



Designation: C 1034 – 85 (Reapproved 1995)

Standard Test Method for Lead and Cadmium Extracted From Glazed Ceramic Cookware¹

This standard is issued under the fixed designation C 1034; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the precise determination of lead and cadmium extracted by acetic acid from glazed ceramic surfaces under boiling conditions. The procedure of extraction may be expected to accelerate the release of lead and cadmium from the glaze and to serve, therefore, as a severe test that simulates the most severe conditions of usage of such ceramic cookware. This method is specific for lead and cadmium.

1.2 This test method is applicable to ceramic cookware intended for use in contact with food, for example cookware made of china, crockery, porcelain, and earthenware.

1.3 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 242 Terminology of Ceramic Whitewares and Related Products²

D 1193 Specification for Reagent Water³

E 438 Specification for Glasses in Laboratory Apparatus⁴

E 694 Specification for Volumetric Ware⁴

2.2 ISO Standard:

3585 Glass Plate Pipeline and Fittings—Properties of Borosilicate Glass 3.3⁵

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *cookware*—ceramic articles including those intended to be heated in the preparation of foodstuffs, for example, china, crockery, porcelain, and earthenware; excluding items not specifically designed for cooking, such as cups, plates, and

soup bowls, which may be occasionally used to warm foods in a microwave oven.

3.2 *Definitions*—For definitions of terms used in this test method, see Terminology C 242.

4. Summary of Method

4.1 The lead and cadmium extracted from cookware, by boiling acetic acid after 2 h of leaching, are measured by atomic absorption spectrophotometry using specific hollow cathode lamps for lead and cadmium respectively.

5. Significance and Use

5.1 The consequences of lead and cadmium release from cookware require effective means of control to ensure the protection of the population against a possible health hazard.⁶ This potential arises with improperly formulated, applied and fired glazes and decorations. There is a particular concern for cookware because the normal conditions of use (heating acid foods for prolonged periods) are conducive to extracting the soluble lead and cadmium into the food. Therefore, this test method deals specifically with ceramic cookware intended to be used for the preparation of foods by heating.

6. Interferences

6.1 Since specific hollow cathode lamps for lead and cadmium are used, there are no interferences.

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer*, equipped with a 4-in. (102-mm) single slot or Belling burner head and digital concentration readout attachment (DCR) if available.⁷ This instrument should have a sensitivity of about 0.5 ppm of lead for 1 % absorption and a sensitivity of about 0.05 ppm of cadmium for 1 % absorption. Use the operating conditions as specified in the instrument manufacturer's analytical methods manual.

¹ This test method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Fundamental Properties.

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² *Annual Book of ASTM Standards*, Vol 15.02.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁶ *Proceedings, International Conference on Ceramic Foodware Safety*, Lead Industries, Inc., 1975, pp. 8–17.

WHO Food Additives Series, No. 4, World Health Organization, 1972.

“Ceramic Foodware Safety, Sampling, Analysis and Results for Release,” Report of a WHO Meeting, Geneva, Switzerland June 8–10, 1976, *WHO/Food Additives 77.44* World Health Organization, 1977.

⁷ Perkin-Elmer models 303 and 403 and Jarrell-Ash model 82-546 have been found suitable for this determination.

NOTE 1—1 ppm = 1 mg/L

7.2 *Hollow-Cathode Lead Lamp*, set at 283.3 or 217.0 nm.

7.3 *Hollow-Cathode Cadmium Lamp*, set at 228.8 nm.

7.4 *Glassware*, borosilicate glass, as specified in Specifications E 438 and E 694 and ISO 3585 shall be used to make reagents and solutions.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. It is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁸ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean distilled water, deionized water or water of equivalent quality meeting the requirements for one of the types of reagent water covered by Specification D 1193.

8.3 *Simulating Solvent*—The simulating solvent employed in the extraction of lead and cadmium shall be 4 % acetic acid (by volume). The solution is prepared by adding sufficient glacial acetic acid to water (preheated in the test vessel, with a hot plate or self-contained heating unit, to a simmer), to make the solution 4 % acetic acid. The volume of acetic acid required is calculated as follows:

$$0.042 (V_w) = V_a \quad (1)$$

where:

V_w = volume of water in the cookware vessel, mL and

V_a = volume of glacial acetic acid to be added mL.

