Designation: D3718 – 85a (Reapproved 2015)

Standard Test Method for Low Concentrations of Chromium in Paint by Atomic Absorption Spectroscopy[1]

This standard is issued under the fixed designation D3718; 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 the content of chromium (including chromium oxide) in the range between 0.005 and 1.0 % 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 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 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:*[2]

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 sample of liquid coating or dried film is prepared for analysis by dry ashing at 500°C followed by digestion with potassium permanganate and sulfuric acid in a polytetrafluoroethylene (PTFE)-lined acid decomposition vessel at an elevated temperature. The chromium in the filtered digestion mixture 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 chromium 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 nitrous oxide (N_2O) and acetylene; a chromium hollow cathode 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 ± 10°C.

5.3 *Force-Draft Oven,* maintained at 105 ± 2°C.

5.4 *Acid Decomposition Vessel,* with PTFE digestion cup.[3]

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

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

5.7 *Pipets,* 5, 10, 15, and 20-mL capacity.

5.8 *Filter Paper,* ashless, medium filtering 15-cm.

5.9 *Paint Shaker.*

[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.

Current edition approved June 1, 2015. Published June 2015. Originally approved in 1978. Last previous edition approved in 2010 as D3718 – 85a (2010). DOI: 10.1520/D3718-85AR15.

[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] The sole source of supply of an acid decomposition cup, (Catalog Number 4745), known to the committee at this time is the Parr Instrument Co., 211 Fifty-third St., Moline, IL 61265. 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.

5.10 *Crucibles,* wide form, porcelain, approximately 30-mL capacity.[4]

5.11 *Mortar and Pestle.*

5.12 *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.[5] 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 D1193.

6.3 *Chromium Standard Stock Solution* (1 mg/mL)—Dissolve 3.735 g of potassium chromate (K_2CrO_4) in 100 mL of water and dilute to 1 L.

6.4 *Chromium Standard Working Solution* (0.1 mg/mL)—Pipet 10 mL of the chromium standard stock solution into a 100-mL volumetric flask and dilute to volume with water.

6.5 *Oxidizing Solution*—Dissolve 0.2 g of potassium permanganate ($KMnO_4$) in 100 mL of H_2SO_4 (1 + 1). Stir until completely dissolved. The color of this solution is dark brown.

6.6 *Reducing Solution*—Dissolve 1 g of hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) in 100 mL of water.

6.7 *Reagent Blank*—Pipet 5 mL of oxidizing solution into 25 mL of water contained in a 50-mL volumetric flask. Add reducing solution dropwise until the permanganate color has been discharged; then dilute to 50 mL with water.

6.8 *Sulfuric Acid* (1 + 1)—Carefully add 1 volume of concentrated H_2SO_4 (sp gr 1.84) to 1 volume of water.

6.9 *Sulfuric Acid* (5 % volume per volume)—Carefully add 50 mL of concentrated H_2SO_4 (sp gr 1.84) to 500 mL of water and dilute to 1 L.

7. Hazards

7.1 Use care in handling concentrated H_2SO_4 because it is corrosive and may cause severe burns of the skin or eyes. Refer to suppliers' Material Safety Data Sheet.

7.2 The National Institute for Occupational Safety and Health has stated that hexavalent chromium compounds are hazardous to health. Care should be exercised in preparation of a sample for test. The wearing of a respirator and rubber or synthetic gloves is recommended. In case of contact, wash thoroughly with soap and water. Mixtures containing hexavalent chromium compounds should not be flushed down a drain but disposed of as hazardous waste.

7.3 Use only a rubber bulb aspirator for pipetting liquids.

8. Calibration and Standardization

8.1 Prepare 50-mL quantities of at least four standard solutions bracketing the expected chromium concentration in the sample to be tested. To suitable aliquots of the 100-ppm chromium standard working solution, add 10 mL of the oxidizing solution with a pipet, followed by the dropwise addition of reducing solution until the permanganate color has been discharged, then dilute to 50 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 357.9-nm chromium line. Apply the recommended current to the chromium hollow-cathode lamp. Allow the instrument to warm up for about 15 min and set the slit width. Adjust the nitrous oxide and acetylene pressures and ignite the burner according to 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. Zero the instrument while aspirating reagent blank solution (6.7). Aspirate each of the appropriate standard solutions and record the corresponding instrument readings. Aspirate water between each standard.

8.5 Transfer a 25-mL aliquot from each of the standard solutions prepared in 8.1 to 50-mL volumetric flasks and dilute to volume with water. Repeat the steps outlined in 8.4 for the diluted aliquot solutions.

8.6 Construct a calibration curve on linear graph paper by plotting the absorbance versus concentration (micrograms per millilitre) for each set of standard solutions.

NOTE 1—To obtain maximum accuracy one should complete calibration and standardization just prior to sample analysis.

9. Procedure

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

9.2 Determine the ash content of the material under test in duplicate.

9.2.1 Weigh to the nearest 0.1 mg approximately 5 g of liquid coating or 3 g of dried film into each of two tared porcelain crucibles.

NOTE 2—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.

[4] The sole source of supply of No. 25007 crucibles, known to the committee at this time is Coors Co. 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.

[5] *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.