



Designation: D 3717 – 85a (Reapproved 1999)

Standard Test Method for Low Concentrations of Antimony in Paint by Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation D 3717; 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 determination of the content of antimony in the range between 50 and 200 ppm (mg/kg) present in the solids of liquid coatings or in dried films obtained from previously coated substrates. There is no reason to believe that higher levels could not be determined by this test method, provided that appropriate dilutions and adjustments in specimen size and reagent quantities are made.

1.2 *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

3. Summary of Test Method

3.1 The sample of liquid coating or dried film is prepared for analysis by dry ashing at 500°C, followed by refluxing with hydrochloric acid and stannous chloride. The antimony content of the acid extract 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 antimony 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; an antimony 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 *Muffle Furnace*, maintained at $500 \pm 10^\circ\text{C}$.

5.3 *Oven*, maintained at $105 \pm 2^\circ\text{C}$.

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

5.5 *Reflux Condenser*, water-cooled, and fitted with a standard-taper joint.

5.6 *Erlenmeyer Flask*, 125-mL, with standard-taper joint to fit condenser.

5.7 *Volumetric Flasks*, 100 and 1000-mL.

5.8 *Dropping Bottles*, $\frac{1}{4}$ or $\frac{1}{2}$ -oz (8 or 15-mL) capacity.

5.9 *Glass or Disposable Syringes*, 10-mL capacity.

5.10 *Pipets*, 1, 5, 10, and 15-mL capacity.

5.11 *Filter Paper*, ashless, medium or slow filtering.

5.12 *Paint Shaker*.

5.13 *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.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.

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.

Current edition approved May 31 and Nov. 29, 1985. Published January 1986. Originally published as D 3717 – 78. Last previous edition D 3717 – 84.

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.