



Standard Test Method for Detection of Lead in Paint by Direct Aspiration Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 4834; 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 is intended as a screening test to determine if the solids in a paint contain more than 0.06 % lead. The test described can differentiate between 0.05 and 0.06 %. Paints giving a result greater than 0.05 % should be analyzed quantitatively for lead using Test Method D 3335. This test method provides a more definitive and reliable screening test than Test Method D 3618.

1.2 There is no reason to believe that higher levels of lead could not be determined by this test method provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

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:

- D 1193 Specification for Reagent Water²
- D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings³
- D 3335 Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy³
- D 3618 Test Method for Detection of Lead in Paint and Dried Paint Films³

3. Summary of Test Method

3.1 A weighed quantity of the paint sample is diluted (solvent-reducible paints in methyl isobutyl ketone and water-reducible paints in water) and then aspirated into the burner of an atomic absorption spectrometer. Lead content of the paint is determined from a calibration plot of absorbance versus concentration, prepared from standard coatings containing known amounts of lead.

4. Significance and Use

4.1 Current U.S. regulations restrict the amount of lead that may be present in consumer paint products to less than 0.06 % based on the total solids. This test method provides a rapid means for screening paints to determine compliance with those regulations. The test method may be adjusted to meet regulations with a different limit on lead content.

5. Apparatus

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

5.2 *Volumetric Flasks*, 50 mL.

6. Reagent and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. 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.⁴ 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 water conforming to Type II of Specification D 1193.

6.3 *Methyl Isobutyl Ketone (MIBK)*, technical grade.

6.4 *Standard Paints*—Prepare at least 3 standard paints by adding accurately weighed quantities of an analytically assayed compatible lead compound to a solvent-reducible or water-reducible paint that has been analyzed by Test Method D 3335 and shown to contain less than 10 ppm of lead. Determine the nonvolatile content of the base paint and choose quantities of the lead compound so that the lead concentrations of the

¹ This test method is under the jurisdiction of ASTM Committee D-1 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 *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *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.

standard paints will be approximately 300, 600, and 900 µg/g based on the nonvolatile content.

7. Calibration and Standardization

7.1 Prepare at least three solvent-reducible or water-reducible standard paint specimens (see 6.4) that bracket the expected lead concentration in the sample to be tested. Following the procedure in 8.2 dilute the standards for aspiration.

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

7.3 Turn the instrument on and set the wavelength to the 283.3-nm lead line. Apply the current recommended by the manufacturer to the lead 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. A background corrector should be used.

7.4 Aspirate each of the diluted standard paints and record the corresponding absorbance readings. Aspirate MIBK or water, as appropriate, between standards.

NOTE 1—Caution: It is important that MIBK or water be aspirated immediately after each diluted standard paint to minimize fouling of the aspirator/nebulizer system which could change the aspiration rate and absorbance reading. Continued response for lead while aspirating MIBK or water may indicate contamination of the burner head with pigment and require cleaning of the burner head.

NOTE 2—To be certain that the diluted paint specimen remains homogeneous during aspiration, the flask containing the specimen may be stirred continuously with a small magnetic stirring bar.

7.5 Construct a calibration curve on linear graph paper by plotting the absorbance readings versus micrograms of lead present in each diluted standard paint as follows:

$$A = (B \times D \times E) \quad (1)$$

where:

- A = lead present, µg,
- B = lead in standard paint based on nonvolatile content (see 6.4), µg/g,
- D = weight of standard paint, g, and
- E = nonvolatile portion of standard paint, %.

8. Procedure

8.1 Mix the paint to be tested until homogeneous, preferably on a mechanical shaker. Determine the nonvolatile content in accordance with the appropriate test method selected by consulting Guide D 2832.

8.2 Prepare at least two replicate specimens by weighing by difference from a weighing bottle or disposable syringe to 0.1 mg, 0.5 ± 0.02-g quantities of the paint being tested, into 10 mL volumetric flasks. Dilute to the mark with MIBK, if the paint is solvent reducible, or with water, if it is water reducible. Mix thoroughly.

NOTE 3—Adjustment of specimen size and dilution volume may be made as required for linear calibration and reasonable response for the instrument used as well as for differing lead content limits.

8.3 Aspirate the diluted paint specimens *immediately* (**Caution**—See Note 1) after mixing and measure the absorbance (see Note 2). Aspirate MIBK or water, as appropriate between specimen aspirations. Determine the micrograms of lead present from the calibration curve.

NOTE 4—For maximum accuracy, calibration and standardization (See Section 7) should be completed just prior to aspirating the diluted paint specimen.

9. Calculation

9.1 Calculate the mean concentration of lead in the nonvolatile portion of the paint specimen as follows:

$$L = (C \times 100)/(NV \times S) \quad (2)$$

where:

- L = lead in nonvolatile portion of the paint specimen, ppm (µg/g),
- C = lead in the aspirated specimen solution (8.3), µg,
- 100 = factor derived from multiplying the numerator by 100 (to convert NV used to a whole number) and 10⁶ (to obtain ppm) and multiplying the denominator by 10⁶ (to convert grams of specimen to micrograms),
- NV = nonvolatile portion of paint specimen, %, and
- S = weight of specimen, g.

10. Report

10.1 Report the lead concentration expressed on the nonvolatile content of the paint sample.

11. Precision and Bias ⁵

11.1 *Precision*—The precision estimates are based on an interlaboratory study in which five different laboratories analyzed in duplicate on 2 days, three samples of solvent-reducible paints and three samples of water-reducible paints containing from 464 to 742 ppm lead. The within-laboratory coefficient of variation was found to be 3.34 % relative at 24 df and the between-laboratories coefficient of variation was 6.72 % relative at 18 df. Based on these coefficients, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 9.76 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 19.96 % relative.

11.2 *Bias*—Bias is under investigation.

12. Keywords

12.1 lead content—paint; lead screening test; spectroscopy—atomic adsorption

⁵ Supporting data is available from ASTM Headquarters. Request RR: D01-1060.

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