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Standard Test Methods for Analysis of Barium Sulfate Pigment[1]

This standard is issued under the fixed designation D715; 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 the analysis of barium sulfate pigment.

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 the safety concerns 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]

D185 Test Methods for Coarse Particles in Pigments
D280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments
D1193 Specification for Reagent Water
D1208 Test Methods for Common Properties of Certain Pigments
E832 Specification for Laboratory Filter Papers

3. Significance and Use

3.1 These test methods are used to determine the purity of barium sulfate and to determine the concentration of known impurities. This information is useful to producers and users as an aid in the manufacture of coatings.

4. Purity of Reagents

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.[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.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D1193.

BARIUM SULFATE

5. Reagents

5.1 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH_4OH).

5.2 *Ammonium Sulfate* ($(NH_4)_2SO_4$).

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

5.4 *Hydrochloric Acid (1+1)*—Mix equal volumes of concentrated HCl (sp gr 1.19) and water.

5.5 *Methyl Red Indicator Solution*—Dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

5.6 *Sodium Carbonate Solution (30 g/L)*—Dissolve 30 g of Na_2CO_3 in water and dilute to 1 L.

6. Procedure

6.1 Weigh to 0.1 mg approximately 0.5 g of the sample into a platinum crucible, add 3 g of Na_2CO_3, mix thoroughly, and fuse until the melt is clear. Allow the melt to cool and then leach in a platinum dish with hot water until it is entirely disintegrated. Filter on a close-texture paper and wash thoroughly with hot Na_2CO_3 solution (30 g/L).

6.2 Transfer the filter paper containing the insoluble carbonates to a 250-mL beaker and acidify with concentrated HCl (sp gr 1.19). Wash the fusion crucible with HCl (sp gr 1.19) so that no barium is lost. Boil the solution, filter into a 600-mL beaker, and wash well with water. Add methyl red indicator solution,

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[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Committee D01.31 on Pigment Specifications.

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[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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and add NH_4OH (sp gr 0.90) until the solution is slightly alkaline. Add 6 mL of HCl (1+1), and dilute to 300 mL.

6.3 Heat the solution to boiling, and add 5 g of $(NH_4)_2SO_4$ dissolved in 40 mL of water. If low-grade material is being analyzed, the $(NH_4)_2SO_4$ solution should be added drop by drop from a buret to minimize inclusion. If the barytes is a rather pure product (95 to 99 % $BaSO_4$), this is not necessary, since the only nonvolatile constituents of the solution will be barium salts. Allow the precipitate of $BaSO_4$ to digest for 4 h or overnight, and filter through a close-texture paper. Wash the precipitate with as little cold water as possible (consistent with the purity of the precipitate), ignite in an oxidizing atmosphere, and weigh as $BaSO_4$.

7. Calculation

7.1 Calculate the percent of $BaSO_4$ as follows:

$$BaSO_4, \% = \left(P/S_1\right) \times 100 \quad (1)$$

where:

P = $BaSO_4$, g, and
S_1 = sample used, g.

FERRIC OXIDE

8. Apparatus

8.1 *Colorimetric Apparatus*—Nessler type or other similar 100-mL colorimetric tubes.

9. Reagents

9.1 *Ammonium Thiocyanate Solution (76.1 g/L)*—Dissolve 76 g of ammonium thiocyanate (NH_4CNS) in water and dilute to 1 L.

9.2 *Iron, Standard Solution (100 mL = 0.00002 g Fe)*—Dilute and divide a ferric solution of known iron content so as to obtain 0.4 mg of iron. Dilute the solution to 2 L with water containing 200 mL of iron-free H_2SO_4.

9.3 *Potassium Permanganate Solution (0.1 g/L)*—Dissolve 0.1 g of potassium permanganate ($KMnO_4$) in water and dilute to 1 L.

9.4 *Standard Color Solution*—Mix thoroughly 10 parts by volume of NH_4CNS and 90 parts by volume of standard iron solution (100 mL = 0.02 mg Fe). One hundred millilitres of the solution will thus contain 0.000018 mg of Fe.

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

9.6 *Sulfuric Acid (1+1)*—Carefully mix 1 volume of concentrated H_2SO_4 (sp gr 1.84) with 1 volume of water.

