INTERNATIONAL STANDARD

ISO 7086-1

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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

Reference number
ISO 7086-1:2000(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 7086 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 7086-1 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 7086-1:1982), which has been technically revised.

ISO 7086 consists of the following parts, under the general title *Glass hollowware in contact with food — Release of lead and cadmium*:

— *Part 1: Test method*

— *Part 2: Permissible limits*

Introduction

Lead and cadmium release 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 glassware 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 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 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 7086 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.

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

Part 1:
Test method

1 Scope

This part of ISO 7086 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 part of ISO 7086 is applicable to glass hollowware intended for use in the preparation, cooking, serving and storage of food and beverages, excluding glass ceramic ware, glass flatware and all 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 7086. 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 7086 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 — Specification and test methods*.

3 Terms and definitions

For the purposes of this part of ISO 7086, the following terms and definitions apply.

3.1
atomic absorption spectrometry (AAS)
spectroanalytical method for qualitative determination and quantitative evaluation of element concentrations. 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 where 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
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

3.6
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

3.7
extraction solution

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

3.8
flame atomic absorption spectrometry (FAAS)

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

3.9
flatware

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

3.10
foodware

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

3.11
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

3.12
glassware

glass articles that are intended to be used in contact with foodstuffs

3.13
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.

3.14
hollowware

glassware having an internal depth greater than 25 mm, measured from the lowest point to the horizontal plane passing through the point of overflow

NOTE Hollowware is subdivided into three categories based on volume:

- small: hollowware with a capacity of less than 600 ml;
- large: hollowware with a capacity between 600 ml and 3 l;
- storage: hollowware with a capacity of 3 l or greater.

3.15
optimum working range
range of concentrations of an analyte over which the relationship between absorption and concentration is practically linear

3.16
reference surface area
the area that is intended to come into contact with foodstuffs in normal use

3.17
test solution
the solvent used in the test to extract lead and cadmium from the glassware (acetic acid, 4 % (V/V)

4 Principle

Silicate surfaces are placed in contact with 4 % (V/V) 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 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), 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/l ± 1 mg/l of lead 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/l ± 1 mg/l of cadmium 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 solution (5.1.3) ten-fold with test solution (5.1.2) to produce a lead standard solution which is 100 mg/l Pb, or 0,1 g/l of lead.

5.1.6 Cadmium standard solution, dilute the cadmium stock solution (5.1.4) 100-fold with test solution (5.1.2) to produce a cadmium standard solution which is 10 mg/l Cd, or 0,01 g/l of cadmium.

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

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 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.

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, watchglasses, Petri dishes of various sizes all of which shall 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 plus 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 plus 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 millimetres.

7 Sampling

7.1 Priority

When selecting samples from a mixed lot of ware, articles having the highest surface area/volume ratio within each category shall be given preference. Articles that are highly coloured or decorated on their food contact surfaces shall 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 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 wiping with a new piece of filter paper. Do not use any sample that shows residual staining. After cleaning do not handle the surfaces to be tested.

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 Extraction

8.1.1 Extraction temperature

Carry out the extraction at a temperature of (22 ± 2) °C. When cadmium is present, carry out the extraction in the dark.

8.1.2 Leaching

Fill each specimen with the test solution (5.1.2) to 1 mm from overflowing, as measured vertically. Cover the specimen. Leach for 24 h ± 30 min.

8.1.3 Sampling of the extraction solution for analysis

Prior to sampling, mix the extraction solution by stirring or other 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 onto the walls of the storage container, particularly when Pb and Cd are present in low concentrations.

8.2 Drinking rim and other special tests

NOTE This is an optional procedure for evaluating drinking rims.

The drinking rims of glass hollowware may be tested by marking each of four units 20 mm below the rim on the outside. Each glass is placed inverted in a suitable laboratory glassware container with a diameter between 1,25 and 2 times that of the glass. Add sufficient 4 % acetic acid to the glassware container to fill to the 20 mm mark on the test glass. 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 to re-establish the 20 mm level. Determine lead and cadmium by AAS and report the results as mg/article.

8.3 Calibration

Set up the atomic absorption spectrometer 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.

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 4 % acetic acid and prepare calibration curves over a linear range. Suggested ranges:

- 0,5 – 10 mg/l Pb
- 0,05 – 0,5 mg/l Cd

8.4 Determination of lead and cadmium

Set up the spectrometer as described previously. Aspirate distilled water and then 4 % acetic acid, and verify the absorbance is zero. Aspirate the extraction solution, interspersed with 4 % acetic acid 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 the 4 % acetic acid, 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 was diluted, an appropriate correction factor, d, is used in the formula.