Designation: D2350 – 90 (Reapproved 2015)

Standard Test Method for Antimony Oxide in White Pigment Separated From Solvent-Reducible Paints[1]

This standard is issued under the fixed designation D2350; 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 total antimony oxide in white pigment separated from solvent-reducible paints.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D2371 Test Method for Pigment Content of Solvent-Reducible Paints

3. Summary of Test Method

3.1 The pigment is dissolved in hydrochloric acid (HCl). Sulfuric acid (H_2SO_4) is added. The mixture is titrated with potassium permanganate ($KMnO_4$) and calculated to antimony oxide (Sb_2O_3) which gives antimony in the *ous* condition.

3.2 The pigment is dissolved in H_2SO_4 with potassium sulfate (K_2SO_4) and reduced. Sodium sulfite (Na_2SO_3) is added and sulfur dioxide gas (SO_2) is expelled. The solution is diluted and HCl added after which the solution is titrated with $KMnO_4$, which gives total antimony calculated to Sb_2O_3.

3.3 Sb_2O_3 from *ous* condition is subtracted from total Sb_2O_3 and residual Sb_2O_3 is calculated to Sb_2O_5.

3.4 The procedure is also described for antimony oxide in presence of large amounts of iron.

4. Significance and Use

4.1 Antimony trioxide is often used in fire-retardant paints, so it is useful to formulators and users to be able to monitor the amount of this compound in whole paints.

5. Reagents

5.1 *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.[3] 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent grade water in accordance with Specification D1193.

5.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated HCl.

5.4 *Hydrogen Sulfide* (H_2S).

5.5 *Potassium Permanganate, Standard Solution* (0.1 *N*)—Dissolve 3.2 g of pure potassium permanganate ($KMnO_4$) in 1 L of water, let stand 8 to 14 days, and siphon off the clear solution (or filter through a glass filter). For use in determining antimony, the $KMnO_4$ solution is best standardized as follows: To 0.25 g of pure metallic antimony in a 500-mL resistant-glass Erlenmeyer flask, add 12 to 15 mL of H_2SO_4 (sp gr 1.84) and

[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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This standard has been approved for use by agencies of the Department of Defense to replace Method 7016 of Federal Test Method Standard No. 141. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been approved by the Department of Defense.

[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] *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.