Designation: D3335 – 85a (Reapproved 2020)

Standard Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy[1]

This standard is issued under the fixed designation D3335; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of lead[2] contents between 0.01 and 5 %, cadmium contents between 50 and 150 ppm (mg/kg), and cobalt contents between 50 and 2000 ppm (mg/kg) present in the nonvolatile portion of liquid coatings or contained in dried films. There is no reason to believe that higher levels of all three elements could not be determined by this test method, provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

1.2 Only pigmented coatings were used for evaluating this test method, but there is no reason to believe that varnishes and lacquers could not be analyzed successfully, provided that appropriate precautions are taken.

1.3 This test method is not applicable to the determination of lead in samples containing antimony pigments (low recoveries are obtained).

1.4 If lead is present in the sample to be analyzed in the form of an *organic* lead compound at a concentration greater than 0.1 %, small losses of lead may occur, resulting in slightly poorer precision than shown in Section 12.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1193 Specification for Reagent Water

D2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings

3. Summary of Test Method

3.1 The specimen of liquid coating or dried film is prepared for analysis by dry ashing. The content of lead, cadmium, or cobalt of an acid extract of the ash is determined by atomic absorption spectroscopy.

4. Significance and Use

4.1 The permissible level of heavy metals in certain coatings is specified by governmental regulatory agencies. This test method provides a fully documented procedure for determining low concentrations of lead, cadmium, and cobalt present in both water and solvent-reducible coatings to determine compliance.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer,* consisting of an atomizer and either a single- or three-slot burner; gas pressure regulating and metering devices for air and acetylene; lead, cadmium, and cobalt source lamps[4] with a regulated constant-current supply; a monochromator and associated optics; a photosensitive detector connected to an electronic amplifier; and a readout device.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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[2] Vandeberg, J. T., Swafford, H. D., and Scott, R. W., "Determination of Low Concentrations of Lead in Paint," *Journal of Paint Technology*, Vol 47, No. 604, May 1975.

[3] 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.

[4] Both hollow cathode lamps and electrodeless discharge lamps have been found satisfactory for this purpose.

5.2 *Muffle Furnace,* capable of maintaining 500 ± 10°C.

5.3 *Crucibles,* wide-form, porcelain, glazed inside and outside except for the outside bottom surface, approximately 30-mL capacity, 50-mm rim diameter and 31-mm height.[5]

5.4 *Hot Plate,* with variable surface temperature control over the range from 70 to 200°C.

5.5 *High-Silica Glass Beakers,*[6]100 and 250-mL.

5.6 *Volumetric Flasks,* 50, 100, and 1000-mL.

5.7 *Dropping Bottles,* ¼ or 7 or 15-mL (½-oz) capacity.

5.8 *Glass or Disposable Syringes,* 5 or 10-mL capacity.

5.9 *Pipets,* 1, 2, 5, and 10-mL capacity.

5.10 *Paint Shaker.*

5.11 *Paint Draw-Down Bar.*

6. Reagents

6.1 *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.[7] 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent grade water conforming to Type II of Specification D1193.

6.3 *Ammonium Acetate Solution* (50 % weight/volume)—Dissolve 500 g of ammonium acetate ($NH_4C_2H_3O_2$) in water and dilute to 1 L.

6.4 *Ammonium Acetate Diluting Solution*—Add 50 mL of HNO_3 (sp gr 1.42) to 150 mL of 50 % weight/volume ammonium acetate solution and dilute to 1 L.

6.5 *Cadmium Standard Stock Solution* (1 mg/mL)—Dissolve 2.1032 g of cadmium nitrate ($Cd(NO_3)_2$) in 10 mL of water, add 10 mL of HNO_3 (sp gr 1.42), and dilute to 1 L.

6.6 *Cobalt Standard Stock Solution* (1 mg/mL)—Dissolve 4.9387 g of cobalt nitrate hexahydrate ($Co(NO_3)_2{\cdot}6H_2O$) in 10 mL of water, add 10 mL of HNO_3 (sp gr 1.42), and dilute to 1 L.

6.7 *Lead Standard Stock Solution* (1 mg/mL)—Dissolve 1.5980 g of lead nitrate ($Pb(NO_3)_2$) in 10 mL of water, add 10 mL of HNO_3 (sp gr 1.42), and dilute to 1 L.

NOTE 1—10.00 mg/mL concentrations of cadmium, lead, silver and zinc are available as SRM 2121; and the same concentration of cobalt, copper, iron and nickel as SRM 2124 from: Office of Standard Reference Materials, Room B-311, Chemistry Building, NIST, Washington, DC 20234.

6.8 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

6.9 *Nitric Acid* (1 + 1)—Add 1 volume of HNO_3 (sp gr 1.42) to 1 volume of water.

7. Hazards

7.1 Concentrated nitric acid is corrosive and may cause severe burns of the skin or eyes; the vapor is irritating to mucous membranes. Use care in handling this acidic substance. Refer to suppliers' Material Safety Data Sheet.

7.2 Use only a rubber bulb aspirator for pipeting liquids.

8. Calibration and Standardization

8.1 Prepare 100-mL quantities of at least four standard solutions bracketing the expected lead, cadmium, or cobalt concentration in the sample to be tested. To suitable aliquots of the 1 mg/mL standard lead, cadmium, or cobalt solution, add 5 mL of HNO_3 (sp gr 1.42) and 15 mL of 50 % ammonium acetate solution. Dilute to 100 mL with water.

8.2 Operational instructions for atomic absorption spectrophotometers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

8.3 Turn the instrument on and set the wavelength to the 283.3-nm lead line, the 228.8-nm cadmium line, or the 240.7-nm cobalt line. Apply the current recommended by the manufacturer to the lead, cadmium, or cobalt source lamp. Allow the instrument to warm up for about 15 min and set the slit width. Adjust the air and acetylene pressure or flow rates and ignite the burner in accordance with the manufacturer's instructions.

8.4 Aspirate water to rinse the atomizer chamber. Aspirate a standard solution and make any necessary readjustment in instrument parameters to obtain maximum absorption.

8.5 Aspirate each of the appropriate standard solutions and record the corresponding instrument readings. Aspirate water between each standard.

8.6 Construct a calibration curve on linear graph paper by plotting the absorbance versus concentration (micrograms per millilitre) for each standard solution. Alternatively, the calibration results may be stored in the instrument, if so equipped, and readings made directly in concentration.

9. Procedure

9.1 If the sample is a liquid coating, mix it until homogeneous, preferably on a mechanical shaker. Determine the nonvolatile content in accordance with Guide D2832.

[5] The sole source of supply of No. 25007 crucibles, known to the committee at this time is Coors Manufacturer. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, [1] which you may attend.

[6] The sole source of supply of Vycor beakers, known to the committee at this time is Corning Glass Co., Houghton Park, Corning, NY 14831. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, [1] which you may attend.

[7] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.