International Standard 6486/1

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Ceramic ware in contact with food — Release of lead and cadmium — Part 1 : Method of test

Articles en céramique en contact avec les aliments — Émission de plomb et de cadmium — Partie 1 : Méthode d'essai

First edition — 1981-06-01

UDC 642.72 : 666.5 : 620.1 **Ref. No. ISO 6486/1-1981 (E)**

Descriptors : ceramics, earthenware, tableware, chemical analysis, determination of content, toxic substances, lead, cadmium, composition tolerances.

Price based on 4 pages

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6486/1 was developed by Technical Committee ISO/TC 166, *Ceramic ware, glassware and glass ceramic ware in contact with food*, and was circulated to the member bodies in June 1979. It results from the division into two parts of ISO/DIS 6486.

It has been approved by the member bodies of the following countries :

Austria	Italy	South Africa, Rep. of
Brazil	Japan	Thailand
Canada	Korea, Rep. of	United Kingdom
Czechoslovakia	Philippines	USA
Germany, F. R.	Poland	
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Denmark
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Ceramic ware in contact with food — Release of lead and cadmium — Part 1 : Method of test

0 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 food and beverages. As a secondary consideration, different requirements from country to country for the control of the release of toxic materials from the surfaces of ceramic ware present non-tariff barriers to international trade in these commodities. Accordingly, there is a need to establish internationally accepted methods of testing ceramic ware for lead and cadmium release, and to define permissible limits for the release of these toxic heavy metals.

An expert panel convened by the World Health Organization (WHO) met in Geneva, in June 1976, and recommended the adoption of sampling methods, testing procedures and limits for the release of toxic materials from ceramic ware. A further meeting was convened by WHO in November 1979. The method of test specified in this International Standard is based on the WHO recommendations[1, 2, 3, 4, 5].

1 Scope

This part of ISO 6486 specifies a method of test for the release of lead and cadmium by ceramic ware which may be used in contact with food, for example ceramic ware made of china, porcelain and earthenware, whether glazed or not, but excluding glass, glass ceramic and porcelain enamel articles.

2 Field of application

This part of ISO 6486 is applicable to ceramic ware which may be used for the preparation, serving and storage of food and beverages, excluding articles used in food manufacturing industries or those in which food is sold.

3 Reference

ISO 3585, *Glass plant, pipeline and fittings — Properties of borosilicate glass 3.3.*

4 Definitions

For the purpose of this part of ISO 6486, the following definitions apply.

4.1 ceramic ware : Ceramic articles which may be used in contact with foodstuffs, for example foodware made of china, porcelain and earthenware, whether glazed or not.

4.2 flatware : Ceramic ware having an internal depth not exceeding 25 mm, measured from the lowest point to the horizontal plane passing through the point of overflow.

4.3 hollow-ware : Ceramic ware having an internal depth greater than 25 mm, measured from the lowest point to the horizontal plane passing through the point of overflow.

Hollow-ware may be termed large or small according to its capacity as follows :

a) large hollow-ware : hollow-ware with a capacity of 1,1 litres or more;

b) small hollow-ware : hollow-ware with a capacity of less than 1,1 litres.

4.4 test solution : The solvent used in the test to extract lead and cadmium from the ceramic ware.

5 Principle

Extraction of lead and cadmium by an acetic acid solution from the ceramic ware surfaces that would normally come into contact with food. Determination by atomic absorption spectrometry of the amounts of lead and cadmium extracted.

6 Reagents

All reagents shall be of recognized analytical grade. Distilled water or water of equivalent purity shall be used throughout.

6.1 Acetic acid (CH_3COOH), glacial, ϱ = 1,05 g/ml.

Store this reagent in darkness.

6.2 Test solution : acetic acid, 4 % (V/V) solution.

Add 40 ml of the glacial acetic acid (6.1) to distilled water, and dilute to 1 000 ml. This solution shall be freshly prepared for use.

6.3 Analytical stock solutions

Prepare analytical stock solutions containing 1 000 mg of lead per litre and at least 500 mg of cadmium per litre in the acetic acid solution (6.2) or in a 2 % (V/V) nitric acid solution.

Alternatively, appropriate, commercially available, standardized lead and cadmium AAS solutions may be used.

7 Apparatus

7.1 Atomic absorption spectrometer, having a minimum sensitivity of 0,50 mg of lead per litre, and 0,05 mg of cadmium per litre, for 1 % absorption. It shall be operated in accordance with the manufacturer's instructions. A digital concentration reader (DCR) is optional but useful for rapid analysis.

7.2 Glassware, of borosilicate glass, as specified in ISO 3585.

8 Sampling

8.1 Priority

Carry out sampling of ceramic ware in the following order of priority :

a) large hollow-ware;

b) small hollow-ware;

c) flatware.

Articles having the highest surface area/volume ratio within each category should be given preference. Articles that are highly coloured or decorated on their food contact surfaces should be especially considered for sampling.

8.2 Sample size

It is desirable to develop a system of control that is regarded as appropriate to the circumstances. If available, six articles shall be tested. Each of the articles shall be identical in size, shape, colour and decoration.

8.3 Preparation and preservation of test samples

Samples of dinnerware shall be clean and free from grease or other matter likely to affect the test.

Briefly wash the specimens at a temperature of about 40 °C with a solution containing a non-acidic detergent. Rinse in tap water and then in distilled water or water of equivalent purity. Drain, and dry in either a drying oven or by means of a new filter paper so as to avoid any stains. Do not handle the surfaces to be tested after cleaning.

