



Standard Test Method for Lead and Cadmium Extracted from the Lip and Rim Area of Glass Tumblers Externally Decorated with Ceramic Glass Enamels¹

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1. Scope

1.1 This test method covers the determination of lead and cadmium extracted by acetic acid from the lip and rim area of glassware used for drinking and which is exteriorly decorated with ceramic glass enamels. The procedure of extraction may be expected to accelerate the release of lead and cadmium from the decorated area and to serve, therefore, as a severe test that is unlikely to be matched under the actual conditions of usage of such glassware. This test method is specific for lead and cadmium.

NOTE 1—For additional information see Test Method C738.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns 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

3. Terminology

3.1 *Definitions:*

¹ This test method is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.10 on Glass Decoration. It was developed jointly by ASTM Committee C-14 and C-21 on Ceramic Whitewares and Related Products, the Society of Glass Decorators A-20 Subcommittee on Ceramic Enamelled Decorated Glass Tumblers, and an Inter-agency Task Force consisting of FDA, EPA, and CPSC of the U.S. Government.

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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.

3.1.1 *ceramic glass decorations*—ceramic glass enamels fused to glassware at temperatures above 425°C (800°F) to produce a decoration.

3.1.2 *ceramic glass enamels (also ceramic enamels or glass enamels)*—predominately colored, silicate glass fluxes used to decorate glassware.

3.1.3 *lip and rim area*—that part of a drinking vessel which extends 20 mm below the rim on the outside of the specimen.

4. Summary of Test Method

4.1 Lead and cadmium are extracted from the lip and rim area of the article under test by leaching with a 4 % acetic acid solution at 20 to 24°C (68 to 75°F) for 24 h and are measured by atomic absorption spectrophotometry using specific hollow cathode or electrodeless discharge lamps for lead and cadmium respectively. Results are reported as micrograms per millilitre (ppm) extracted relative to the internal volume of the glass article.

5. Significance and Use

5.1 The heavy metals, lead and cadmium, are known to cause serious health effects in man if consumed in excess. It is, therefore, important to measure the amount that may be extracted from an area of the glass drinking vessel in contact with the lip. Even though the amount of lead and cadmium extracted by this test method is in no way representative of the amount of the metals extracted by actual lip contact, the relative magnitude of metals extracted from one test specimen in relation to another test specimen provides an effective tool for discrimination.

6. Interferences

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

7. Apparatus

7.1 *Atomic Absorption Spectrophotometer (AAS)*, equipped with a 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 µg/mL of lead for 1 % absorption and a sensitivity of about 0.025 µg/mL of cadmium for 1 % absorption. Use the operating conditions as specified in the instrument manufacturer's analytical methods manual.

7.2 Hollow Cathode or Electrodeless Discharge Lead Lamp, set at 283.3 nm.

7.3 Hollow Cathode or Electrodeless Discharge Cadmium Lamp, set at 228.8 nm.

7.4 Glassware of chemically resistant borosilicate glass for use in preparing and storing reagents and solutions, and for use as test specimen containers.

7.5 Detection limits of lead and cadmium shall be determined and reported for individual instruments. In this test method, the detection limit shall be defined as twice the mean noise level at 0 µg/mL. Representative detection limits would be approximately 0.01 to 0.03 µg/mL for lead and 0.0005 to 0.0010 µg/mL for cadmium.

8. Reagents

8.1 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.³ 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. Analyze each new batch of reagents for lead and cadmium.

8.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean distilled or deionized water.

8.3 Acetic Acid (4 volume %) —Mix 1 volume of glacial acetic acid with 24 volumes of water.

8.4 Cadmium Standard Stock Solution (1000 µg/mL of cadmium)—Dissolve 0.9273 g of anhydrous cadmium sulfate in 250 mL of 1 % HCl (8.6) and dilute to 500 mL with 1 % HCl. Commercially available standard cadmium solutions may also be used.

8.5 Detergent Rinse—Add 2 mL of hand dishwashing detergent to 1 L of lukewarm tap water.

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

8.7 Lead Standard Stock Solution (1000 µg/mL)—Dissolve 1.598 g of lead nitrate (Pb(NO₃)₂) in 4 % acetic acid and dilute to 1 L with 4 % acetic acid. Commercially available standard lead solutions may also be used.

³ *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.

9. Sampling

9.1 Continuous Process—Since the amount of metal released from a decoration can be affected by the firing conditions, which may not be uniform across the width of the lehr, a minimum of six samples should be taken representing both sides and the center of the lehr.

9.2 Load or Pile—A minimum of six samples should be randomly selected from throughout the load.

10. Preparation of Standards

10.1 Lead Standard Working Solutions—Dilute lead nitrate solution (8.7) with acetic acid (8.3) to obtain working standards having final lead concentrations of 0, 5, 10, 15, and 20 µg/mL.

