Designation: C 738 – 94 (Reapproved 2006)

Standard Test Method for Lead and Cadmium Extracted from Glazed Ceramic Surfaces[1]

This standard is issued under the fixed designation C 738; 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. The procedure of extraction may be expected to accelerate the release of lead from the glaze and to serve, therefore, as a severe test that is unlikely to be matched under the actual conditions of usage of such ceramic ware. This test method is specific for lead and cadmium.

1.2 The values stated in SI (metric) units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of 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. Summary of Test Method

2.1 Lead and cadmium are extracted from the test article by leaching with 4 % acetic acid for 24 h at 20 to 24°C (68 to 75°F) and are measured by flame atomic absorption spectroscopy.

3. Interferences

3.1 There are no interferences when instrumental background correction and light sources specific for lead and cadmium are used.

4. Apparatus

4.1 *Atomic Absorption Spectrometer* equipped with light sources (hollow cathode or electrodeless discharge lamps) specific for lead and cadmium, instrumental background correction, and a 4-in. (102-mm) single slot or Boling burner head. Digital concentration readout may be used. Use air-acetylene flame, instrumental background correction, and operating conditions recommended by instrument manufacturer. Using these conditions, characteristic concentration (concentration that gives 0.0044 absorbance) should be approximately (±20 %) 0.2 and 0.45 ppm for Pb measured at 217.0 and 283.3 nm, respectively. Characteristic concentration should be approximately (±20 %) 0.02 ppm for Cd.

NOTE 1—1 ppm = 1 µg/mL.

4.2 *Lead Lamp*, set at 283.3 or 217.0 nm.

4.3 *Cadmium Lamp*, set at 228.8 nm.

4.4 *Glassware* of chemically resistant borosilicate glass, to make reagents and solutions. Clean by rinsing with dilute nitric acid (10 % by volume) followed by copious quantities of water.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, 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.[2] 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water.

5.3 *Acetic Acid* (*4 % by Volume*)—Mix 1 volume of glacial acetic acid with 24 volumes of water.

5.4 *Detergent Wash*—Use detergent designed for washing household dishes by hand. Mix with lukewarm tap water according to product instructions.

5.5 *Lead Nitrate Solution* (*1000-ppm Pb*)—Dissolve 1.598 g of lead nitrate ($Pb(NO_3)_2$) in 4 % acetic acid and dilute to 1 L with 4 % acetic acid. Commercially available standard lead solutions may also be used.

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[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Methods for Whitewares and Environmental Concerns. This test method was developed jointly by the AOAC and ASTM, and adopted official final action by the Association of Official Analytical Chemists (method 973.32 *AOAC Official Methods of Analysis* (1990) 15th ed, AOAC International, Arlington, VA.

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[2] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

5.6 *Hydrochloric Acid* (*1 % by weight*)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 37 volumes of water.

5.7 *Cadmium Solution* (*1000-ppm Cd*)—Dissolve 0.9273 g of anhydrous cadmium sulfate in approximately 250 mL of 1 % HCl (see 5.6) and dilute to 500 mL with 1 % HCl. Commercially available standard cadmium solutions may also be used.

6. Procedure

NOTE 2—Take a method control through entire procedure. Use a laboratory beaker with dimensions similar to ware being tested.

6.1 *Preparation of Sample*—Take, at random, six identical units and the method control vessel and clean with detergent wash. Then rinse with tap water followed by distilled water. Dry, and fill each unit with 4 % acetic acid to within approximately 6 to 7 mm (¼ in.) of overflowing. (Distance shall be measured along the surface of the item tested, not in the vertical direction.) Record the volume of acid required for each unit in the sample (Note 3). Cover each unit with fully opaque glass plate (so that extraction is carried out in total darkness) o prevent evaporation of solution, avoiding contact between cover and surface of leaching solution. (If opaque glass is not available, cover glass with aluminum foil or other material to prevent exposure to light.) Let stand for 24 h at room temperature (20 to 24°C (68 to 75°F)).

