



# SLOVENSKI STANDARD

## SIST EN 1388-1:1997

01-januar-1997

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### Materiali in predmeti v stiku z živili - Silikatne površine - 1. del: Ugotavljanje izločanja svınca in kadmija iz keramičnih izdelkov

Materials and articles in contact with foodstuffs - Silicate surfaces - Part 1: Determination of the release of lead and cadmium from ceramic ware

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Silicatische Oberflächen - Teil 1: Bestimmung der Abgabe von Blei und Cadmium aus keramischen Gegenständen

Matériaux et articles en contact avec les denrées alimentaires - Surfaces silicatées - Partie 1: Détermination de l'émission de plomb et de cadmium par les articles en céramique

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Ta slovenski standard je istoveten z: **EN 1388-1:1995**

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#### **ICS:**

67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
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**SIST EN 1388-1:1997**

**en**

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EUROPEAN STANDARD

EN 1388-1

NORME EUROPÉENNE

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October 1995

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Descriptors: food industry, human nutrition, food, food-container contact, silicates, lead, cadmium, emission, ceramics, tests, chemical analysis, samples, determination of content, computation

English version

**Materials and articles in contact with foodstuffs -  
Silicate surfaces - Part 1: Determination of the  
release of lead and cadmium from ceramic ware**

Matériaux et articles en contact avec les  
denrées alimentaires - Surfaces silicatées -  
Partie 1: Détermination de l'émission de plomb  
et de cadmium par les articles en céramique

Werkstoffe und Gegenstände in Kontakt mit  
Lebensmitteln - Silicatische Oberflächen - Teil  
1: Bestimmung der Abgabe von Blei und Cadmium  
aus keramischen Gegenständen

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**CEN**

European Committee for Standardization  
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## Introduction

The problem of lead and cadmium release from ceramic ware requires effective means of control to ensure the protection of the population against possible hazards arising from the use of improperly formulated, applied and fired glazes and decorations on the food contact surfaces of ceramic ware used for the preparation, serving and storage of foodstuffs.

NOTE: Articles which are highly coloured or decorated on their food contact surfaces or which have a high surface area/volume ratio are more likely than other articles to release lead and/or cadmium.

The Council of the European Communities has adopted a specific Directive for the measurement of possible migration of lead and cadmium from ceramic articles which, in their finished state, are intended to come into contact with foodstuffs, or which are in contact with foodstuffs, and are intended for that purpose.

In Directive 84/500/EEC annex I describes "Basic rules for determining the migration of lead and cadmium". In annex II atomic absorption spectroscopy is prescribed as the method of analysis.

Technical Committee 194 of the European Committee for Standardization has undertaken the task of setting up European Standards for methods of test for silicate surfaces on the basis of the Directive and to harmonize at the same time existing national standards, International Standard ISO 6486 Part 1 was considered in this work.

This Part of the European Standard specifies the method of test for the determination of lead and cadmium released from ceramic surfaces. At the time of development of the Directive 84/500/EEC, flame atomic absorption spectroscopy (FAAS) was the usual method and this test method is used, although now other analytical methods exist.

## 1 Scope

This European Standard specifies a reference method for determining the release of lead and cadmium from ceramic articles which are intended to come into contact with foodstuffs.

## Foreword

This European Standard has been prepared by a Working Group (3) of CEN/TC 194 'Utensils in contact with food' of which the secretariat is held by BSI, as one of two standards for release of lead and cadmium. A further Part of this European Standard will be prepared with the following title:

EN 1388-2 *Materials and articles in contact with foodstuffs - silicate surfaces - Part 2: Release of lead and cadmium from silicate surfaces other than ceramic ware*

The text of the draft was submitted to the unique acceptance procedure and was approved by CEN as a European Standard.

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 April 1996, and conflicting national standards shall be withdrawn at the latest by April 1996.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of the EC Directive(s).

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According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

## 2 Normative references

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

- ISO 385-1 Laboratory glassware - Burettes - Part 1: General requirements
- ISO 385-2 Laboratory glassware - Burettes - Part 2: Burettes for which no waiting time is specified
- ISO 648 Laboratory glassware - One-mark pipettes
- ISO 835-1 Laboratory glassware - Graduated pipettes - Part 1: General requirements
- ISO 835-2 Laboratory glassware - Graduated pipettes - Part 2: Pipettes for which no waiting time is specified
- ISO 835-3 Laboratory glassware - Graduated pipettes - Part 3: Pipettes for which no waiting time of 15 s is specified
- ISO 1042 Laboratory glassware - One-mark volumetric flasks
- ISO 3585 Glass plant, pipeline and fittings - Properties of borosilicate glass 3.3
- ISO 3696 Water for analytical laboratory use - Specifications and test methods
- ISO 4788 Laboratory glassware - Graduated measuring cylinders

## 3 Definitions

For the purposes of this standard, the following definitions apply:

**3.1 foodware:** Articles which are intended to be used for the preparation, cooking, serving and storage of food or drinks, including tanks and vessels

**3.2 flatware:** Articles which cannot be filled and articles which can be filled, the internal depth of which, measured from the lowest point to the horizontal plane passing through the upper rim, does not exceed 25 mm.

NOTE: These articles are in category 1 of Directive 84/500/EEC.

**3.3 holloware:** Articles which can be filled the internal depth of which, measured from the horizontal plane passing through the lowest point of the upper rim, exceeds 25 mm, excepting holloware items having a filling volume exceeding 3 litres which are classed as storage vessels.

NOTE: Those articles with an internal depth of more than 25 mm are in category 2 of Directive 84/500/EEC and storage vessels are in category 3 of Directive 84/500/EEC.

