



Designation: D 717 – 86 (Reapproved 1999)

Standard Test Methods for Analysis of Magnesium Silicate Pigment¹

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1. Scope

1.1 These test methods cover the analysis of magnesium silicate 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 234 Specification for Raw Linseed Oil²

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²

D 718 Test Methods for Analysis of Aluminum Silicate Pigment²

D 1193 Specification for Reagent Water³

D 1208 Test Methods for Common Properties of Certain Pigments²

D 2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment²

E 97 Test Method for Directional Reflectance Factor, 45-deg 0-deg, of Opaque Specimens by Broad-Band Filter Reflectometry⁴

3. Significance and Use

3.1 These test methods may be used to confirm the stated SiO₂, CaO, and MgO content of magnesium silicate for quality control.

4. Apparatus

4.1 *Platinum Crucible.*

4.2 *Electric Furnace, capable of 1200°C.*

5. Purity of Reagents

5.1 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.⁵ 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 Unless otherwise indicated, references to water shall be understood to mean Type II of Specification D 1193.

SILICON DIOXIDE

6. Reagents

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

6.2 *Hydrochloric Acid* (1+20)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 20 volumes of water.

6.3 *Hydrofluoric Acid* (48 %)—Concentrated hydrofluoric acid (HF).

6.4 *Sodium Carbonate* (Na₂CO₃).

6.5 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

7. Procedure

7.1 Transfer 1 g of the sample weighed to 0.1 mg to a platinum crucible and fuse with 5 g of Na₂CO₃ until the entire contents of the crucible are in a molten state. Continue heating for 20 min. Keep a close-fitting platinum cover on the crucible during the fusion. When the fusion is complete, allow the crucible and contents to cool, and transfer to a 600-mL porcelain casserole containing 200 mL of water (Note 1). Boil until the melt is disintegrated.

NOTE 1—If, during the cooling period, the crucible is partially immersed several times in cold water to chill the outer portions of the melt, the subsequent removal of the melt is facilitated. Do not allow the water to enter the crucible while the contents are hot to avoid spattering.

7.2 Remove crucible and lid, being careful to scrub and rinse out any adhering particles of the melt. Carefully acidify the contents of the casserole with concentrated HCl (sp gr

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ Discontinued; see 1992 *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

ASTM D 717

1.19); introduce the HCl in small portions, keeping a watch glass over the crucible to avoid loss by spattering. Add 30 mL of HCl in excess and evaporate to dryness on a steam bath; take care to break up any crusts that form. When the material appears completely dry, and no odor of HCl can be detected, remove the casserole from the steam bath, and allow to cool.

7.3 Wash down the sides of the casserole with 20 mL of HCl (sp gr 1.19) and then with water. Repeat the evaporation as described in 7.2, then bake for 1 h in an oven at 105°C. Cool the residue, drench with 25 mL of HCl (sp gr 1.19), add 175 mL of water, and warm, while stirring, until all soluble salts are dissolved. Filter off the silica on a close-texture paper, wash five times with HCl (1+20), wash five times with hot water, and reserve the filtrate for determination of other oxides (Section 9).

7.4 Transfer the paper and washed silica to a clean platinum crucible, ignite, first gently until the filter paper is consumed, and then at 1200°C for 20 min, cool, and weigh. Moisten the residue with water, add 5 drops of H₂SO₄ (sp gr 1.84), and 15 mL of HF. Evaporate to dryness on a steam bath, heat gently until H₂SO₄ has been expelled, and ignite at 1200°C for 5 min. Cool and weigh. The loss in weight represents the SiO₂.

8. Calculation

8.1 Calculate the percent of silica as follows:

$$\text{SiO}_2, \% = (P/S) \times 100 \quad (1)$$

where:

P = SiO₂, g, and

S = sample used, g.

AMMONIUM HYDROXIDE GROUP (Alumina and Iron Oxide)

9. Reagents

9.1 *Ammonium Chloride Solution* (2 g/100 mL)—Dissolve 2 g of ammonium chloride (NH₄Cl) in 100 mL of water.

