BRITISH STANDARD

BS EN
12497:2005

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

The European Standard EN 12497:2005 has the status of a British Standard

ICS 67.250; 85.060

BSI
British Standards

National foreword

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The UK participation in its preparation was entrusted by Technical Committee CW/47, Materials and articles in contact with foodstuffs, to Subcommittee CW/47/3, Paper and board in contact with foodstuffs, which has the responsibility to:

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Summary of pages

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EUROPEAN STANDARD

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EUROPÄISCHE NORM

EN 12497

August 2005

ICS 67.250; 85.060

Supersedes ENV 12497:1998

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

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Foreword

This European Standard (EN 12497:2005) has been prepared by Technical Committee CEN/TC 172 “Pulp, paper and board”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.

This European Standard supersedes ENV 12497:1998. With regard to ENV 12497:1998 the following changes have been made:

a) implementation in a European Standard;

b) addition of the clause "Precision";

c) editorial updating.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard is one in a series of standards for the determination of heavy metals in an aqueous extract of paper and paperboard intended for contact with food. This European Standard 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 5 times below the actual limit existing today or proposed in Europe.

NOTE 2 Mercury content levels below 0,06 mg per kg can be measured by this European Standard, if very sensitive equipment is available and if all other laboratory conditions fulfil the requirements for trace element analysis.

2 Normative references

The following referenced documents are indispensable for the application of this European Standard. 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*

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

4.1 General

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

Store the solutions in high-density polyethylene/polypropylene bottles.

4.2 Nitric acid (HNO3), 65 % (d = 1,42)

4.2.1 Nitric acid (4.2), diluted 1 : 1 (*V/V*) with water

4.2.2 Nitric acid (4.2), diluted to 1,5 % (*V/V*) with water

4.3 Potassium permanganate ($KMnO_4$), 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 4.4).

4.4 Mercury, stock solution, 1000 mg/l

Warning: Mercury is toxic.

Dissolve 1,080 g of mercury(II)oxide (HgO) in the minimum volume of nitric acid (4.2.1). Add 0,2 ml of potassium permanganate solution (4.3) and make up to 1000 ml with water.

NOTE Commercially available standard solutions can be used if preferred.

4.5 Sulphuric acid (H_2SO_4), (d = 1,84)

4.6 Potassium dichromate ($K_2Cr_2O_7$), 50 g/l in sulphuric acid solution

Warning: Potassium dichromate is carcinogenic.

Dissolve 5 g of potassium dichromate in 80 ml of water. Add with caution 5 ml of sulphuric acid (4.5) and dilute with water to 100 ml.

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

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

4.8 Reducing solutions

4.8.1 Tin (II) chloride ($SnCl_2 \cdot 2H_2O$), 50 g/l in 10 % hydroxhloric acid (4.9.1)

4.8.2 Sodium tetrahydroborate ($NaBH_4$), 0,2 g/l in 0,05 % sodium hydroxide solution (4.10)

NOTE Either tin chloride or sodium tetrahydroborate should be used depending on the type of spectrometer. It is recommended to follow the instructions provided by the manufacturer of the instrument.

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

4.9.1 Hydrochloric acid (4.9) (HCl), diluted 10 % (*V/V*)

NOTE Hydrochloric acid is used only together with tin (II) chloride (see 4.8.1 and 7.3.1).

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

NOTE Sodium hydroxide solution is used only together with sodium tetrahydroborate (see 4.8.2 and 7.3.1).

5 Apparatus

5.1 General

All flasks, pipettes etc. have to be washed with nitric acid before use and stored in dilute nitric acid (4.2.2) until required. Rinse with demineralized water before use.

5.2 General laboratory equipment

5.3 Volumetric flasks, 100 ml

5.4 Analytical balance, accuracy 0,1 mg

5.5 Pipettes from 100 µl to 10 ml, glass or plastics, (high density polyethylene/polypropylene)

5.6 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.

Carry out two parallel extractions.

Stabilize the extract by adding nitric acid (4.2) in the ratio of 3,5 ml per 100 ml of extract.

Store the extract in glass bottles and analyze as soon as possible because the extract is not stable. Decrease of concentration within two weeks is observed.

Add potassium dichromate solution (4.6) 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 iodide 30 g;
— Make up to 100 ml with water.

7.2 Preparation of reference solution

Prepare the reference solution daily. Dilute the stock solution (4.4) with nitric acid (4.2.2) to the appropriate concentration. A concentration of 10,0 µg/l is usually appropriate.

7.3 Determination of mercury

7.3.1 General

Carry out at least two parallel determinations from each stabilized extract (see Clause 6).

Add 4 ml of hydroxyl ammonium chloride solution (4.7) 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 (I)chloride (4.8.1) or sodium tetrahydroborate (4.8.2), 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 + (2*M*) mg mercury.

(*M*) represents a known mass of mercury added by using a suitable volume of the mercury stock solution (4.4) and (2*M*) 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/kg or mg/dm² of paper.

Calculate the mercury content of the sample (C_m see formula (1), C_s see formula (2)) as follows:

$$C_{\mathrm{m}} = C \cdot V_0 \cdot \frac{1}{G} \cdot \frac{100}{100 - f} \cdot \frac{1}{1000} \qquad (1)$$

$$C_{\mathrm{s}} = \frac{C}{1000} \cdot \frac{V_0}{1000} \cdot \frac{1}{G} \cdot \frac{b}{100} \qquad (2)$$

where:

C_m amount of mercury soluble of the sample in mg/kg;

C_s amount of mercury soluble of the sample in mg/dm^2;

C concentration of mercury read from the calibration graph, in µg/l;

V_0 total volume of extract, in ml;

b grammage, in g/m^2;

f moisture content of the sample, in %;

G mass of the sample taken under the same condition as grammage, in g.

NOTE 1 The extractable mercury content of the original paper or board can 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 Precision

From an interlaboratory test (n = 8) with two samples of a water extract with a known amount of added mercury the test method described above gave the overall standard deviation as shown in Table 1.

Table 1

Sample	Mean value of Hg µg/l	Standard deviation (s) µg/l	Reference value (added mercury) µg/l
1	1,66	0,63	3,5
2	6,28	2,23	10

10 Test report

The test report shall include the following information:

a) reference to this European Standard;

b) extraction method;

c) type, origin and designation of sample;

d) date of sampling;

e) date of receipt and date of analyses;

f) test result;

g) whether or not a blank test has been made;

h) any departures from the specified procedure that may have affected the result.

blank

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