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**Materiali in predmeti v stiku z živili - Silikatne površine - 2. del: Ugotavljanje izločanja svınca in kadmija iz nekeramičnih silikatnih površin**

Materials and articles in contact with foodstuffs - Silicate surfaces - Part 2: Determination of the release of lead and cadmium from silicate surfaces other than ceramic ware

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Silicatische Oberflächen - Teil 2: Bestimmung der Abgabe von Blei und Cadmium aus silicatischen Oberflächen ausgenommen keramischen Gegenständen

Matériaux et articles en contact avec les denrées alimentaires - Surfaces silicatées - Partie 2: Détermination de l'émission de plomb et de cadmium par les surfaces silicatées autre que les articles en céramique

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67.250      Materiali in predmeti v stiku z živili      Materials and articles in contact with foodstuffs

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English version

**Materials and articles in contact with foodstuffs -  
Silicate surfaces - Part 2: Determination of the  
release of lead and cadmium from silicate surfaces  
other than ceramic ware**

Matériaux et articles en contact avec les  
denrées alimentaires - Surfaces silicatées -  
Partie 2: Détermination de l'émission de plomb  
et de cadmium par les surfaces silicatées autre  
que les articles en céramique

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

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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.

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**CEN**

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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## 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 1388-1     Materials and articles in contact with foodstuffs - silicate surfaces  
Part 2: Determination of the release of lead and cadmium from 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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In accordance with 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.

## 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 7086 Part 1 was considered in this work.

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Part 1 of this European Standard specifies the method of test for the determination of released lead and cadmium from ceramic surfaces. Because existing national standards also have established that this method of test is applicable to glass, glass ceramic and vitreous enamel silicate surfaces this part of the European Standard has been prepared also to specify tests applicable to such silicate surfaces.

An existing national standard includes a test for surfaces with which the mouth is intended to come into contact, and this European standard also includes a method of test appropriate to such silicate surfaces.

For homogenous, i.e. undecorated or unglazed glassware, such as that made from soda-lime-silicate glass or borosilicate glass, and glass ceramic ware intended to come into contact with foodstuffs no raw materials containing lead and cadmium are used in the manufacture. An exception is crystal glass as defined in the EC-Directive for Crystal Glass (69/493 EEC). From soda-lime-silicate glass and borosilicate glass any release of lead and cadmium can only result from impurities and normally will be below the current detection limits of flame atomic absorption spectrometry (FAAS) for lead and cadmium. For uncoloured, undecorated or unglazed glass articles made from mass produced soda-lime-silicate glass or borosilicate glass, it is not essential to test according to this European Standard.

## 1 Scope

This European Standard specifies a reference method for determining the release of lead and cadmium from articles with silicate surfaces of glass, glass ceramic and vitreous enamel which are intended to come into contact with foodstuffs. This European Standard also applies to vitreous enamelled containers and to parts of equipment intended for industrial use with foodstuffs.

This European Standard specifies also a method of test for determining the release of lead and/or cadmium from a drinking rim; the drinking rim of ceramic articles can also be tested according to this method.

## 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.

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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 1042	Laboratory glassware - One-mark volumetric flasks
ISO 2723	Vitreous and porcelain enamels for sheet steel - Production of specimens for testing

ISO 2724	Vitreous and porcelain enamels for cast iron - Production of specimens for testing
ISO 3585	Borosilicate glass 3.3 - properties
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. Flatware also includes the test specimens for testing vitreous enamelled equipment, containers and water heaters.

**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.

**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.

**3.5 drinking rim:** 20 mm wide section of the external surface of a drinking vessel, measured downwards from the upper edge along the wall of the vessel.

**3.6 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.

**3.7 glass:** Inorganic material produced by the complete fusion of raw materials at high temperature into a homogeneous liquid which is then cooled to a rigid condition, essentially without crystallization.



**3.8 glass ceramic:** Inorganic material produced by the complete fusion of raw materials at high temperatures into a homogeneous liquid which is then cooled to a rigid condition with a certain degree of crystallization.

**3.9 borosilicate glass:** Glass containing a sufficient amount of boric oxide to influence its properties, in particular producing high chemical and thermal resistances.<sup>1)</sup>

NOTE: In borosilicate glass, lead and cadmium are present only in trace amounts as adventitious impurities. The release of these elements will be below the limits of detection of the method of test specified in this European Standard.

**3.10 soda-lime-silicate glass:** Glass in which the main constituents are normally sodium oxide, calcium oxide, and silica.<sup>1)</sup>

NOTE: In soda-lime-silicate glass, lead and cadmium are present only in trace amounts as adventitious impurities. The release of these elements will be below the limits of detection of the method of test specified in this European Standard.

**3.11 vitreous enamel:** Substantially vitreous inorganic coating bonded to metal by fusion at a temperature above 500 °C.

**3.12 vitreous enamelled ware:** Metallic articles coated with vitreous enamel.

**3.13 storage vessel; tank:** Article having a capacity of 3 litres or more.

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

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

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

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

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

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

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<sup>1)</sup> For glass and glass ceramic it is usual to state the content of chemical elements as oxides, irrespective of the actual existing type of compound.

**3.20 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.

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

**3.22 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.

**3.23 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.

**3.24 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)".

**3.25 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.

**3.26 reference surface area:** Area intended to come into contact with food stuffs.

## 4 Principle

Silicate surfaces 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 or test specimens. 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 ( $\text{CH}_3\text{COOH}$ )**, 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/1 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.

**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).

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Alternatively, appropriate commercially available 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 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.