

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

The European Standard EN 12498:2005 has the status of a  
British Standard

ICS 67.250; 85.060

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## Foreword

This European Standard (EN 12498:2005) 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of an identical text or by endorsement, at the latest by February 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.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 12498:1997. With regard to ENV 12498:1997, the following changes have been made:

- a) introduction of the "Inductively coupled plasma emission spectroscopy (ICP)";
- b) deletion of the chromium determination;
- c) addition of the clause "Precision";
- d) 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 or board intended for contact with food. This European Standard specifies the test method for the determination of cadmium and lead 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.

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 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 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 (AAS) using a graphite tube furnace or inductively coupled plasma emission spectroscopy (ICP).

## 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 (HNO<sub>3</sub>), 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 % (V/V) with water

### 4.3 Hydrochloric acid (HCl), 36 % ( $d = 1,19$ )

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

**4.5 Cadmium - stock solution (Cd) = 1000 mg/l**

Dissolve 1,142 g of cadmium oxide ( $\text{CdO}$ ) in the minimum volume of nitric acid (4.2.1). Make up to 1000 ml with diluted nitric acid (4.2.2).

**4.6 Lead - stock solution (Pb) = 1000 mg/l**

Dissolve 1,598 g of lead nitrate ( $\text{Pb}(\text{NO}_3)_2$ ) in the minimum volume of nitric acid (4.2.1). Make up to 1000 ml with diluted nitric acid (4.2.2).

**4.7 Matrix modifiers**

4.7.1 Ammonium dihydrogen phosphate ( $\text{NH}_4\text{H}_2\text{PO}_4$ ) solution, 100,0 g/l

4.7.2 Palladium nitrate ( $\text{Pd}(\text{NO}_3)_2$ ) solution, 21,7 g/l (= 1 % Pd)

4.7.3 Magnesium nitrate ( $\text{Mg}(\text{NO}_3)_2$ ) solution, 61,0 g/l (= 1 % Mg)

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

**4.8 Gases for atomic absorption spectrometry**

— Nitrogen  
— Argon

} as appropriate

**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, 1000 ml****5.4 Analytical balance, accuracy 0,1 mg**

**5.5 Micropipettes** from 5,0 µl to 20,0 µl with plastique tips (high density polyethylene/polypropylene bottles)

**5.6 Atomic absorption spectrometer**, with graphite tube furnace, and with background correction or inductively coupled plasma atomic emission spectrometer with background correction and radio frequency-generator

## 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 addition of nitric acid (4.2) in the ratio of 1,0 ml per 100,0 ml of sample.

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

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 (4.2.2). The concentration to be selected will depend on the instrument used and the expected concentrations in the extract. For lead, a reference solution containing 10,0 µg/l is usually appropriate. For cadmium, a reference solution containing 1,0 µg/l is appropriate.

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

### 7.3 Determination of cadmium and lead

#### 7.3.1 General

Carry out two parallel extractions. 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)

### 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. Take the blank value into consideration in the evaluation.

Express the results in mg/kg or mg/dm<sup>2</sup> of paper.

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

$$C_m = C \cdot V_0 \cdot \frac{1}{G} \cdot \frac{100}{100-f} \cdot \frac{1}{1000} \quad (1)$$

$$C_s = \frac{C}{1000} \cdot \frac{V_0}{1000} \cdot \frac{1}{G} \cdot \frac{b}{100} \quad (2)$$

where:

- $C_m$  amount of cadmium or lead soluble of the sample in mg/kg;
- $C_s$  amount of cadmium or lead soluble of the sample in mg/dm<sup>2</sup>;
- $C$  concentration of cadmium or lead read from the calibration graph, in µg/l;
- $V_0$  total volume of extract, in ml;
- $b$  grammage, in g/m<sup>2</sup>;
- $f$  moisture content of the sample, in %;
- $G$  mass of the sample taken under the same condition as grammage, in g.

NOTE 1 The extractable cadmium, lead 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 Precision

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

Table 1

Sample	Mean value		Overall standard deviation (s)		Reference value (added cadmium/lead)	
	Cd µg/l	Pb µg/l	Cd µg/l	Pb µg/l	Cd µg/l	Pb µg/l
1	4,95	14,8	0,43	0,19	4,5	15
2	20,3	89,0	2,29	2,03	20	90

### 10 Test report

The test report shall include the following information:

- reference to this European Standard;
- extraction method;
- type, origin and designation of sample;
- date of sampling;
- whether or not a blank value has been measured on the water used;
- date of receipt and date of analyses;
- test result;
- any departures from the specified procedure that may have affected the result.