



Standard Test Method for Detection of Lead in Paint and Dried Paint Films¹

This standard is issued under the fixed designation D 3618; 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 0.5 % lead. The test described barely detects the presence of 0.4 % but gives a definite positive result at the 0.5 % level.

NOTE 1—This test method may be used to detect the presence of lead at concentrations higher or lower than 0.5 % by making appropriate changes in the specimen size and reagent quantities specified.

1.2 Paints giving an unexpected positive or questionable result should be analyzed quantitatively for lead, using Test Method D 3335.

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.* Specific hazard statements are given in Section 7.

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

3. Summary of Test Method

3.1 The sample of liquid paint or dried film is prepared by dry ashing a weighed specimen at 475 to 500°C. The ash is extracted with hot sodium hydroxide solution and a drop of the extract is transferred to filter paper. Lead present is oxidized to lead peroxide with bromine water, then treated with “tetrabase” to produce a blue quinoidal salt. Known amounts of lead are added to standard paints that are concurrently tested to provide a base for comparison.

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. 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 present in both water and solvent-reducible coatings to determine compliance.

5. Apparatus

- 5.1 *Burner*, Meker-type.
- 5.2 *Crucibles*, porcelain, high-form, 15-mL, with covers.
- 5.3 *Filter Paper*, ashless, medium texture.
- 5.4 *Hot Plate*, with variable surface temperature control over the range from 70 to 200°C.
- 5.5 *Muffle Furnace*, maintained at $475 \pm 25^\circ\text{C}$.
- 5.6 *Syringe*, glass, 2-mL.
- 5.7 *Volumetric Flasks*, 50, 100, 1000-mL.
- 5.8 *Paint Shaker*.
- 5.9 *Paint Draw-Down Bar*.

6. Reagents

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 grade water conforming to Type II of Specification D 1193.

6.3 *Acetic Acid*, glacial.

6.4 *Ammonium Hydroxide* (1 + 1)—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH , sp gr 0.90) with 1 volume of water.

6.5 *Bromine Water*, saturated.

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

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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

6.7 *Lead Standard Stock Solution, Aqueous* (1 mg/mL)—Dissolve 1.600 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 100 mL of water, add 2 mL of nitric acid (sp gr 1.42), and dilute to 1 L.

6.8 *Lead Standard Working Solution, Aqueous* (0.2 mg/mL)—Pipet 10 mL of the aqueous lead standard stock solution into a 50-mL volumetric flask and dilute to volume with water.

6.9 *Toluene*, technical grade.

6.10 *Lead Standard Stock Solution, Solvent-Based* (1 mg/mL)—Dissolve 4.2 g of lead naphthenate (containing 24 % lead) in 100 mL of toluene and dilute to 1 L.

NOTE 2—Other toluene-soluble lead driers such as lead tallate, lead linoleate, and lead octoate may also be used provided that the lead content is accurately known and the appropriate weight is diluted to 100 mL to provide a toluene solution containing 1 mg/mL of lead.

6.11 *Lead Standard Working Solution, Solvent-Based* (0.2 mg/mL)—Pipet 10 mL of the solvent-based standard stock solution into a 50-mL volumetric flask and dilute to volume with toluene.

6.12 *Sodium Hydroxide Solution* (12 % w/v)—Carefully dissolve 12 g of sodium hydroxide in 100 mL of water.

6.13 *Paint, Standard Solvent-Reducible*, any solvent-reducible paint known to contain less than 100 ppm of lead.

6.14 *Paint, Standard Water-Reducible*, any water-reducible paint known to contain less than 100 ppm of lead.

6.15 *Tetrapase Reagent* (4,4'-Methylenebis (N,N-dimethylaniline)), 1 % in 10 % Acetic Acid—Dissolve 1 g of tetrapase⁵ in 50 mL of water to which 10 mL of glacial acetic acid has been added. Dilute to 100 mL with water.

7. Hazards

7.1 Glacial acetic and concentrated nitric acids will cause burns of the skin and eyes. Sodium hydroxide is corrosive. Use care in handling these materials. Avoid contact with skin. Refer to suppliers' Material Safety Data Sheets. Bromine water should be prepared and used in a laboratory hood.⁶

7.2 Use only a rubber bulb aspirator for pipetting liquids.

8. Procedure

8.1 If the sample is a liquid coating, mix it until homogeneous, preferably on a mechanical shaker. Determine the nonvolatile content in accordance with Guide D 2832.

