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Ceramic ware, glass-ceramic ware and glass dinnerware in contact with food — Release of lead and cadmium —

Part 1:
Test method

Articles en céramique, vaisselle en vitro-céramique et vaisselle plate en verre en contact avec les aliments — Émission de plomb et de cadmium —

Partie 1: Méthode d'essai

Reference number
ISO 6486-1:1999(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 6486 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6486-1 was prepared by Technical Committee ISO/TC 166, *Ceramic ware, glassware and glass ceramic ware in contact with food*.

This second edition cancels and replaces the first edition (ISO 6486-1:1981), which has been technically revised.

The current revision has updated the technical procedures and has brought the permissible limits for metal release in line with current regulatory limits in major markets and in harmony with as many regional or national standards as practical.

ISO 6486 consists of the following parts, under the general title *Ceramic ware, glass-ceramic ware and glass dinnerware in contact with food — Release of lead and cadmium*:

— *Part 1: Test method*

— *Part 2: Permissible limits*

Introduction

Lead- and cadmium-release from ceramic and glassware surfaces is an issue which requires effective means of control to ensure the protection of the population against possible hazards arising from the use of improperly formulated and/or processed ceramic, glass-ceramic and glass dinnerware 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 maintain internationally accepted methods of testing ware for lead- and cadmium-release, and to define permissible limits for the release of these toxic heavy metals.

The limits for lead and cadmium release specified in this part of ISO 6486 are not intended to be regarded as the maximum amount of these metals to which exposure can be considered safe. They are levels which are consistent with good manufacturing practice in the respective industries, harmonize regulatory levels in principal world markets and reflect a general objective of reducing overall exposure to these metals.

INTERNATIONAL STANDARD **ISO 6486-1:1999(E)**

Ceramic ware, glass-ceramic ware and glass dinnerware in contact with food — Release of lead and cadmium —

Part 1:
Test method

1 Scope

This part of ISO 6486 specifies a test method for the release of lead and cadmium from ceramic ware, glass-ceramic ware, and glass dinnerware intended to be used in contact with food, but excluding porcelain enamel articles.

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 6486. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 6486 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 385-2:1984, *Laboratory glassware — Burettes — Part 2: Burettes for which no waiting time is specified.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3585:1998, *Borosilicate glass 3.3 — Properties.*

ISO 3696:1987, *Water for analytical laboratory use — Specifications and test methods.*

3 Terms and definitions

For the purposes of this part of ISO 6486, the following terms and definitions apply.

3.1
atomic absorption spectrometry (AAS)
spectroanalytical method for qualitative determination and quantitative evaluation of element concentrations wherein the technique determines these concentrations by measuring the atomic absorption of free atoms

3.2
atomic absorption
absorption of electromagnetic radiation by free atoms in the gas phase wherein a line spectrum is obtained which is specific for the absorbing atoms

3.3
bracketing technique
analytical method consisting of bracketing the measured absorption or machine reading of the sample between two measurements made on calibration solutions of neighbouring concentrations within the optimum working range

3.4
calibration function
function relating atomic absorption instrument readings, either in absorption or in other machine units, to the concentration of lead or cadmium which generated the instrument reading

3.5
ceramic ware
ceramic articles which are intended to be used in contact with foodstuffs, e.g. foodware made of china, porcelain and earthenware, whether glazed or not

3.6
cooking ware
foodware, specifically intended to be heated in the course of preparation of food and drinks by conventional thermal methods and by microwaves

3.7
dinnerware
articles specially intended for the serving of food on the table, including plates, dishes and salad bowls, but excluding volumetric ware typically used for beverages, such as goblets and decanters

3.8
direct method of determination
analytical method consisting of inserting the measured absorption or machine reading into the calibration function and deducing the concentration of the analyte

3.9
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

3.10
extraction solution
acetic acid, 4 % (*V*/*V*), recovered after the extraction test and which is analysed for lead and cadmium concentration

3.11
flame atomic absorption spectrometry (FAAS)
atomic absorption spectrometry that uses a flame to create free atoms of the analyte in the gas phase

3.12
flatware
ceramic or glassware having an internal depth not exceeding 25 mm, measured from the lowest point to the horizontal plane passing through the point of overflow

3.13
foodware
articles which are intended to be used for the preparation, cooking, serving and storage of food or drinks

3.14
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 and temperature treated in such a way as to produce a mostly micro-crystalline body

3.15
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

NOTE The material may be clear, coloured, or opaque, depending on the level of colouring and opacifying agents used.

