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Designation: D2350 – 90 (Reapproved 2005)

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 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 the standard. The values given in parentheses are for information only.

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 10 to 12 g of K_2SO_4. Heat until all the antimony is dissolved, cool, dilute to 100 mL with water, add 1 to 2 g of sodium sulfite (Na_2SO_3), and boil until all the SO_2 is expelled. This expulsion

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[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 Jan. 1, 2005. Published February 2005. Originally approved in 1965. Last previous edition approved in 1999 as D2350 – 90 (1999).

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. DOI: 10.1520/D2350-90R05.

[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.

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is evident when no blue color is obtained with starch-iodate paper (5.7). The volume will be reduced about one half. Dilute to 250 mL with water, add 20 mL of HCl (sp gr 1.19) and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution.

NOTE 1—For normal routine control work, the reduction with sodium sulfite may be eliminated. The material may be diluted to 250 mL, 20 mL of HCl added, and titrated immediately after cooling the sulfuric acid-potassium sulfate digestion.

5.6 *Potassium Sulfate* (K_2SO_4).

5.7 *Starch-Iodate Paper*—Impregnate filter paper with a solution obtained by heating 2 g of starch with 100 mL of water, and, after solution, adding 0.2 g of potassium iodate (KIO_3) dissolved in 5 mL of water.

5.8 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4).

5.9 *Tartaric Acid*.

6. Preparation of Sample

6.1 Separate and prepare the pigment in accordance with Test Method D2371.

7. Procedure

7.1 Transfer 0.3 g of a straight Sb_2O_3 pigment, or 0.5 g of a mixed pigment, to a 500-mL resistant-glass Erlenmeyer flask, add 15 mL of water and 25 mL of HCl (sp gr 1.19). Cover with a watch glass, warm on the steam bath for 10 to 15 min to dissolve the antimony oxide, wash off the cover, add 250 mL of water, and 15 mL of H_2SO_4(sp gr 1.84). Boil 2 min, cool to 10 to 15°C, and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution. Calculate to Sb_2O_3.

7.2 The procedure described in 7.1 gives only the antimony in the *ous* conditions. The following method gives the total antimony (*ous* and *ic* forms): Transfer 0.3 g of a straight Sb_2O_3 pigment, or 0.5 g of a mixed pigment, to a 500-mL resistant-glass Erlenmeyer flask, and add 15 mL of H_2SO_4(sp gr 1.84), 10 g of K_2SO_4, and a 9-cm filter paper (to furnish carbon to act as a reducing agent). Place a funnel in the neck of the flask and heat until the solution becomes colorless. Cool, wash off the funnel, dilute to 100 mL with water, add 1 to 2 g of Na_2SO_3, and boil until all the SO_2 is expelled. This expulsion is evident when no blue color is obtained with starch-iodate paper (5.7). The volume will be reduced about one half. Dilute to 250 mL with water, add 20 mL of HCl (sp gr 1.19), and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (Note 1).

7.2.1 Calculate total antimony to Sb_2O_3. Subtract the Sb_2O_3 found using the procedure given in 7.1 from the total Sb_2O_3 and calculate the residual Sb_2O_3 to Sb_2O_5(multiply by the factor 1.1098).

7.3 *Procedure in Presence of Appreciable Amounts of Iron*:

7.3.1 Treat 1 g of the mixed pigment, or 0.3 g of a tinted Sb_2O_3 pigment, in a covered 250-mL beaker with 5 mL of water and 20 mL of HCl (sp gr 1.19). Heat on the steam bath for 15 min, cool, wash off the cover, add 3 g of tartaric acid and 100 mL of hot water, and digest a few minutes. Filter, catching the filtrate in a 500-mL resistant-glass Erlenmeyer flask. Wash thoroughly with hot water, dilute to 300 mL with hot water, and pass in H_2S until the precipitation is complete. (If the sample contains no insoluble matter, dissolve directly in a 500-mL resistant-glass Erlenmeyer flask, add tartaric acid, dilute, and pass in H_2S.)

7.3.2 Filter, wash with water containing H_2S until free from HCl, return paper and precipitate to the Erlenmeyer flask, add 15 mL of H_2SO_4(sp gr 1.84) and 10 g of K_2SO_4, place a funnel in the neck of the flask, and heat until the solution is colorless. Cool, wash off the funnel, dilute to 100 mL with water, add 1 to 2 g of sodium sulfite (Na_2SO_3), and boil until all the SO_2 is expelled. This expulsion is evident when no blue color is obtained with starch-iodate paper (5.7). The volume will be reduced about one-half. Dilute to 250 mL with water, add 20 mL of HCl (sp gr 1.19), and titrate to a faint pink tint with 0.1 *N* $KMnO_4$ solution (Note 1).

8. Calculation

8.1 Calculate the percent antimony, *B*, as Sb_2O_3 as follows:

$$B = \frac{AV}{S} \times 100 \quad (1)$$

where:

A = Sb_2O_3 equivalent of the $KMnO_4$ solution, g/mL,
V = $KMnO_4$ solution required, mL, and
S = sample used, g.

9. Precision

9.1 Data are not available to determine the precision of this test method. There are no plans at present to obtain such data. This test method has been in use for several years and is considered acceptable.

10. Keywords

10.1 antimony oxide; white pigment

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