NOTE 3—If the sample unit is extremely shallow, or if it has an irregular brim, the analyst should be aware of evaporation of leaching solution. If such a loss is anticipated, record the headspace upon filling the vessel to 6 to 7 mm (¼ in.) of the brim. Adjust to the same headspace with 4 % acetic acid after the 24-h leaching. Stir the solution and proceed with the determination.

6.2 *Preparation of Standards*:

6.2.1 *Lead Standards*—Dilute lead nitrate solution (see 5.5) with acetic acid (see 5.3) to obtain working standards having final concentrations of 0.0-, 1-, 2-, 3-, 5-, and 10-ppm Pb.

6.2.2 *Cadmium Standards*—Dilute cadmium stock solution (see 5.7) with acetic acid (5.3) to obtain working standards having final concentrations of 0.0-, 0.1-, 0.2-, 0.3-, 0.5-, and 1.0-ppm Cd.

6.3 *Determination of Lead by Atomic Absorption*—Stir the leaching solution and remove a portion by pipetting into a clean flask. Use lead lamp (4.2) and concomitantly measure absorbance of lead working standards (6.2.1) and leach solutions. Dilute with 4 % acetic acid if leach solutions contain over 10-ppm Pb. Concentrate leach solutions containing less than 1-ppm Pb by accurately transferring a minimum of 50.0 mL of solution to a 250-mL beaker and evaporating almost to dryness on a steam bath. Add 1 mL of HCl, then evaporate to dryness. Dissolve the residue in 4 % acetic acid by adding exactly 0.1 volume of solution taken for concentration, cover with watch glass, and swirl to complete dissolution. Calculate lead concentration (ppm Pb) of leach solution by comparison to standard curve.

TABLE 1 Mean Value Required in a Large Lot for Failure Rate 1/10 000 with Coefficient of Variation 60 %

Limit (ppm)	Mean of 6	Worst of 6 (Mean of 1)
7	3.21	1.45
5	2.29	1.03
2.5	1.15	0.52
0.7	0.32	0.14
0.5	0.23	0.10
0.25	0.11	0.05

6.4 *Determination of Cadmium by Atomic Absorption*—Proceed as in 6.3 using the cadmium lamp (4.3) and standards (6.2.2). Dilute with 4 % acetic acid if leach solutions contain over 1-ppm Cd. Concentrate leach solutions containing less than 0.1-ppm Cd as in 6.3.

7. Report

7.1 Report the type of units tested, the volume of acid used, and the lead and cadmium leached in parts per million for each unit tested.

NOTE 4—As indicated in Section 1, this procedure covers the extraction and measurement of lead and cadmium from ceramic surfaces. It is general in that it does not specify specific sample unit types. For special end uses, as for example, process control or interlaboratory testing, a specific size and type of sample unit should be used.

8. Precision and Bias

8.1 *Precision*—In an analysis of variance study from eight laboratories, the standard deviation between laboratories was 0.06 mg/L for lead and 0.007 mg/L for cadmium. The within laboratory precision had a standard deviation of 0.04 mg/L for lead and 0.004 mg/L for cadmium. The standard deviation for interaction between laboratories and samples is 0.06 mg/L for lead and 0.010 mg/L for cadmium. Reproducibility is defined as the square root of the sum of the three component variances. The reproducibilities were 0.10 mg/L for lead and 0.013 mg/L for cadmium.

8.2 *Bias*—The bias of this test method is further limited by the ability to obtain representative samples of the statistical universe being sampled. An analysis of large populations (100 to 500) has shown that the lead and cadmium release data conformed to a Pearson III distribution with a coefficient of variation between 30 and 140 %, typically 60 %.[3] Table 1 shows the mean value required in a large lot so that there will be no more than 1 failure in 10 000 for a limit stated in average of six units and for a limit stated for the worst of six units, for a coefficient of variation of 60 %.

9. Keywords

9.1 cadmium; glazed ceramic surfaces; lead

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[3] Moore, C. F., *Transactions*, Journal of British Ceramic Society, Vol 76 (3), 1977, pp. 52–57.

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