**3.4 cooking ware:** Foodware, specifically intended to be heated in the course of preparation of food and drinks by means such as steaming, boiling, braising, stewing, roasting, baking or by microwaves.

NOTE: These articles are in category 3 of Directive 84/500/EEC.

**3.5 ceramic ware:** Ceramic articles which are intended to come into contact with foodstuffs, for example foodware made of china, porcelain and earthenware, whether glazed or not.

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**3.6 storage vessel:** Article having a capacity of 3 litres or more.

**3.7 test solution:** Solution used to extract lead and cadmium from silicate surfaces.

**3.8 extract solution:** Aqueous acidic solution obtained from the exposure of a silicate surface to the test solution.

**3.9 sample measuring solution:** Solution used for measuring the concentration of the analyte, and may be the extract solution or an appropriately diluted extract solution.

**3.10 analyte:** Element or constituent to be determined.

**3.11 stock solution:** Solution of appropriate composition containing the analyte, in a known high concentration.



**3.12 standard solution:** Solution containing the analyte, in known concentration suitable for the preparation of calibration solutions.

**3.13 set of calibration solutions:** Set of simple or synthetic calibration solutions having different analyte concentrations. The zero number is, in principle, the solution having zero concentration of the analyte.

**3.14 atomic absorption spectrometry (AAS):** Method for determining the concentration of chemical elements based on the measurement of the absorption of characteristic electromagnetic radiation by atoms in the vapour phase.

**3.15 optimum working range:** Range of concentrations of an analyte in solution over which the relationship between absorption and concentration is linear, or sufficiently closely approximates to linearity so that any divergence at the prescribed limit of concentration does not discernably affect any analytical determination.

**3.16 direct method of determination; analytical-curve technique:** Method consisting of inserting the measure obtained in the analytical function, and deducing from it the concentration of the analyte.

**3.17 analytical function; calibration function:** Function relating the value of the concentration to the characteristic value obtained from the set of calibration solutions. The graph of this function is called the "analytical curve (calibration graph)".

**3.18 bracketing technique:** Method consisting of bracketing the measured absorbance or intensity of the sample solution between two measurements made on calibration solutions of neighbouring concentrations.

**3.19 reference surface area;  $S_R$ :** Area intended to come into contact with foodstuffs.

#### 4 Principle

Ceramic articles are placed in contact with 40 ml/l acetic acid solution for 24 h at 22 °C to extract lead and/or cadmium, if present, from the surfaces of the articles.

The proportions of extracted lead and cadmium are determined by flame atomic absorption spectrometry (FAAS).

## 5 Reagents

During the determination, use only reagents of recognized analytical grade and only distilled water, or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696).

It is permissible to prepare proportionately greater quantities of test solution and analytical solutions than specified in the following clauses.

### 5.1 Acetic acid (CH<sub>3</sub>COOH), glacial, density $\rho = 1,05$ g/ml

Store this reagent in darkness or in dark coloured bottles.

### 5.2 Test solution

Acetic acid, 40 ml/l solution

Add to 500 ml of water by means of a graduated measuring cylinder (6.7) 40 ml  $\pm$  1 ml of glacial acetic acid (5.1) and make up to 1 l. Prepare the test solution freshly prior to use in sufficient quantity to enable the whole of any group of tests and analyses to be completed.

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### 5.3 Analytical stock solutions

#### 5.3.1 Stock lead solution (1 gram of lead per litre)

Prepare an analytical stock solution containing 1000 mg  $\pm$  1 mg of lead per litre in the test solution (5.2).

Alternatively, appropriate commercially available stock lead solutions may be used, provided that the concentrations of such solutions are known to an equivalent accuracy.

1 ml of this stock solution  $\hat{=}$  1 mg of lead.

#### 5.3.2 Stock cadmium solution (1 gram of cadmium per litre)

Prepare an analytical stock solution containing 1000 mg  $\pm$  1 mg of cadmium per litre in the test solution (5.2).

Alternatively, appropriate commercially available stock cadmium solutions may be used, provided that the concentrations of such solutions are known to an equivalent accuracy.

1 ml of this stock solution  $\hat{=}$  1 mg of cadmium.

## 5.4 Analytical standard solutions

### 5.4.1 *Standard lead solution (0,1 gram of lead per litre)*

By means of a one-mark pipette (6.4), transfer 10 ml of the stock lead solution (5.3.1) to a 100 ml one-mark volumetric flask (6.3), make up to the mark with the test solution (5.2) and mix well. Renew this solution every four weeks.

1 ml of this standard solution  $\hat{=}$  0,1 mg of lead.

From this solution, by dilution with test solution (5.2), freshly prepare a suitable range of calibration solutions (see 9.3.1), using the burette (6.5).

### 5.4.2 *Standard cadmium solution (0,01 gram of cadmium per litre)*

By means of a one-mark pipette (6.4), transfer 10 ml of the stock cadmium solution (5.3.2) into a 1000 ml one-mark volumetric flask (6.3), make up to the mark with the test solution (5.2) and mix well. Renew this solution every four weeks.

1 ml of this standard solution  $\hat{=}$  0,01 mg of cadmium.

From this solution, by dilution with test solution (5.2), freshly prepare a suitable range of calibration solutions (see 9.3.1), using the burette (6.5).

## 5.5 Paraffin wax, high melting point

NOTE: Suitable wax is specified in the European Pharmacopoeia.

5.6 Washing agent, commercially available non-acidic manual dishwashing detergent in common dilution.

5.7 Silicone sealant in a tube, or dispenser, enabling a ribbon of silicone sealant approximately 6 mm diameter to be formed.