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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Chemical Analysis of Mercuric Oxide Pigment[1]

This standard is issued under the fixed designation D 284; 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 These test methods cover procedures for the chemical analysis of mercuric oxide pigment.

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.* A specific hazard statement is given in the Note 1 of 11.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1208 Test Methods for Common Properties of Certain Pigments[3]

3. Significance and Use

3.1 These test methods are intended as a quick and reliable procedure for measuring purity of mercuric oxide pigment to determine if it meets purity standards as agreed upon between the producer and the consumer.

4. Purity of Reagents and Materials

4.1 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.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

5. Preparation of Sample

5.1 If the sample is large, mix it thoroughly before taking a representative portion. Grind the representative portion to a fine powder and thoroughly mix before taking portions for analysis. Keep the sample in a stoppered glass bottle.

6. Precision

6.1 Repeatability and reproducibility are believed to be well within the limits usually obtained on similar chemical test methods, but no actual figures on precision are available.

ALKALINITY OR ACIDITY

7. Procedure

7.1 Determine alkalinity or acidity in accordance with Test Methods D 1208.

FREE MERCURY

8. Procedure

8.1 Examine a representative specimen of the dry mercuric oxide under a microscope for the presence of free mercury.

TOTAL MERCURY

9. Reagents and Materials

9.1 *Ammonium Thiocyanate, Standard Solution* (1 mL = 0.012 g Hg)—Dissolve 9 g of ammonium thiocyanate (NH_4CNS) in water and dilute to 1 L. Standardize the solution against mercury, as follows: Weigh to 0.1 mg about 4.6 g of mercury, and dissolve it in 40 mL of warm HNO_3 (1+1). Dilute to 200 mL with water and add $KMnO_4$ solution (50 g/L) dropwise until the pink color persists for 5 min in order to ensure the absence of nitrous acid (HNO_2) and monovalent mercury. Add $FeSO_4$ solution (50 g/L) dropwise to destroy excess permanganate. Add 4 mL of ferric ammonium sulfate indicator solution, and titrate with the NH_4CNS solution.

9.2 Calculate the mercury equivalent M of the NH_4CNS solution, in grams per millilitre, as follows:

$$M = W_1/V_1$$

where:

W_1 = mercury used, g, and

V_1 = NH_4CNS solution required for titration, g.

9.3 *Ferric Ammonium Sulfate Indicator Solution*—Dissolve enough ferric ammonium sulfate ($Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H$

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[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D 01.31 on Pigment Specifications.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 06.03.

[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.

$_2$O) in water to make a saturated solution at room temperature and add HNO_3 (sp gr 1.42) dropwise to bleach the brown color of the solution. About 30 g of ferric ammonium sulfate per 100 mL of water will be required for the saturated solution.

9.4 *Ferrous Sulfate Solution* (50 g/L)—Dissolve 5 g of ferrous sulfate ($FeSO_4 \cdot 7H_2O$) in water and dilute to 100 mL.

9.5 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (NHO_3).

9.6 *Nitric Acid* (1+1)—Mix 1 volume of HNO_3 (sp gr 1.42) with 1 volume of water. This acid must be free of nitrous acid (HNO_2).

9.7 *Potassium Permanganate Solution* (50 g/L)—Dissolve 5 g of potassium permanganate ($KMnO_4$) in water and dilute to 100 mL.

10. Procedure

10.1 Weigh to 1 mg about 0.5 g of the sample, previously dried for 1 h at 150°C into a 750-mL Erlenmeyer flask. Add 40 mL of HNO_3 (1+1), and warm gently until the specimen is dissolved.

10.2 Dilute to 200 mL with water, and add 4 mL of ferric ammonium sulfate indicator solution. Titrate with 0.1 *N* NH_4CNS solution until a distinct pink color persists after vigorous shaking.

11. Calculation

11.1 Calculate the percent of mercury *P* as follows:

$$P = [(V_2 \times M)/S] \times 100$$

where:

V_2 = NH_4CNS solution required for titration of the specimen, mL
M = mercury equivalent of the NH_4CNS solution, g/mL, and
S = dried specimen, g.

ASH

12. Procedure

12.1 Ignite 2.0 g of a dry specimen in a weighed porcelain crucible or dish under a well-ventilated hood. (**Warning**—See Note 1) Cool and weigh the residue. Calculate the percent of ash.

NOTE 1—**Warning:** The fumes are poisonous.

13. Keywords

13.1 mercuric oxide; pigments (mercuric oxide)