NOTE 3—Recover dried paint films from previously coated substrates (being careful not to remove any underlying material from the substrate) or prepare in the laboratory from liquid samples. For the laboratory preparation, flow some of the well-mixed sample onto a clean glass plate. The use of a paint draw-down bar is recommended to obtain a uniform film thickness not exceeding 2 mils (50 μm). Allow to dry in an oven at 105°C for a minimum of 1 h. Scrape the dried film off the glass plate, preferably with a single edge razor blade.

8.2 Weigh to 0.1 mg, 50 mg of paint solids into a porcelain crucible or 50-mL glass beaker. For a liquid coating, determine the specimen weight to be taken by the following equation:

$$S = \frac{5}{C} \quad (1)$$

where:

S = specimen weight, g, and

C = nonvolatile content, %

NOTE 4—Crucibles or beakers used in this test method should be new or in very good condition. Otherwise, it is possible that some lead may be lost and a false negative obtained.

8.3 Weigh 50 mg of solids from the appropriate standard paint (solvent-reducible or water-reducible) into each of two additional porcelain crucibles or 50-mL glass beakers. Using a 2-mL glass syringe, add 1.0 mL of the appropriate 200-ppm standard working solution (solvent-reducible or water-reducible) to one of the crucibles or beakers, and 1.8 mL to the other.

NOTE 5—The standard paint to which has been added 1.0 mL of standard working solution contains approximately 0.4 % lead based on the paint solids, and the one to which has been added 1.8 mL of standard working solution will contain approximately 0.72 % lead based on the paint solids.

8.4 Place all three crucibles or beakers on a cold hot plate and slowly increase the temperature until the material is dried. With some types of coatings, an initial oven-drying at 105°C may be necessary to remove solvents without incurring losses due to spattering.

8.5 When the specimens appear to be dry, or when starting with a dried film, gradually increase the temperature of the hot plate until the material chars.

8.6 After charring is complete, place the crucibles or beakers in a preheated muffle furnace and ash at 475 to 500°C.

8.7 When the ashing appears to be complete, (approximately 1 to 2 h) remove the crucibles or beakers from the muffle furnace and allow them to cool to room temperature. Add 5 mL of 3 *N* sodium hydroxide solution to each crucible or beaker and mix thoroughly with a stirring rod. Scrape any adhering residue from the sides or bottom of the container and break up any particles, using a separate stirring rod for each specimen.

8.8 Place covers on the crucibles or watch glasses on the beakers, and boil gently for approximately 5 min. Remove from the hot plate and allow the contents to settle. Do not filter.

8.9 With the tip of the stirring rod, transfer 1 to 2 drops of each extract to the center of a piece of filter paper. Using separate droppers, successively apply directly on each specimen spot a drop of bromine water and a drop of NH_4OH solution. Remove excess ammonia by holding each filter paper over a hot plate until no odor is detectable, but do not take to complete dryness. Place 2 drops of tetrapase reagent on each spot and compare the color developed on the specimen spot with those on the standard paint spots. To ensure that maximum color development has been achieved, add 1 or 2 more drops of tetrapase reagent on the center of each spot after the first addition has almost-dried (if still wet, the color may diffuse).

NOTE 6—Although the color is stable, it is barely visible in the range from 0.4 to 0.5 %. Comparing results with those obtained on the two standard paints helps to orient the observer to the color intensity to be expected.

NOTE 7—Certain iron-based pigments have high natural manganese

⁵ The sole source of supply of tetrapase, Eastman-244, known to the committee at this time is Eastman Organic Chemicals, Rochester, NY 14650. 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.

⁶ Saturated bromine water may be purchased from chemical supply houses.

levels. The ash extract of products containing high manganese may produce a diffuse blue ring on the filter paper. With experience, this is easily distinguished from the small blue spot produced by lead.

9. Report

9.1 Report a positive test (lead content equal to or in excess of 0.5 % of paint solids) when a blue color forms and persists for several seconds.

10. Precision and Bias ⁷

10.1 An interlaboratory test was conducted in which analysts from eight laboratories tested water and solvent-based coatings having lead contents slightly lower and slightly higher than 0.5 % based on the solids. All analysts made correct judgments for greater than 0.5 % lead (positive) and less than 0.5 % lead (negative), with only one analyst reporting a single result as “questionable.”

11. Keywords

11.1 lead; lead in paints and dry paint films; spat test for lead

⁷ Supporting data are available from ASTM Headquarters. Request RR: D01-1007.

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