Only glacial acetic acid that has been stored in darkness to avoid degradation should be used:

8.4 *Analytical Stock Solution:*

8.4.1 *Lead* (1000 mg/L)—Dissolve 1.598 g Pb (NO₃)₂ in distilled or deionized water. Add 40 mL glacial acetic acid and dilute to 1 L with distilled or deionized water.

8.4.2 *Cadmium* (1000 mg/L)—Dissolve 1.8546 g of anhydrous cadmium sulfate in approximately 500 mL of 1 % HCl and dilute to 1 L with 1 % HCl. Prepare 1 % HCl by mixing 1 part of concentrated hydrochloric acid (HCl, sp gr 1.19) with 37 parts of distilled water.

8.4.3 Alternatively, appropriate, commercially available, standardized lead and cadmium ASS solutions may be used.

9. Sampling

9.1 *Preference*—Preference should be given to items that have the highest surface area/volume ratio and to those that have decorated or cadmium red colored food contact surfaces (including the interior of the lid).

9.2 *Sample Size*—Where feasible test six pieces. Each of the pieces should be identical in size, shape, and color.

10. Procedure

10.1 *Preparation and Preservation of Test Samples*—Samples of cookware must be free of grease or other matter likely to prevent contact between the vessel's surface and the simulating solvent. Wash the specimen with a mild liquid detergent solution at a temperature of about 40°C. Rinse with distilled, deionized water or water of equivalent quality. Then drain the vessel and dry it either in a drying oven or with clean filter paper. Store the sample in a clean air hood or other clean environment to prevent contamination of the surface by airborne particles.

10.2 *Filling, Temperature, and Duration:*

10.2.1 Determine the effective volume of the specimen by measuring the volume of water necessary to fill it to the rim. Fill the vessel (cleaned as in 10.1) to two thirds its effective volume with distilled or deionized water or water of equivalent quality noting the source of the water used. Then cover the vessel with its own cover, if available, or else with a flat, opaque piece of borosilicate glass.

10.2.2 Place the vessel containing the known volume of water on a hot plate and heat to a simmer (slow boil) at which time add enough glacial acetic acid to produce 4 % acetic acid solution (by volume as in 8.3). For vessels possessing their own heating element (as for example a self-contained crock pot), maintain the temperature at simmer (slow boil) using that vessel's heating element. In the event that the heating element produces vigorous boiling, use a Variac (*R*) (or similar device) use to control the temperature (to a simmer). Should the heating element on maximum power setting not produce a temperature high enough to simmer the solvent, then use the highest temperature attainable by the heating element. Maintain slow boil or maximum heat attainable, as appropriate, for 2 h beginning from the time of adding the acetic acid.

10.2.3 If during the 2-h heating period a loss of simulating solvent occurs (as for example with teapots), replace the loss with 4 % acetic acid solution to maintain the level of solution at two thirds the vessel's effective volume. If desired, use a scored borosilicate glass rod to measure liquid level. At the end of the 2-h heating period, promptly remove the heat source and cool the liquid to room temperature.

10.3 *Sample of the Test Solution for Analysis:*

10.3.1 Prior to analyzing the test solution to determine the lead or cadmium concentration, or both, mix the contents of the ceramic article to be tested by an appropriate method to obviate any loss of solution and any abrasion of the surface of the article to be tested.

10.3.2 Make the analysis of the solution as soon as possible. This is because of a risk of significant adsorption of lead or cadmium onto the walls of the storage container.

10.4 *Preparation of Standards:*

10.4.1 *Lead Standards*—Dilute lead analytic stock solution (see 8.4.1) with 4 % acetic acid (see 8.3) to obtain working standards having final concentrations of 0.0, 1, 3, 5, 10, 15, and 20 ppm Pb.

10.4.2 *Cadmium Standards*—Dilute cadmium stock solution (see 8.4.2) with acetic acid (see 8.3) to obtain working

⁸ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.