10.2 Cadmium Standard Working Solutions—Dilute cadmium stock solution (8.4) with acetic acid (8.3) to obtain working standards having final cadmium concentrations of 0.0, 0.3, 0.5, 1, 1.5, and 2.0 µg/mL.

10.3 Fresh working solutions should be prepared daily.

11. Procedure

11.1 Preparation of Sample—Take six identical units and cleanse each with a detergent rinse. Then rinse with tap water followed by distilled water followed by air drying. Mark each unit 7 mm below the rim. Record the internal volume of each article in millilitres by filling from a graduated cylinder to approximately 6 to 7 mm (¼ in.) of overflowing. Mark each article, in a nondecorated area (if possible), 20 mm below the rim on the outside. Invert the article in an appropriate laboratory glassware container whose diameter is a minimum of 1.25 times and a maximum of 2.0 times the diameter of the test specimen at the rim. Carefully add 4 % acetic acid leaching solution from a graduated cylinder to the 20-mm mark. Record the volume of solution used. Cover the glassware containers, if possible, to prevent evaporation and to protect them from contamination. Let stand for 24 h at room temperature (20 to 24°C) in the dark. Remove the article after the 24-h leaching period and determine the lead and cadmium by atomic absorption. Record the lead and cadmium found in micrograms per millilitre.

NOTE 2—The possibility of a significant amount of evaporation exists. The analyst should determine whether the acetic acid leaching is noticeably below the 20-mm mark before removing the article. If it is, sufficient acetic acid solution should be added to restore the leaching solution to the 20-mm mark.

11.2 Determination of Lead—Set the instrument (7.1) for maximum signal at 283.3 nm using the lead hollow cathode lamp (7.2) (Note 3) and air/acetylene (C₂H₂) flow rates recommended by the manufacturer. Stir the sample (leaching) solution and pour off a portion into a clean flask or aspirate from the extraction container if suitable. Flush the burner with water and check zero point between readings. Determine lead from a standard curve of absorbance against µg/mL of lead or calibrate the direct concentration reading (DCR) unit in the concentration mode with lead working solutions (11.1) and read and record the sample concentration directly. Bracket the sample solution with the next higher and lower working

LABORATORY TEST DATA

Lead and Cadmium Released from Lip and Rim Area of Drinking Glassware Decorated Externally with Ceramic Glass Enamels

Date _____	Laboratory _____
Manufacturer _____	
Pattern _____	
Detection Limit Lead _____	Reagent Blank Lead _____
Cadmium _____	Cadmium _____
Internal Volume, mL _____	

Sample	Volume of Leaching Solution, mL	Concentration, µg/mL Lead,	Total µg	ppm Relative to Internal Volume
1				
2				
3				
4				
5				
6				
Avg				
		Cadmium		
1				
2				
3				
4				
5				
6				
Avg				

FIG. 1 Report Form

solutions. Dilute samples containing more than 20 µg/mL of lead with 4 % acetic acid and reanalyze.

NOTE 3—Electrodeless discharge lamps may be substituted for hollow cathode lamps.

11.3 *Determination of Cadmium*—Proceed as in 11.2 using the cadmium hollow cathode lamp (7.3) and cadmium standards (10.2). If the sample (leaching) solution contains more than 2 µg/mL of cadmium, dilute with 4 % acetic acid and reanalyze.

12. Calculation

12.1 Use the following equations to calculate the total amount of lead or cadmium metal released from the lip and rim area of the article expressed (1) in total micrograms and (2) parts per million of lead or cadmium metal leached relative to the internal volume of the article.

12.1.1 Determine lead or cadmium, *A*, in micrograms as follows:

$$A = C \times V_1 \quad (1)$$

12.1.2 Determine lead or cadmium, *A*, in parts per million as follows:

$$A = \frac{C \times V_1}{V_2} \quad (2)$$

where:

C = concentration of lead or cadmium in leaching solution, µg/mL;

*V*₁ = volume of leaching solution, mL; and

*V*₂ = internal volume of article, mL (Note 4).

NOTE 4—The internal volume of the article expressed in millilitres of water closely approximates its weight in grams. Therefore, in this instance microgram per millilitre equals microgram per gram which equals parts per million.

13. Report

13.1 A suggested report form is given in Fig. 1.

14. Precision and Bias

14.1 Precision for the analytical method for single or multiple operator within a single laboratory is within the sensitivity of the AAS used and as specified is about 0.5 µg/mL for lead and 0.25 µg/mL for cadmium.

14.2 The accuracy and between-laboratory precision are dependent upon the ability to obtain representative samples for the statistical universe being sampled.

15. Keywords

15.1 atomic absorption; cadmium; ceramic glass enamels; glaze; heavy metals; lead

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