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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

Vaisselle en céramique, vaisselle en vitrocéramique et vaisselle de table en verre en contact avec les aliments — Émission de plomb et de cadmium —

Partie 1: Méthode d'essai

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Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 166, *Ceramic ware, glassware and glass ceramic ware in contact with food*.

This third edition cancels and replaces the second edition (ISO 6486-1:1999), which has been technically revised. The main changes to the previous edition are as follows:

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

A list of all parts in the ISO 6486 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Release of potentially toxic metals, particularly lead and cadmium, 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, cooking, 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 metals 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 the release of potentially toxic metals.

The revision of this document was necessary to take into consideration recent developments in the application of the analytical technique inductively coupled plasma mass spectrometry (ICP-MS).

The test method is a combination of a leach procedure, which is the core of the document, and of the analytical method.

ICP-MS is the reference analytical method as it is generally considered to be the most accurate analytical method, although other methods have their own merits. Flame atomic absorption is kept as an alternative method. Other validated analytical methods, such as graphite furnace atomic absorption spectrometry (GFAAS) or inductively coupled optical emission spectrometry (ICP-OES), may also be used, considering the appropriate accuracy to the level of release of lead and cadmium to be measured.

The limits in ISO 6486-2 are set on the basis of a single extraction into the extraction solution. This document specifies that all repeat-use articles are tested three times with fresh extraction solution and the results of the third test reported for conformity with the permissible limits. It has been demonstrated that metal release into the third extraction is always less than the release into the first extraction. Therefore, data from a third extraction will show false conformity with the limits specified in ISO 6486-2. New limits that are appropriate to third extraction data are currently being agreed.

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

Part 1: Test method

WARNING — The use of this document may involve hazardous materials, operations and equipment. This document does not purport to address all the risks associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of national regulatory limitations prior to use.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document 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 vitreous and porcelain enamel articles (covered by ISO 4531).

This document 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 all articles used in food manufacturing industries or in which food is sold.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

ISO 3585, *Borosilicate glass 3.3 — Properties*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
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

3.2
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* (3.1) of free atoms

3.3
flame atomic absorption spectrometry
FAAS
atomic absorption spectrometry (3.2) that uses a flame to create free atoms of the analyte in the gas phase

3.4
graphite furnace atomic absorption spectrometry
GFAAS
atomic absorption spectrometry (3.2) involving electrothermal atomization in a graphite furnace

3.5
inductively coupled plasma mass spectrometry
ICP-MS
analytical method for qualitative determination and quantitative evaluation of element concentrations by measuring the ions produced by a radiofrequency inductively coupled plasma

Note 1 to entry: In the mass spectrometer the ions are separated and the elements identified according to their mass-to-charge ratio m/z, while the concentration of the elements is proportional to the numbers of ions.

3.6
inductively coupled optical emission spectrometry
ICP-OES
trace-level, elemental analysis technique that uses the emission spectra of a sample to identify and quantify the elements present

3.7
extraction solution
4 % per volume acetic acid solution recovered after the extraction test and which is analysed for lead and cadmium concentration

3.8
reference surface area
area that is intended to come into contact with foodstuffs in normal use

3.9
drinking rim
20 mm-wide section of the external surface of the item, measured downwards from the upper edge along the wall of the item

3.10
test solution
4 % per volume acetic acid solution used in the test to extract lead and cadmium from the article

3.11
ceramic ware
ceramic articles which are intended to be used in contact with foodstuffs, for example *foodware* (3.16) made of china, porcelain and earthenware, whether glazed or not

3.12
cup
small glass or *ceramic hollowware* (3.19) of approximately 240 ml capacity with a handle, commonly used for consumption of beverages, for example coffee or tea, at elevated temperature

Note 1 to entry: Cups typically have curved sides and are generally used with a saucer.

3.13
mug
small glass or *ceramic hollowware* (3.19) of approximately 240 ml capacity with a handle, commonly used for consumption of beverages, for example coffee or tea, at elevated temperature

Note 1 to entry: Mugs typically have cylindrical sides.

3.14
dinnerware
combination of ceramic ware, *glass ceramic ware* (3.18) and glass ware specially intended for the serving of food on the table, including plates, dishes and salad bowls, but excluding ware typically used for beverages

3.15
flatware
ceramic or *glassware* (3.17) which has an internal depth not exceeding 25 mm, measured from the lowest point to the horizontal plane passing through the point of overflow

3.16
foodware
articles which are intended to be used for the preparation, cooking, serving and storage of food or drinks

3.17
glassware
articles which are intended to be used in contact with foodstuffs and made of glass

Note 1 to entry: Glass is an 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. The material may be clear, coloured or opaque, depending on the level of colouring and opacifying agents used.

3.18
glass ceramic ware
articles which are intended to be used in contact with foodstuffs and made of glass ceramic

Note 1 to entry: Glass ceramic is an 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 microcrystalline body.