3.16
hollowware

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. Hollowware is subdivided into three categories based on volume:

- small: hollowware with a capacity $< 1,1$ l;
- large: hollowware with a capacity of $\geqslant 1,1$ l;
- storage: hollowware with a capacity of $\geqslant 3$ l;
- cups and mugs: small ceramic hollowware commonly used for consumption of beverages, e.g., coffee or tea at elevated temperature.

NOTE cups and mugs are vessels of approximately 240 ml capacity with a handle. Cups typically have curved sides whereas mugs have cylindrical sides.

3.17
optimum working range

range of concentrations of an analyte over which the relationship between absorption and concentration is practically linear

3.18
reference surface area

the area that is intended to come into contact with foodstuffs in normal use

3.19
test solution

the solvent used in the test to extract lead and cadmium from the article [acetic acid, 4 % (*V*/*V*)]

3.20
vitreous enameled ware

metallic articles coated with a vitreous inorganic coating bonded by fusion at temperatures above 500 °C

4 Principle

Silicate surfaces are placed in contact with 4 % (*V*/*V*) acetic acid solution for 24 h at (22 ± 2) °C to extract lead and/or cadmium, if present, from the surfaces of the articles or test specimens.

The amounts of extracted lead and cadmium are determined by flame atomic absorption spectrometry (FAAS). In routine tests other equivalent analysis methods may be used.

5 Reagents and materials

5.1 Reagents

All reagents shall be of recognized analytical grade. Distilled water or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696) shall be used throughout.

5.1.1 Acetic acid, (CH_3COOH), glacial, $\rho = 1{,}05$ g/ml.

5.1.2 Acetic acid test solution, 4 % (*V/V*) solution

Add 40 ml of acetic acid (5.1.1) to distilled water, and dilute to 1 l. This solution shall be freshly prepared for use. Proportionately greater quantities may be prepared.

5.1.3 Lead stock solution

Prepare analytical stock solutions containing 1 000 mg ± 1 mg of lead per litre in the test solution (5.1.2). Alternatively, an appropriate commercially available standardized lead AAS solutions may be used.

5.1.4 Cadmium stock solution

Prepare analytical stock solutions containing 1 000 mg ± 1 mg of cadmium per litre in the test solution (5.1.2). Alternatively, an appropriate commercially available standardized cadmium AAS solution may be used.

5.1.5 Lead standard solution

Dilute the lead stock solutions ten-fold with test solution (5.1.2) to produce a lead standard solution which is 100 mg/l Pb, or 0,1 g of lead per litre.

5.1.6 Cadmium standard solution

Dilute the cadmium stock solutions 100-fold with test solution (5.1.2) to produce a cadmium standard solution which is 10 mg/l Cd, or 0,01 g of cadmium per litre.

NOTE 1 Standard solutions may be kept in suitable, aged, tightly closed containers (i.e. polyethylene) for four weeks without loss of quality. New containers may be aged by filling with standard solution and allowing to stand for 24 h. The aging solution is discarded.

NOTE 2 Use one-mark glass pipettes or precision piston pipettes with a fixed stroke, typically 1 000 µl and 500 µl, and appropriate volumetric glassware (e.g. 500 ml to 2 000 ml) to prepare proper calibration solutions by dilution of the standard stock solutions (5.1.5 and 5.1.6) with test solution (5.1.2). Keep the solutions in suitable and aged containers. Renew these solutions every four weeks.

