International Standard ISO 8391/1

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

Articles de cuisson en céramique 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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8391/1 was prepared by Technical Committee ISO/TC 166, *Ceramic ware, glassware and glass ceramic ware in contact with food*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

0 Introduction

The problems of lead and cadmium release from cookware require effective means of control to ensure the protection of the population against a possible health hazard. This potential arises when improperly formulated, applied or fired glazes are applied to cookware. There is a particular concern for cookware because the normal conditions of use (heating acid foods for prolonged periods) are conducive to the extraction of soluble lead and cadmium into food.

As a secondary consideration, the varying standards for cookware that exist from country to country present non-tariff barriers to international trade. Accordingly, there is a need to establish internationally accepted methods of testing cookware for lead and cadmium release, and to define permissible limits for the extraction of these toxic metals.

An expert panel convened by the World Health Organization (WHO) met (with ISO participation) in Geneva, in November 1979, and recommended limits for the release of toxic materials from ceramic cookware for a proposed hot test method. Further, the meeting directed that the proposed method be further studied to determine its repeatability and reproducibility. The method specified in this part of ISO 8391 is based on the WHO recommendations and subsequent collaborative studies in which 14 laboratories throughout the USA, Europe and Japan participated.

ISO 8391 consists of the following parts :

Part 1 : Method of test.

Part 2 : Permissible limits.

1 Scope

This part of ISO 8391 specifies a method of test for the release of lead and cadmium by ceramic cookware intended for use in contact with food.

2 Field of application

This part of ISO 8391 is applicable to ceramic cookware intended to be used for the preparation of foods by heating.

3 References

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

ISO 6955, *Analytical spectroscopic methods — Flame emission, atomic absorption and atomic fluorescence — Vocabulary.*

4 Definitions

For the purpose of this part of ISO 8391, the following definitions apply.

4.1 ceramic cookware : Ceramic articles which are intended to be heated in the preparation of foodstuffs, for example china, porcelain and earthenware, but excludes glass, glass ceramic and porcelain enamel articles.

4.2 extraction solution : The solvent used in the test to extract lead and cadmium from cookware.

5 Principle

Extraction of lead and cadmium by a hot acetic acid solution from the cookware surfaces that would normally come in contact with food and its vapours during cooking.

6 Reagents

All reagents shall be of recognized analytical grade. Unless otherwise stated, distilled water or water of equivalent purity shall be used throughout.

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

Store this reagent in the dark.

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

Add 40 ml of the glacial acetic acid (6.1) to water and dilute to 1 000 ml.

This solution shall be freshly prepared for use.

6.3 Stock standard solutions.

Prepare stock standard 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 lead and cadmium AAS standard solutions may be used.

7 Apparatus

Laboratory glassware shall comply with the requirements of the appropriate International Standards, wherever such International Standards exist. It shall be made of borosilicate glass, as specified in ISO 3585.

Usual laboratory apparatus, and in particular

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) attachment is optional, but is useful for rapid analysis.

8 Sampling

8.1 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.

8.2 Preparation and preservation of test samples

Samples of cookware 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 Filling volume

Determine the effective volume of the sample by measuring the volume of water necessary to fill each vessel to the rim. Fill each vessel (cleaned as in 8.3) to two-thirds of its effective volume with water. Then cover the vessels with their own lids. If the cookware vessels under test do not have lids, cover them with flat, opaque, unleaded glass (or other suitable cover) to prevent evaporation and to prevent the surface under test from being exposed to light.

9.2 Extraction

Place each vessel on a hotplate and heat the water to a simmer (slow boil) at which time add enough glacial acid (6.1) to produce a 4 % (V/V) acetic acid solution.

The volume, V_a, of acetic acid required may be calculated from the equation

$$V_a = 0{,}041\ V_w$$

where V_w is the volume of water in the cookware vessel.

For vessels possessing their own heating element, the temperature shall be maintained at simmer (slow boil) using that vessel's heating element. In the event that the heating element produces vigorous boiling, a Variac[1] (or similar device) may be used to control the temperature to a simmer. Should the heating element not produce a temperature high enough to simmer the solvent, then the highest temperature attainable by the heating element shall be employed. Maintain slow boil or maximum heat attainable, as appropriate, for 2 h commencing from the time of addition of acetic acid.

If during the 2 h heating period a loss of solvent occurs (as for example with teapots), replace the loss with preheated acetic acid solution (6.2) to maintain the level of solution at two-thirds of the vessel's effective volume. At the end of the 2 h heating period, remove the heat source promptly.

9.3 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 on to, 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.

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

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[1] This trade name for a commercially available product is given for the convenience of the user of this part of ISO 8391 and does not constitute an endorsement of the product by ISO.

Analyse the extraction solution as soon as possible after it attains room temperature as there is a risk of adsorption of lead or cadmium on to the walls of the storage container, particularly when the metals are present in low concentrations.

9.4 Calibration and determination

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 calibration solutions by diluting the stock standard solutions (6.3) with acetic acid (6.2), and use the bracketing technique or construct a calibration graph having, for example, the absorbances of the calibration 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. The blank test solution may be used as a zero number for the calibration if the calibration graph technique is used.

Alternatively, calibration 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 (see ISO 6955, "Determination by the 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, expressed in milligrams per litre, of the lower bracketing solution;

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

NOTE — If the extraction was diluted (see 9.5), an appropriate correction factor has to be used in the formula.

10.2 Calibration graph technique

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

10.3 Reporting

Report the results to the nearest 0,1 mg of lead per litre and to the nearest 0,01 mg of cadmium per litre.

11 Precision

Table [1]

Range or level (mg/l)		Repeatability, r	Reproducibility, R
Lead	$0,48 \leqslant c_{Pb} \leqslant 1,93$	0,69	1,63
Cadmium	$0 < c_{Cd} \leqslant 0,10$	0,01	0,01

[1] The precision data were obtained from a collaborative study conducted in 1981 by the US Food and Drug Administration. The study involved 14 laboratories in the USA, Puerto Rico, Japan, Germany, F.R., Israel, United Kingdom, Ireland, Canada and the Netherlands. The test specimens were found to contain a mean level of 1,0 mg/l lead. No cadmium was present.

11.1 Repeatability

The difference between two single results found on identical test material by one analyst using the same apparatus within a short time interval will exceed the repeatability given in the table on average not more than once in 20 cases in the normal and correct operation of the method.

11.2 Reproducibility

The difference between two single and independent results found by two operators working in different laboratories on identical test material will exceed the reproducibility given in the table on average not more than once in 20 cases in the normal and correct operation of the method.

12 Test report

The test report shall include the following information :

a) a reference to this part of ISO 8391;

b) identification of the sample;

c) the results and the form in which these are expressed;

d) any unusual features noted during the determination;

e) any operation not included in this part of ISO 8391, or regarded as optional.

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