3.19
ceramic hollowware
ceramic ware which has an internal depth greater than 25 mm, measured from the lowest point to the horizontal plane passing through the point of overflow

Note 1 to entry: Hollowware is subdivided into three categories based on volume:

— small: hollowware with a capacity of < 1,1 l;

— large: hollowware with a capacity of ≥ 1,1 l;

— storage: hollowware with a capacity of ≥ 3 l.

4 Principles

Ceramic ware, glass ceramic ware and other silicate surfaces are placed in contact with test solution (5.1.3) for (24 ± 0,5) h at (22 ± 2) °C to extract lead and/or cadmium, if present, from the surfaces of the articles or test samples.

The amounts of extracted lead and cadmium are determined by an adequate analytical method. Inductively coupled plasma mass spectrometry (ICP-MS) is the reference analytical method as it is generally considered as the most accurate analytical method, although other methods have their own

merits. Flame atomic absorption spectrometry (FAAS) is kept as an alternative. Both methodologies are described in detail in Annexes A and B.

Other validated analytical methods, such as graphite furnace atomic absorption spectrometry (GFAAS) or inductively coupled optical emission spectrometry (ICP-OES), may also be used considering the appropriate accuracy to the level of release of lead and cadmium to be measured. In the case of ICP-OES, see the methodology described in Annex C for additional information.

5 Reagents and materials

5.1 Reagents

All reagents shall be of recognized analytical grade.

For the determination of lead and cadmium at trace and ultra trace level, the reagents shall be of adequate purity. The concentration of the analyte or interfering substances in the reagents and the water should be negligible compared with the lowest concentration to be determined.

5.1.1 Water grade 1, as specified in ISO 3696, for all sample preparations and dilutions.

5.1.2 Acetic acid, (CH_3COOH), glacial, ρ = 1,05 g/ml. Acetic acid purity better than 99 %, with Pb and Cd only at trace level.

5.1.3 Acetic acid test solution, with a volume fraction of 4 %.

Add 40 ml of acetic acid (5.1.2) to water (5.1.1) with a one-mark pipette (6.2.4) and dilute to 1 l in a one-mark volumetric flask (6.2.5). This solution shall be freshly prepared for use. Proportionately greater quantities may be prepared.

5.2 Materials and supplies

5.2.1 Paraffin wax, with a melting point in the range 50 °C to 57 °C.

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.

6 Apparatus

6.1 Analytical techniques

ICP-MS, FAAS or ICP-OES are described in Annexes A, B and C, respectively.

GFAAS is also a permitted option.

6.2 Accessories

6.2.1 Assorted laboratory ware, 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, conforming with ISO 385, class B or better.

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

6.2.4 One-mark pipettes, of capacities 10 ml and 100 ml, conforming 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, conforming with ISO 1042, class B or better. Other sizes as required.

6.2.6 Precision piston pipettes, typically 1 000 µl and 500 µl.

6.2.7 Straight edge and depth gauge, calibrated in millimetres.

7 Sampling

7.1 Priority

When selecting samples from a mixed lot of foodware, articles that have the highest surface area/volume ratio should be prioritized.

7.2 Sample size

At least four items shall 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 hand-hot temperature using tap water containing a non-acidic detergent. Rinse in tap water and then in water (5.1.1). Drain and dry either at a temperature of (40 ± 5) °C in 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.

8 Procedures

8.1 Determination of reference surface area for flatware

For circular articles, the reference surface area shall be calculated from the diameter of the article.

For others cases, 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 decimetres to two decimal places.

8.2 Preparation of articles which cannot be filled

Articles shall be filled to within 6 mm of overflowing as measured along the sloping side of flatware, or to within 1 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 mould 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 in the dark.

8.3.2 Leaching

8.3.2.1 Fillable articles

Fill each specimen with test solution (5.1.3) to within 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 the test solution (5.1.3) used to fill the article. Cover the specimen.

Leach for (24 ± 0,5) h depending on the case studied.

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 (5.1.3) is added in sufficient quantity to completely cover the sample. Record the amount of test solution (5.1.3) added to an accuracy of 2 %.

Leach for (24 ± 0,5) h depending on the case studied.

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 the amount of the extraction solution required by the considered analytical method with a 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.

8.3.4 Drinking rim test

Cups, mugs or other ceramic hollowware shall be tested by marking each of four units (20 ± 1) mm below the rim on the outside. Each item is placed inverted in a suitable laboratory glassware container with a diameter of between 1,25 times and 2 times that of the cup. Add sufficient test solution (5.1.3) to the glassware container to fill to the 20 mm mark on the cup. Leave to stand for (24 ± 0,5) h, depending on the case considered, at (22 ± 2) °C and protect from excessive evaporation. Before sampling the leachate, add test solution (5.1.3) to the glass container as necessary in order to re-establish the (20 ± 1) mm level. Determine lead and cadmium by the appropriate analytical methodology.

8.4 Articles used in repeated contact with foodstuffs

When an article is intended to come into repeated contact with foodstuffs, the release tests are carried out three times on the same test sample, using a fresh sample of the test solution (5.1.3) on each

