the extract value.

NOTE: Although not deprecated, the extract can only be supplied to the laboratory, together with the water used for the extraction. Without this, no blank can be determined and therefore not deducted from the extract value. If a partial blank is determined this should be reported.

8 Expression of results

Calculate the results with a computer or graphically. Correct, where appropriate, for background absorption. Take the blank value into consideration in the evaluation.

Express the results in mg/l or µg/l of the extract.

NOTE 1: The extractable metals content of the original paper or board may be calculated if data are available.

NOTE 2: Trace element determinations are sensitive to a number of sources of error. It is, therefore, recommended to check the performance of the system by running standard reference materials.

Standard reference solutions are commercially available.

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

The detection limit should be established by measuring a sufficient number of blanks to allow calculation of the standard deviation of the blank. The detection limit is determined as three times this standard deviation.

9 Test report

The test report shall refer to this European Prestandard and state:

a) extraction method;

b) type, origin and designation of sample;

c) date of sampling;

d) whether or not a blank value has been measured on the water used;

e) date of receipt and date of investigation;

f) investigation test result;

g) any deviation from this European Prestandard.