5.2 Materials and supplies

5.2.1 Paraffin wax, with a high melting point.

5.2.2 Washing agent, commercially available non-acidic manual dishwashing detergent in dilution recommended by a manufacturer.

5.2.3 Silicone sealant, capable of forming a ribbon of sealant approximately 6 mm in diameter. This sealant shall not leach acetic acid, cadmium or lead to the test solution (5.1.2).

6 Apparatus

6.1 Atomic absorption spectrometer

Atomic absorption spectrometer equipped with light sources (hollow cathode or electrodeless discharge lamps) specific for lead and cadmium, instrumental background correction, and a single slot (approximately 100 mm) or boiling burner head. Digital concentration readout may be used. Use air-acetylene flame and operating conditions recommended by the instrument manufacturer. Using these conditions, characteristic concentration (concentration that gives 0,004 4 absorbance) should be approximately (± 20 %) 0,2 mg/l for Pb measured at 217 nm. Characteristic concentration should be approximately (± 20 %) 0,02 mg/l for Cd measured at 228,8 nm.

NOTE Where appropriate, a wavelength of 283,3 nm may be used for the analytical confirmation of lead.

6.2 Accessories

6.2.1 Assorted glassware, as required, made of borosilicate glass as specified in ISO 3585.

6.2.2 Burette, of capacity 25 ml, graduated in divisions of 0,05 ml, complying with ISO 385-2, class B or better.

6.2.3 Covers for the articles under test, e.g. plates, watch-glasses, Petri dishes of various sizes. Covers must be opaque if a darkroom is not available.

6.2.4 One-mark pipettes, of capacities 10 ml and 100 ml, complying with ISO 648, class B or better. Other sizes as required.

6.2.5 One-mark volumetric flasks, of capacities 100 ml and 1 000 ml, complying with ISO 1042, class B or better. Other sizes as required.

6.2.6 Precision piston pipettes, with a fixed stroke, typically 1 000 µl and 500 µl.

6.2.7 Straight edge and depth gauge, calibrated in millimeters.

7 Sampling

7.1 Priority

When selecting samples from a mixed lot of foodware, 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.

7.2 Sample size

It is desirable to develop a system of sampling control that is appropriate to circumstances. In no case shall less than four items be measured. Each of the articles shall be identical in size, shape, colour and decoration.

7.3 Preparation and preservation of test samples

Samples of ware 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 using 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 wiping with a new piece of filter paper. Do not use any sample that shows residual staining. Do not handle the surfaces to be tested after cleaning.

If an area of the surface of the sample is not intended to come into contact with foodstuffs in normal use, other than the interior of any lid, cover this area after the initial washing and drying with a protective coating such as paraffin wax or silicone which will withstand the effect of the test solution and which will not release any detectable levels of lead or cadmium into the test solution.

8 Procedure

8.1 Determination of reference surface area for flatware

Place a specimen on a sheet of smooth paper and draw a contour around the rim. Determine the enclosed area by a suitable means. One recommended method is to cut out and weigh the enclosed area and to determine the area by comparison of the weight with the weight of a rectangular sheet of known area. Record this area, S_R, in square decimeters to two decimal places. For circular articles, the reference surface area may be calculated from the diameter of the article.

8.2 Preparation of articles which cannot be filled

Articles are normally filled to within 6 mm of overflowing as measured along the sloping side of flatware, or to within one mm of the rim as measured vertically for hollowware. Articles which cannot be filled in this manner to produce an acid depth at the deepest point of at least 5 mm are defined as non-fillable. Articles of this type may be tested by one of the following methods.

a) Standard articles may be fitted into a silicone rubber mold which forms a water-tight seal with the article and which encroaches no more than 6 mm from the rim and forms a depth of at least 5 mm but no more than 25 mm. Specimens prepared in this way are tested as fillable flatware articles.

b) A bead of silicone sealant may be formed around the edge of the article to permit filling of the article to a depth of at least 5 mm but no more than 25 mm. The bead shall encroach no more than 6 mm from the rim of the article. Specimens prepared in this way are tested as fillable flatware articles.

c) The article may be coated on all surfaces except the reference surface with melted paraffin wax and subsequently tested by immersion in test solution. Specimens prepared in this way are tested as non-fillable flatware articles.