9 Procedure

9.1 Determination of filling volume

Place each specimen on a flat horizontal surface and fill it with water to 5 mm from overflowing, as measured along the surface of the specimen. Measure the volume (V) of the water to an accuracy of ± 2 %.

9.2 Determination of reference surface area for flatware

Invert the specimen on graph paper marked in millimetre squares and draw the contour round the rim. Calculate the area enclosed by the contour and record this as the reference surface area (A_R) in square decimetres.

9.3 Extraction

9.3.1 Extraction temperature

Carry out the extraction at a temperature of 22 ± 2 °C.

9.3.2 Leaching

Fill each specimen with the test solution (6.2) to 5 mm from overflowing, as measured along the surface of the specimen. Cover the specimen to prevent exposure of the surface under test to light. Leach for 24 h ± 10 min.

9.4 Sampling of the extraction solution for analysis

Prior to sampling the extraction solution to determine the lead and/or cadmium concentration, mix the extraction solution of each specimen by an appropriate method which avoids any loss of extraction solution or any abrasion of the surface being tested (for example, using a pipette, remove and allow the extraction solution to run back onto, and into, the specimen several times). Do not dilute the extraction solution (for example by rinsing the specimen). Transfer the extraction solution to a suitable storage container made of borosilicate glass. It is not necessary to transfer all the extraction solution.

Analyse the extraction solution as soon as possible as there is a risk of adsorption of lead or cadmium onto the walls of the storage container, particularly when the metals are present in low concentrations.

9.5 Calibration

Establish and carefully standardize instrument operating techniques so as to utilize maximum sensitivity, as determinations of lead concentrations as low as 0,50 mg/l, or cadmium concentrations as low as 0,05 mg/l, require the full potential of most instruments.

Prepare standard solutions by diluting the analytical stock solutions (6.3) with the test solution (6.2), and use the bracketing technique or construct a calibration curve having, for example, the absorbances of the standard solutions as abscissae, and the corresponding lead or cadmium contents, in milligrams per litre, as ordinates. Carry out a blank test on the reagents used for each set of determinations.

9.6 Determination of lead and cadmium

Determine the approximate amount of lead and cadmium in the extraction solution by use of the bracketing technique using the prepared standard solutions (see 9.5). This procedure may be used with any available read-out device. An averaging device, if available on the read-out device, will reduce the effects of background "noise" and improve both accuracy and precision.

If the lead content of the extraction solution is found to be higher than 20 mg/l, take a suitable aliquot portion and dilute it with the test solution (6.2) to reduce the concentration to less than 20 mg/l.

Alternatively, standard solutions of higher concentration may be used.

Similar considerations apply to the determination of cadmium.

Determine the lead and cadmium contents of the extraction solution by atomic absorption spectrometry using the procedure specified by the instrument manufacturer.

10 Expression of results

10.1 Bracketing technique

The lead or cadmium content, c_0, expressed in milligrams per litre of the extraction solution, is given by the formula

$$\frac{A_0 - A_1}{A_2 - A_1} \times (c_2 - c_1) + c_1$$

where

A_0 is the absorbance of the lead or cadmium in the extraction solution;

A_1 is the absorbance of the lead or cadmium in the lower bracketing solution;

A_2 is the absorbance of the lead or cadmium in the upper bracketing solution;

c_1 is the lead or cadmium content, in milligrams per litre, of the lower bracketing solution;

c_2 is the lead or cadmium content, in milligrams per litre, of the upper bracketing solution.

NOTE — If the extraction solution was diluted (see 9.6), an appropriate correction factor has to be used in the formula.

10.2 Calibration curve technique

Read the lead or cadmium content directly from the calibration curve or from the direct read-out.

10.3 Calculation of release of lead and cadmium from flatware

The lead or cadmium released per unit area from flatware, a_0, expressed in milligrams per square decimetre, is given by the formula

$$\frac{c_0 V}{A_R}$$

where

c_0 is the lead or cadmium content, expressed in milligrams per litre, of the extraction solution (10.1 or 10.2);

V is the filling volume, expressed in litres, of the specimen (9.1);

A_R is the reference surface area, expressed in square decimetres, of the specimen (9.2).

10.4 Reporting

For hollow-ware, report the result to the nearest 0,1 mg of lead per litre and to the nearest 0,01 mg of cadmium per litre.

For flatware, report the result to the nearest 0,1 mg of lead per square decimetre and to the nearest 0,01 mg of cadmium per square decimetre.

11 Test report

The test report shall include the following particulars :

a) a reference to this part of ISO 6486;

b) identification of the sample;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or regarded as optional.

Bibliography

[1] *Proceedings, International Conference on Ceramic Foodware Safety,* pp. 8-17, 1975, Lead Industries Association Inc., 292 Madison Avenue, New York, N.Y. 10017, USA.

[2] WHO Food Additives Series No. 4, 1972

[3] WHO Technical Report Series No. 505, 1972.

[4] WHO/Food Additives 77.44. *Ceramic Foodware Safety, Sampling, Analysis and Limits for release* (Report of a WHO Meeting, Geneva 8-10 June 1976).

[5] WHO/Food Additives HCS/79.7. *Ceramic Foodware Safety, Critical Review of Sampling, Analysis, and Limits for Lead and Cadmium Release* (Report of a WHO Meeting, Geneva 12-14 November 1979).

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