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Paper and board - Paper and board intended to come into contact with foodstuffs -
Determination of mercury in an aqueous extract

Papier und Pappe - Papier und Pappe für den Kontakt mit Lebensmitteln - Bestimmung
von Quecksilber in einem wässrigen Extrakt

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

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Ta slovenski standard je istoveten z: ENV 12497:1998

ICS:

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

SIST ENV 12497:2000

en

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EUROPEAN PRESTANDARD
PRÉNORME EUROPÉENNE
EUROPÄISCHE VORNORM

ENV 12497

May 1998

ICS 67.250; 85.060

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

English version

Paper and board - Paper and board intended to come into contact with foodstuffs - Determination of mercury in an aqueous extract

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

Papier und Pappe - Papier und Pappe für den Kontakt mit Lebensmitteln - Bestimmung von Quecksilber in einem wässrigen Extrakt

This European Prestandard (ENV) was approved by CEN on 18 April 1998 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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels


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 IRELAND
 NATIONAL STANDARDS AUTHORITY OF IRELAND
 IRISH STANDARD
 SIST ENV 12497:2000
 ENVIRONMENTAL PROTECTION
 DETERMINATION OF
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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 implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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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 and paperboard intended for contact with food. This European Prestandard specifies the test method for the determination of mercury in an aqueous extract.

It is applicable to paper and board with extractable mercury content exceeding 0,06 mg per kg.

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

NOTE 2: Mercury content levels below 0,06 mg per kg can be measured by this European Prestandard if very sensitive equipment is available and if all other laboratory conditions fulfil 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 of 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 cold vapour generation.

4 Reagents

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

Store the solutions in high density polyethylene/polypropylene bottles.

4.1 Nitric acid (HNO₃), 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 to 1,5% (V/V) with water

4.2 Potassium permanganate (KMnO₄), 5% aqueous solution (*m/V*)

NOTE: Potassium permanganate solution is used to prepare the mercury stock solution. It is not needed if a commercially available standard solution is used (see 4.3).

4.3 Mercury, stock solution 1000 mg/l

Warning: Mercury is toxic.

Dissolve 1,080 g of mercury(II) oxide (Hg O) in the minimum volume of nitric acid (4.1.1). Add 0,2 ml of potassium permanganate solution (4.2) and make up to 1000 ml with water.

NOTE: Commercially available standard solutions can be used if preferred.

4.4 Sulfuric acid (H₂SO₄), (*d* = 1,84)

4.5 Potassium dichromate ($K_2Cr_2O_7$), 50 g/l in sulfuric acid solution

Warning: Potassium dichromate is carcinogenic.

Dissolve 5 g of potassium dichromate in 80 ml of water. Add with caution 5 ml of sulfuric acid (4.4) and dilute with water to 100 ml.

4.6 Hydroxylammonium chloride ($HONH_3Cl$), 20 g/l aqueous solution.

Dissolve 5 g of hydroxylammonium chloride in 250 ml of water.

4.7 Reducing solutions**4.7.1 Tin(II) chloride ($SnCl_2 \cdot 2H_2O$), 50 g/l in 10% hydrochloric acid (4.8.1)****4.7.2 Sodium tetrahydroborate ($NaBH_4$), 0,2 g/l in 0,05% sodium hydroxide solution (4.9)**

NOTE: Either tin chloride or sodium tetrahydroborate should be used depending on the type of spectrometer. Follow the instructions provided by the manufacturer of the instrument.

4.8 Hydrochloric acid (HCl), 36% ($d = 1,19$)**4.8.1 Hydrochloric acid (4.8) (HCl), diluted 10% (V/V)**

NOTE: Hydrochloric acid is used only together with tin (II) chloride (see 4.7.1 and 7.3.1).

4.9 Sodium hydroxide (NaOH), 0,05% aqueous solution (m/V)

NOTE: Sodium hydroxide solution is used only together with sodium tetrahydroborate (see 4.7.2 and 7.3.1).

5 Apparatus**5.0 General**

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

5.1 General laboratory equipment**5.2 Volumetric flasks, 1000 ml****5.3 Analytical balance, accuracy 0,1 mg****5.4 Pipettes from 100 μ l to 10 ml, glass or plastics, (high density polyethylene/polypropylene)****5.5 Atomic absorption spectrometer with an appropriate detection system and sensitivity****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.

Two parallel extractions shall be carried out.

Stabilize the extract by adding nitric acid (4.1) in the ratio of 3,5 ml per 100 ml of extract. Add potassium dichromate solution (4.5) to a content of approximately 10 mg of potassium dichromate per 100 ml of extract.

NOTE: Organic mercury compounds will not respond to the flameless technique unless they are decomposed into mercury (II) ions. Potassium dichromate oxidizes these compounds.

7 Procedure**7.1 General**

Absorb the exhaust from the spectrometer in a suitable mercury absorbent. The following solution is suitable.

- Iodide 2,5 g;
- Potassium iodid 30,0 g;
- Make up to 1000 ml with water.

7.2 Preparation of reference solution

Prepare the reference solution daily. Dilute the stock solution (4.3) with nitric acid (4.1.2) to the appropriate concentration. A concentration of 10,0 µg/l is usually appropriate.

7.3 Determination of mercury

7.3.1 General

From each stabilized extract (6) at least two parallel determinations shall be carried out.

Add 4 ml of hydroxyl ammonium chloride solution (4.6) per 100 ml of extract to inactivate the surplus of potassium dichromate.

Follow the instructions given by the manufacturer of the spectrometer in order to reduce the mercury(II) ions to mercury. The reducing agent to be used is either tin(II) chloride (4.7.1) or sodium tetrahydroborate (4.7.2), and the appropriate amount is specified in the instructions.

The details of the measurement depends on the type of spectrometer. Follow the instructions and record the mercury peak.

7.3.2 Standard additions

The matrix of some samples is such that it is impossible to record the mercury peak. In such a case the standard addition method may be useful. The following is a guide to the application of standard additions:

- stabilized extract 50 ml;
- stabilized extract 50 ml + (*M*) mg mercury;
- stabilized extract 50 ml + (*2M*) mg mercury.

(*M*) represents a known mass of mercury added by using a suitable volume of the mercury stock solution (4.3) and (*2M*) represents double this volume. The masses selected shall give clear readings on the instrument.

Follow the further instructions provided in 7.3.1.

7.4 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 should 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. 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 mercury 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.

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. The limit of determination is determined as three times the limit of detection.

Standard reference solutions are commercially available.

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) date of receipt and date of investigation;