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Glass hollowware in contact with food — Release of lead and cadmium —

Part 1: Test method

Vaisselle creuse en verre en contact avec les aliments — Émission de plomb et de cadmium —
Partie 1: Méthode d'essai

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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.

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).

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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 7086-1:2000), which has been technically revised. The main changes to the previous edition are as follows:

— technical procedures have been updated;

— permissible limits for metal release have been 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 7086 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 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 glass hollowware 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 metals from the surfaces of glassware present non-tariff barriers to international trade in these commodities. Accordingly, there is a need to maintain internationally accepted methods of testing glassware for potentially toxic metal release.

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 as 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 7086-2 are set on the basis of a single extraction into the extraction solution. ISO 7086-2:2000, 8.5 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 7086-2. New limits that are appropriate to third extraction data are currently being agreed.

Glass hollowware 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 glass hollowware that is intended to be used in contact with food.

This document is applicable to glass hollowware intended for use in the preparation, cooking, serving and storage of food and beverages, excluding glass ceramic ware and glass flatware.

This document is also applicable to glass articles used for packaging in the food industry.

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
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 vessel, measured downwards from the upper edge along the wall of the vessel

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

3.11
foodware
articles which are intended to be used for the preparation, cooking, serving and storage of food or drinks

3.12
glassware
articles which are intended to be used in contact with foodstuff 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.

Note 2 to entry: The material may be clear, coloured or opaque, depending on the level of colouring and opacifying agents used.

3.13
flatware
glassware (3.12) that has an internal depth not exceeding 25 mm, measured from the lowest point to the horizontal plane passing through the point of overflow

3.14
glass hollowware
glassware (3.12) that 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: glass hollowware is subdivided into three categories based on volume:

— small: hollowware with a capacity of less than 600 ml;

— large: hollowware with a capacity of between 600 ml and 3 l;

— storage: hollowware with a capacity of 3 l or greater.

3.15
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.

4 Principles

Glassware 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 specimens.

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 Pb and Cd to be measured. In the case of ICP-OES, it is recommended that the methodology described in Annex C is applied.

For certain specific articles and applications as defined in ISO 7086-2, the (24 ± 0,5) h duration is replaced by (2 ± 0,1) h using the same test solution and temperature.

5 Reagents and materials

5.1 Reagents

All reagents shall be of recognized analytical grade.

For the determination of lead and cadmium at traces and ultra traces 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 determined.

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

5.1.2 Acetic acid, (CH3COOH), glacial, ρ = 1,05 g/ml.

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 56 °C to 58 °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 and inductively coupled plasma optic emission spectrometry (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 in an aqueous solution at (40 ± 5) °C at hand-hot temperature using tap water containing 1 ml/l of a non-acidic domestic 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 Extraction

8.1.1 Extraction temperature

Conduct the extraction at a temperature of (22 ± 2) °C in the dark.

8.1.2 Leaching

Fill each specimen with test solution to 1 mm from overflowing, as measured vertically. Cover the specimen. Leach for (24 ± 0,5) h, or (2 ± 0,1) h depending on the case studied.

8.1.3 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.

8.1.4 Drinking rim

The drinking rims of glass 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 between 1,25 and 2 times that of the item. Add sufficient test solution (5.1.3) to the glassware container to fill to the 20 mm mark on the item. Leave to stand for (24 ± 0,5) h, or (2 ± 0,1) h depending on the considered case, at (22 ± 2) °C (in the dark for cadmium determinations) and protect from excessive evaporation. Carefully cover the portion of the external surface of the article that is not to be tested with melted paraffin wax. Cover any handle present in the drinking rim region to be tested in the same way.

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.

It is permissible for the drinking rim to be cut off and tested separately.

Determine lead and cadmium by the appropriate analytical methodology and report the results.

8.2 Articles used in repeated contact with foodstuffs or beverages

When an article is intended to come into repeated contact with foodstuffs or beverages, 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 occasion. If the level of release conforms with the first migration, further testing is not necessary.

According to the specific category of article defined in ISO 7086-2, the conformity of the material is then checked on the basis of the concentration in the extraction solution in the third test.

Wash the article between each contact with water (5.1.1).

9 Analytical methods

9.1 General

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

GFAAS is also a permitted option.

9.2 Calculation of release of lead and cadmium from hollowware

The lead or cadmium released is obtained directly by the lead or cadmium concentrations of the sample extraction solution and expressed in micrograms per litre.

9.3 Calculation of release of lead and cadmium from drinking rim

The release of lead and cadmium per article from the drinking rim shall be calculated by multiplying the lead or cadmium concentrations by the volume of the test solution. This value shall be expressed in micrograms per article.

Another option is to calculate the release of lead and cadmium from the drinking rim per unit of surface by multiplying the lead or cadmium concentrations by the volume of the test solution, and dividing by the rim area. In this case, the value shall be expressed in micrograms per square decimetre.

10 Test report

The test report shall include the following information:

a) reference to this document, i.e. ISO 7086-1, including the year of publication;

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

c) the reference to the calculation used, as listed in Clause 9, with the following information, when appropriate: the surface area, the filling volume and the surface of the rim area for drinking rim testing;

d) the number of samples tested (minimum four samples);

e) the analytical method used, with the information required for the settings according to Annexes A to C;

