Designation: C895 – 87 (Reapproved 2014)

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

This standard is issued under the fixed designation C895; 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 tile that are intended for use in areas of food preparation. 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 tile. This test method is specific for lead and cadmium.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

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. Referenced Documents

2.1 *ASTM Standards:*[2]

C738 Test Method for Lead and Cadmium Extracted from Glazed Ceramic Surfaces

C1034 Test Method for Lead and Cadmium Extracted From Glazed Ceramic Cookware (Withdrawn 2001)[3]

C1035 Specification for Lead and Cadmium Extracted from Glazed Ceramic Cookware (Withdrawn 2001)[3]

3. Summary of Test Method

3.1 The lead and cadmium extracted from the article under test, by acetic acid at 20 to 24°C (68 to 75°F) after 24 h of leaching, are measured by atomic absorption spectrophotometry using specific hollow-cathode lamps for lead and cadmium, respectively.

4. Significance and Use

4.1 There are several test methods available to measure the lead and cadmium release from dinnerware and cookware (see Test Methods C738 and C1034 and Specification C1035). These standards are used as a control to ensure the protection of the population against a possible health hazard.[4] This potential hazard arises with improperly formulated, applied, fired glazes and decorations. This test method deals specifically with ceramic tile that are intended to come in contact with food during its preparation (for example, counter top tile).

5. Interferences

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

6. Apparatus

6.1 *Atomic Absorption Spectrophotometer,* equipped with a 4-in. (102-mm) single slot or Boling burner head and digital concentration readout attachment (DCR) if available. This instrument should have a sensitivity of about 0.5 ppm of lead for 1 % absorption and a sensitivity of about 0.25 ppm of cadmium for 1 % absorption. Use the operating conditions as specified in the instrument manufacturer's analytical methods manual.

NOTE 1—1 ppm = 1 mg/L or one part per million is one milligram per litre.

6.2 *Hollow-Cathode Lead Lamp,* with wavelength set at 283.3 or 217.0 nm.

6.3 *Hollow-Cathode Cadmium Lamp,* with wavelength set at 228.8 nm.

6.4 *Glassware* of chemically resistant borosilicate glass to make reagents and solutions.

6.5 *Test Cell*—Chemically resistant borosilicate glass cylinder to contain the leaching solution. An open-ended cell approximately 80 mm in length and 60 mm in internal diameter has proven suitable.

[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Productsand is the direct responsibility of Subcommittee C21.06 on Ceramic Tile.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] "Lead Industries, Inc.," *Proceedings, International Conference on Ceramic Foodware Safety*, 1975, pp. 8–17.

7. Reagents

7.1 *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.[5] 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.

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

7.3 *Acetic Acid (4 % by Volume)*—Mix 1 volume of glacial acetic acid with 24 volumes of water. Run a reagent blank each time a 4 % acetic acid solution is prepared.

7.4 *Detergent Rinse*—Add 15 g of suitable alkaline detergent to 1 gal (3.79 L) of lukewarm tap water.

7.5 *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.

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

7.7 *Cadmium Solution (1000-ppm Cd)*—Dissolve 0.9273 g of anhydrous cadmium sulfate in approximately 250 mL of 1 % HCl (see 7.6), and dilute to 500 mL with 1 % HCl. Commercially available standard cadmium solutions may also be used.

8. Procedure

8.1 *Preparation of Sample*—Take, at random, six identical tiles and cleanse the surface of each with a detergent rinse. Then rinse with tap water, followed by distilled water, and dry. Using a suitable silicone rubber sealant free of lead and cadmium, seal the test cell to the glazed surface. Fill each cell with sufficient 4 % acetic acid to achieve a ratio of 25 mL of solution to each 1 $in.^2$ (6.45 cm^2) of exposed glaze surface. Record the volume of acid used in each cell. If the tile being tested has a surface dimension less than 60 mm (smaller than the diameter of the test cell), a suitable number of tiles shall be mounted and grouted[6] to provide the required test area. Mounting, grouting, and grout cleanup should precede the detergent rinse. Each mounted set constitutes one test sample. Cover each test with a glass plate to prevent evaporation of solution, avoiding contact between cover and surface of leaching solution. The extraction shall be run in total darkness, and the test array must be covered with an opaque cloth or foil to prevent exposure to light. Let stand for 24 h at 20 to 24°C (68 to 74°F).

8.2 *Preparation of Standards:*

8.2.1 *Lead Standards*—Dilute lead nitrate solution (see 7.5) with acetic acid (see 7.3) to obtain working standards having final concentrations of 0, 5, 10, 15, and 20 ppm of Pb.

8.2.2 *Cadmium Standards*—Dilute cadmium stock solution (see 7.7) with acetic acid (see 7.3) to obtain working standards having final concentrations of 0.0, 0.3, 0.5, 1, 1.5, and 2.0 ppm of Cd.

8.3 *Determination of Lead by Atomic Absorption*—Stir the sample (leaching) solution and pour off a portion into a clean flask. Using the atomic absorption spectrophotometer (see 6.1) and hollow-cathode lamp (see 6.2), at the same time determine the absorption of the lead working standards (see 8.2.1) and sample (leaching) solutions, diluting the latter with 4 % acetic acid if required (if solution contains over 20 ppm). Concentrate samples containing less than 1 ppm of lead by accurately transferring a minimum of 50.0 mL of solution to a 250-mL beaker and evaporating to dryness on a steam bath. Dissolve the residue in 4 % acetic acid by adding exactly 0.1 of the volume of the solution taken for concentration, cover with watch glass, and swirl to complete dissolution. Prepare a standard curve of absorption versus concentration (ppm). Determine the lead content (ppm Pb) of sample (leaching) solution from the standard curve. If digital concentration readout is used, the standard curve is not necessary. However, standards bracketing the solution under test should be used.

8.4 *Determination of Cadmium by Atomic Absorption Spectrophotometry*—Proceed as in 8.3 using the cadmium hollow-cathode lamp (see 6.3) and cadmium standards (see 8.2.2). If the sample (leaching) solutions contain more than 2 ppm of Cd, dilute with 4 % acetic acid. Concentrate samples containing less than 0.1 ppm as in 8.3.

9. Report

9.1 Report the type of tile tested, the volume of acid used, the test cell area, and the lead and cadmium leached in parts per million for each tile or sample mount tested.

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

10. Precision and Bias

10.1 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 cadium.

10.2 The bias of this test method is further limited by the ability to obtain representative samples of the statistical universe being sampled.

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[5] *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.

[6] To ensure no loss of solution, a silicone rubber, as described, may be used for grouting. Care should be taken to minimize the amount of grout on the glaze surface.