8.3 Extraction

8.3.1 Extraction temperature

Conduct the extraction at a temperature of (22 ± 2) °C. When cadmium is to be determined, conduct the extraction in the dark.

8.3.2 Leaching

8.3.2.1 Fillable articles

Fill each specimen with test solution (5.1.2), to 1 mm of overflowing measured vertically for hollowware or 6 mm from overflowing as measured along the surface of flatware. For flatware determinations measure and record the volume of acetic acid, 4 %, used to fill the article. Cover the specimen. Leach for 24 h ± 30 min.

8.3.2.2 Non-fillable articles

These articles, which have been masked with paraffin wax according to 8.2.c), are placed in a suitable vessel such as borosilicate glass of suitable size and test solution (5.1.2) is added in sufficient quantity to completely cover the sample. Record the amount of acetic acid added to an accuracy of 2 %. Leach for 24 h ± 30 min.

8.3.3 Sampling of the extraction solution for analysis

Prior to sampling, mix the extraction solution by stirring or another appropriate method that avoids loss of the extraction solution or abrasion of the surface. Remove a sufficient amount of the extraction solution with pipette and transfer it to a suitable storage container.

Analyse the extraction solution as soon as possible since there is a risk of adsorption of lead or cadmium on to the walls of the storage container, particularly when Pb and Cd are present in low concentrations.

8.4 Drinking rim and other special tests

Cups may be tested by marking each of four units 20 mm below the rim on the outside. Each cup is placed inverted in a suitable laboratory glassware container with a diameter between 1,25 and 2 times that of the cup. Add sufficient 4 % acetic acid to the glassware container to fill to the 20 mm mark on the cup. Let stand for 24 h at (22 ± 2) °C (in the dark for cadmium determinations) and protect from excessive evaporation. Before sampling the leachate, add 4 % acetic acid to the glass container as necessary in order to re-establish the 20 mm level. Determine lead and cadmium by AAS and report the results as milligrams per article.

NOTE This is an optional procedure for evaluating drinking rims.

8.5 Calibration

Set up the atomic absorption spectrometer (6.1) according to the manufacturer's instructions using wavelengths of 217 nm for lead determination and 228,8 nm for cadmium determination with an appropriate correction for background absorption effects.

NOTE Where appropriate, a wavelength of 283,3 nm may be used for the analytical confirmation of lead.

Aspirate the zero member of the set of calibration solutions and adjust zero. Aspirate the set of calibration solutions, prepared by dilution of the standard solution with test solution (5.1.2) and prepare calibration curves over a linear range. Suggested ranges:

0,5 – 10,0 mg/l Pb

0,05 – 0,5 mg/l Cd

8.6 Determination of lead and cadmium

Set up the spectrometer as described previously. Aspirate distilled water and then 4 % acetic acid and verify that the absorbance is zero. Aspirate the extraction solution, interspersed with test solution (5.1.2) and record the absorbance values of the extraction solutions.

If the lead concentration of the extraction solution is found to be higher than 10 mg/l, dilute a suitable aliquot portion with test solution (5.1.2) to reduce the concentration to less than 10 mg/l.

Similar considerations apply to the determination of cadmium.

9 Expression of results

9.1 Bracketing technique

The lead or cadmium concentration, ρ_0, expressed in milligrams per litre of the extraction solution, is given by the formula

$$\rho_0 = \left[\left(\frac{A_0 - A_1}{A_2 - A_1} \right) (\rho_2 - \rho_1) + \rho_1 \right] d$$

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;

ρ_1 is the lead or cadmium concentration, in milligrams per litre, of the lower bracketing solution;

ρ_2 is the lead or cadmium concentration, in milligrams per litre, of the upper bracketing solution.

NOTE If the extraction solution is diluted, an appropriate correction factor, d, is used in the formula.

