



SLOVENSKI STANDARD
SIST EN 12498:2005

01-december-2005

BUXca Yý U
SIST ENV 12498:2000

DUD]fž_Ufrcb`]b`YdYb_U!`DUD]fž_Ufrcb`]b`YdYb_Uj`bYdcgfYXbYa`gh_i`n`ýj`]]`È
8c`c`YjUb`Y`UXa`]U]b`gj`]bWUj`j`cXbYa`Y_gfU_hi

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

Papier und Pappe - Papier und Pappe für den Kontakt mit Lebensmitteln - Bestimmung
von Cadmium und Blei in einem wässrigen Extrakt
(standards.iteh.ai)

Papier et carton - Papiers et cartons destinés à entrer en contact avec les denrées
alimentaires - Détermination du cadmium et du plomb dans un extrait aqueux
fd3e4e2a1c64/sist-en-12498-2005

Ta slovenski standard je istoveten z: EN 12498:2005

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 EN 12498:2005 en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN 12498:2005

<https://standards.iteh.ai/catalog/standards/sist/53529a0f-9078-4180-8bbb-fd3e4e2a1c64/sist-en-12498-2005>

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 12498

August 2005

ICS 67.250; 85.060

Supersedes ENV 12498:1997

English Version

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

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

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

This European Standard was approved by CEN on 27 June 2005.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of 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.

<https://standards.iteh.ai/catalog/standards/sist/53529a0f-9078-4180-8bbb-fd3e4e2a1c64/sist-en-12498-2005>



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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

page

Foreword	3
1 Scope	4
2 Normative references	4
3 Principle	4
4 Reagents	4
4.1 General	4
4.2 Nitric acid (HNO ₃), 65 % (d = 1,42)	4
4.2.1 Nitric acid (4.2), diluted 1 : 1 (V/V) with water	4
4.2.2 Nitric acid (4.2), diluted to 1 % (V/V) with water	4
4.3 Hydrochloric acid (HCl), 36 % (d = 1,19)	4
4.4 Hydrochloric acid (HCl), 0,3 mol/l solution	4
4.5 Cadmium - stock solution (Cd) = 1000 mg/l	5
4.6 Lead - stock solution (Pb) = 1000 mg/l	5
4.7 Matrix modifiers	5
4.7.1 Ammonium dihydrogen phosphate (NH ₄ H ₂ PO ₄) solution, 100,0 g/l	5
4.7.2 Palladium nitrate (Pd(NO ₃) ₂) solution, 21,7 g/l (= 1 % Pd)	5
4.7.3 Magnesium nitrate (Mg(NO ₃) ₂) solution, 61,0 g/l (= 1 % Mg)	5
4.8 Gases for atomic absorption spectrometry	5
5 Apparatus	5
5.1 General	5
5.2 General laboratory equipment	5
5.3 Volumetric flasks, 1000 ml	5
5.4 Analytical balance, accuracy 0,1 mg	5
5.5 Micropipettes from 5,0 µl to 20,0 µl with plastique tips (high density polyethylene/polypropylene bottles)	6
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
6 Preparation of sample	6
7 Procedure	6
7.1 General	6
7.2 Preparation of reference solutions	6
7.3 Determination of cadmium and lead	6
7.3.1 General	6
7.3.2 Calibration curve	6
7.3.3 Suggested spectrometer settings	6
7.3.4 Determination of blank value	7
8 Expression of results	7
9 Precision	7
10 Test report	8

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.

SIST EN 12498:2005

<https://standards.iteh.ai/catalog/standards/sist/53529a0f-9078-4180-8bbb-fd3e4e2a1c64/sist-en-12498-2005>

EN 12498:2005 (E)**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

<https://standards.iteh.ai/catalog/standards/sist/53529a0f-9078-4180-8bbb-fd3e4e2a1c64/sist-en-12498-2005>

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 (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 % (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 (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 (Pb(NO₃)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 (NH₄H₂PO₄) solution, 100,0 g/l

4.7.2 Palladium nitrate (Pd(NO₃)₂) solution, 21,7 g/l (= 1 % Pd)

4.7.3 Magnesium nitrate (Mg(NO₃)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

iTeh STANDARD PREVIEW
} as appropriate
(standards.iteh.ai)

[SIST EN 12498:2005](https://standards.iteh.ai/catalog/standards/sist/53529a0f-9078-4180-8bbb-fd3e4e2a1c64/sist-en-12498-2005)

5 Apparatus <https://standards.iteh.ai/catalog/standards/sist/53529a0f-9078-4180-8bbb-fd3e4e2a1c64/sist-en-12498-2005>

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

EN 12498:2005 (E)

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/dm² 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/dm²;
 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/m²;
 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.