



## Standard Test Methods for Analysis of Barium Sulfate Pigment<sup>1</sup>

This standard is issued under the fixed designation D 715; 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 ( $\epsilon$ ) 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 *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:

D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints<sup>2</sup>

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

D 1208 Test Methods for Common Properties of Certain Pigments<sup>2</sup>

E 832 Specification for Laboratory Filter Papers<sup>4</sup>

### 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.<sup>5</sup> 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.

## BARIUM SULFATE

### 5. Reagents

5.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide ( $\text{NH}_4\text{OH}$ ).

5.2 *Ammonium Sulfate* ( $(\text{NH}_4)_2\text{SO}_4$ ).

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  $\text{Na}_2\text{CO}_3$  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  $\text{Na}_2\text{CO}_3$ , 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  $\text{Na}_2\text{CO}_3$  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, and add  $\text{NH}_4\text{OH}$  (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  $(\text{NH}_4)_2\text{SO}_4$  dissolved in 40 mL of water. If low-grade material is being analyzed, the  $(\text{NH}_4)_2\text{SO}_4$  solution should be added drop by drop from a buret to minimize inclusion. If the barytes is a rather pure product (95 to 99 %  $\text{BaSO}_4$ ), this is not necessary, since the only nonvolatile constituents of the solution will be barium salts. Allow the precipitate of  $\text{BaSO}_4$  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  $\text{BaSO}_4$ .

<sup>1</sup> 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 Committee D01.31 on Pigment Specifications.

Current edition approved Sept. 26, 1986. Published November 1986. Originally published as D 715 – 43. Last previous edition D 715 – 75 (1981)  $\epsilon^1$ .

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>5</sup> *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.