9.2 Calibration curve technique

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

9.3 Calculation of release of lead and cadmium from flatware

The lead or cadmium released per unit area from flatware, R_0, expressed in milligrams per square decimeter, is given by the formula

$$R_0 = \frac{\rho_0 \times V}{S_R}$$

where

ρ_0 is the lead or cadmium concentration, expressed in milligrams per litre, of the sample extract solution;

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

S_R is the reference surface area of the article, expressed in square decimeters.

For hollowware articles, 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 decimeter and to the nearest 0,01 mg of cadmium per square decimeter. Also report the concentration of lead and cadmium in the leach solution to the nearest 0,1 mg of lead per litre and to the nearest 0,01 mg of cadmium per litre.

10 Reproducibility and variability

Lead- and cadmium-release measurements from ceramic foodware are subject to analytical reproducibility errors and sampling variability. The material presented in this section are of scientific and technological interest but are not of normative or statutory value in the context of this part of of ISO 6486.

10.1 Reproducibility

Three types of determination errors occur in the analytical measurement of lead and cadmium concentrations. Each is listed in Table 1 with an approximate value for the standard deviation of each [4].

Table 1 — Sources of Variation in analytical determination of Pb and Cd

1	**Source of Variation**	**Standard deviation, Pb determination** mg/l	**Standard deviation, Cd determination** mg/l
2	Analysis, within laboratory	0,04	0,004
3	Analysis, between laboratories	0,06	0,007
4	Laboratory × sample Interaction	0,06	0,01
5	Reproducibility	0,094	0,012

The statistical interaction term, row 4 in Table 1, reflects the failure of the differences in sample analyses to be the same from laboratory to laboratory. A detailed discussion may be found in elementary statistical texts which address Analysis of Variance (ANOVA) methods. The reproducibility is the square root of the sum of the squares of the standard deviations from the three sources of variation.

10.2 Variability

Analytical reproducibility is quite good compared to the intrinsic variability of the extraction behaviour of glass and ceramic surfaces. This variability, termed sampling variability, is by far the greatest source of experimental error. Moore [5] has shown that the coefficient of variability for lead- and cadmium-release for large samples is typically 60 %. Thus, the true average lead-release value for a large population must be approximately 0,58 mg/l in order to avoid one of four test specimens from exceeding a 2 mg/l limit 1 in 10 000 times. Table 2 illustrates the effect of population mean and standard deviation values on the probability that 1 in 4 or 1 in 6 specimens will exceed a 2 mg/l limit value.

Table 2 — Probabilities of exceeding 2 mg/l limit

Population mean	**Population standard deviation**	**Probability of 1 in 4 > 2 mg/l**	**Probability of 1 in 6 > 2 mg/l**
0,4	0,24	< 0,000 01	< 0,000 01
0,8	0,48	0,138 26	0,200 05
1,2	0,72	0,758 36	0,881 22
0,4	0,12	< 0,000 01	< 0,000 01
0,8	0,24	0,000 02	0,000 04
1,2	0,36	0,325 68	0,446 27

11 Test report

The test report shall include the following information:

a) reference to this part of ISO 6486, i.e ISO 6486-1;

b) identification of the sample, including type, origin, and destination;

c) the surface area or the reference surface area and the filling volume or contact volume for non-fillable articles and test specimens;

d) the number of samples tested;

e) the test results, expressed as individual values for each specimen and the mean value for test sample groups. Test values for hollowware articles should be reported to the nearest 0,1 mg of lead per litre and to the nearest 0,01 mg of cadmium per litre. Test values for flatware should be reported to the nearest 0,1 mg/dm^2 of lead and to the nearest 0,01 mg/dm^2 of cadmium.

NOTE As supplementary information, the concentration of solutions from tests on flatware articles should also be included and reported to the nearest 0,1 g/l of lead and to the nearest 0,01 mg/l of cadmium.

f) any unusual features noted during the determination;

g) any optional tests, or tests not included in this part of ISO 6486.

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