10. Procedure

10.1 Dissolve the soluble portion of a 1-g specimen in H_2SO_4 (1+1), filter, and wash, keeping the volume of the solution under 100 mL. Oxidize any iron present in the filtrate by adding potassium permanganate ($KMnO_4$) until a faint pink color is obtained. Dilute the solution to 100 mL and pour into a buret graduated in 0.1-mL divisions.

10.2 Pour 100 mL of the standard color solution into a 100-mL colorimetric tube. Into a second colorimetric tube pour 10 mL of concentrated H_2SO_4 (sp gr 1.84) and 10 mL of NH_4CNS, dilute to 60 or 70 mL and then add the test solution from the buret until the depth of color thus produced on dilution to 100 mL exactly matches that of the standard. Record the number of millilitres required.

11. Calculation

11.1 Calculate the percent of Fe_2O_3 as follows:

$$Fe_2O_3, \% = \left[\left(I \times 1.4298\right)/S_2\right] \times 100 \quad (2)$$

where:

I = iron in standard, g,
1.4298 = $\frac{Fe_2O_3}{Fe_2} = \frac{159.694}{111.694}$, and
S_2 = sample used, g.

HYDROGEN ION CONCENTRATION

12. Procedure

12.1 Determine the pH in accordance with Test Methods D1208.

MATTER SOLUBLE IN WATER

13. Procedure

13.1 Determine the amount of water soluble material present in accordance with Test Methods D1208.

MOISTURE AND OTHER VOLATILE MATTER

14. Procedure

14.1 Determine the moisture and other volatile matter in accordance with Test Methods D280.

COARSE PARTICLES

(*Total Residue Retained on No. 325* (*45-µm*)*Sieve*)

15. Procedure

15.1 Determine the coarse particles in accordance with Test Methods D185.

FREE SILICA

16. Reagent

16.1 *Heavy Liquid (sp gr 2.9)*—Dissolve 4 parts by weight of potassium iodide (KI) in 6 parts of warm water, and add, while stirring constantly, 5 parts of mercuric iodide (HgI_2). Evaporate over a hot plate or gas flame, protected by screen cloth, until a light crystalline scum forms. Cool, and filter through heavy paper. The solution will be a clear, deep, amber color of about 3.2 specific gravity. All filter washings showing a yellow color should be saved and evaporated. Determine the 3.2 specific gravity accurately by means of a specific gravity bottle, and correct the solution to a specific gravity of 2.9 at room temperature by adding water. Prepare approximately 200 mL of the solution.

17. Procedure

17.1 Carefully weigh 10 g of the finely ground dry barium sulfate pigment and transfer to a carefully dried 125-mL clear glass separatory funnel of the long narrow type. Add 100 mL of the heavy liquid (sp gr 2.9), stopper, and shake well to liberate any air bubbles that might adhere to the particles, and also to break up all agglomerates. Set the separatory funnel aside and allow to stand in a perpendicular position for 2 h, or for a sufficient time for the particles to rise or sink.

17.2 Draw off the heavy particles through the stopcock into a beaker along with 75 mL of the liquid. Add 50 mL of fresh heavy liquid (sp gr 2.9) to the float particles and liquid remaining in the separatory funnel and repeat the procedure as described in 17.1.

17.3 Draw off the heavy particles along with 75 mL of the liquid and add to those drawn off in accordance with 17.1, leaving the float particles in the funnel. Wash the particles remaining in the funnel with fresh heavy liquid (sp gr 2.9), filter through a No. 2/0 paper (see Specification E832), and wash free of solution with warm water.

17.4 Transfer the filter paper and silica to an ignition cup, dry, and ignite carefully in an electric furnace or over a flame to keep mechanical loss at a minimum. Weigh the floated material, and transfer to a tared platinum dish or crucible. Add 20 mL of hydrofluoric acid (HF) and 3 drops of H_2SO_4 (1+1), evaporate to dryness, and ignite carefully to expel all fumes. Cool and weigh. The loss in weight represents the free silica in the floated material.

18. Calculation

18.1 Calculate the percent of free SiO_2 as follows:

$$\text{Free SiO}_2, \% = (A/S_3) \times 100 \tag{3}$$

where:

A = SiO_2, g, and
S_3 = sample used, g.

19. Precision and Bias

19.1 The precision and bias of these test methods has not been determined.

20. Keywords

20.1 analysis of pigment; barium sulfate; barium sulfate content; barium sulfate pigment; ferric oxide in barium sulfate; free silica in barium sulfate

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