9.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

9.3 *Hydrochloric Acid* (1+3)—Mix 1 volume of concentrated HCl (sp gr 1.19) with 3 volumes of water.

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

9.5 *Potassium Pyrosulfate*—(K₂S₂O₇).

10. Procedure

10.1 If an appreciable residue remains after the treatment with HF in accordance with 7.4, fuse the residue with a small amount of K₂S₂O₇ until it is dissolved. Leach the pyrosulfate melt out of the crucible with water and combine the solution with the filtrate reserved in accordance with 7.3.

10.2 Using the methyl red indicator solution, neutralize the combined solutions from the silica determination with NH₄OH and add an excess of 2 drops. Bring to a boil adding NH₄OH 1 drop at a time if necessary to maintain a slight alkalinity. Allow the precipitate to settle (not more than 5 min) and filter. Wash four times with hot NH₄Cl solution.

10.3 Set aside the filtrate and transfer the precipitate and filter paper to the same beaker in which the first precipitation

was effected. Dissolve the precipitate with 40 mL of hot HCl (1+3), dilute the solution to about 100 mL, and reprecipitate the hydroxides as described in 10.2. Filter and wash the precipitate four times with hot NH₄Cl. Combine the filtrate and washings with the filtrate set aside and reserve for the determination of CaO (Section 10).

10.4 Place the precipitate in a weighed platinum crucible, heat slowly until the papers are charred, and finally ignite to constant weight at 1050 to 1100°C with care to prevent reduction. Cool and weigh as R₂O₃ (aluminum and iron oxides).

11. Calculation

11.1 Calculate the percent of R₂O₃ as follows:

$$\text{R}_2\text{O}_3, \% = (P_2/S) \times 100 \quad (2)$$

where:

P_2 = R₂O₃, g, and

S = sample used, g.

CALCIUM OXIDE

12. Reagents

12.1 *Ammonium Hydroxide* (1+1)—Mix 1 volume of concentrated NH₄OH (sp gr 0.90) with 1 volume of water.

12.2 *Ammonium Oxalate Solution*—((NH₄)₂C₂O₄·H₂O), saturated.

12.3 *Ammonium Oxalate Solution* (1 g/L)—Dilute 1.0 g of ammonium oxalate ((NH₄)₂C₂O₄·H₂O) in 1 L of water.

12.4 *Hydrochloric Acid* (1+3)—See 9.3.

12.5 *Hydrochloric Acid* (1+1)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1 volume of water.

12.6 *Methyl Red Indicator Solution*—See 9.4.

13. Procedure

13.1 Acidify the combined filtrates obtained in the precipitations of the ammonium hydroxide group (Section 10) with HCl and evaporate them to a volume of about 300 mL. Add 5 mL of HCl (1+1), a few drops of methyl red indicator solution, and 30 mL of warm ammonium oxalate solution (saturated). Heat the solution to 70 to 80°C and add NH₄OH (1+1) dropwise while stirring, until the color changes from red to yellow. Allow the solution to stand without further heating for 1 h (no longer) with occasional stirring during the first 30 min. Filter and wash moderately with cold dilute ammonium oxalate solution. Reserve the filtrate and washings.

13.2 Transfer the precipitate and filter paper to the beaker in which the precipitate was effected. Dissolve the oxalate in 40 mL of hot HCl (1+3) and macerate the filter paper. Dilute to 200 mL with water, add a few drops of methyl red indicator solution and 5 mL of ammonium oxalate solution (saturated). Heat the solution nearly to boiling, and precipitate calcium oxalate again by neutralizing the acid solution with NH₄OH as described in 13.1. Allow the solution to stand 1 to 2 h and wash as before. Combine the filtrate with that already obtained and reserve for the determination of MgO (Section 16).

13.3 Dry the precipitate in a weighed covered platinum crucible. Char the paper without inflaming, burn the carbon at