



Standard Test Methods for Analysis of Magnesium Silicate Pigment¹

This standard is issued under the fixed designation D 717; 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 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

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 D01.31 on Pigment Specifications.

Current edition approved April 25, 1986. Published June 1986. Originally published as D 717 – 43. Last previous edition D 717 – 73 (1979)^{ε2}.

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.

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

as low a temperature as possible, and, finally, heat with the crucible tightly covered in an electric furnace or over a blast lamp at a temperature of 1100 to 1200°C. Cool in a desiccator and weigh as CaO. Repeat the ignition to a constant weight.

14. Calculation

14.1 Calculate the percent of CaO as follows:

$$\text{CaO, \%} = (P_3/S) \times 100 \quad (3)$$

where:

P_3 = CaO, g, and

S = sample used, g.

MAGNESIUM OXIDE

15. Reagents

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

15.2 *Ammonium Hydroxide* (5+95)—Mix 5 volumes of concentrated NH_4OH (sp gr 0.90) with 95 volumes of water.

15.3 *Diammonium Phosphate Solution* (250 g/L)—Dissolve 250 g of diammonium phosphate ($(\text{NH}_4)_2\text{HPO}_4$) in enough water to yield 1 L of solution.

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

16. Procedure

16.1 Acidify the filtrates reserved in the determination of CaO (Section 13) with HCl and concentrate to about 400 mL. Add to this solution about 50 mL of $(\text{NH}_4)_2\text{HPO}_4$ (250 g/L) and if the solution becomes alkaline, add HCl (1+3) to keep the solution acidic. Cool the solution by placing the beaker in a cold water trough or in a tray of ice water. After cooling add NH_4OH drop by drop, while stirring constantly, until the crystalline magnesium ammonium phosphate begins to form, and then in moderate excess (5 to 10 % of the volume of the solution), the stirring being continued for several minutes. Set the solution aside for at least 8 h in a cool atmosphere.

16.2 Filter the solution on a close-texture paper and wash with NH_4OH (5+95). Unfold the filter paper and, using hot water, wash the precipitate into the beaker in which the precipitation was effected. Rinse the filter paper with hot HCl (1+3) and again with hot water; if necessary add more hot HCl (1+3) to dissolve the precipitate. Dilute the solution to about 250 mL, add 1 mL of $(\text{NH}_4)_2\text{HPO}_4$ solution, and then add concentrated NH_4OH drop by drop, while stirring constantly, until the precipitate is again formed as described in the NH_4OH is in moderate excess. Cool and allow to stand for about 4 h.

16.3 Filter the solution on a close-texture paper and wash six times with NH_4OH (5+95). Transfer the precipitate wrapped in the moist paper to a weighed platinum or porcelain crucible, slowly char the paper without allowing it to ignite and carefully burn off the carbon over a gradually increasing flame which shall never heat the crucible to more than the faintest red. Finally ignite at 1000 to 1100°C to constant weight, taking care to avoid melting the pyrophosphate.

17. Calculation

17.1 Calculate the percent of MgO to 0.1 as follows:

$$\text{MgO, \%} = [(P_4 \times 0.3623)/S] \times 100 \quad (4)$$

where:

P_4 = $\text{Mg}_2\text{P}_2\text{O}_7$, g, and

S = sample used, g.

$$\frac{\text{molecular weight } 0.2 \text{ MgO}}{\text{molecular weight } \text{Mg}_2\text{P}_2\text{O}_7} = \frac{2 \times 40.32}{222.60} = 0.3623 \quad (5)$$

17.2 Consider the sum of MgO, CaO, and SiO_2 as combined magnesium and calcium silicate.

LOSS ON IGNITION

18. Procedure

18.1 Determine the loss on ignition in accordance with Test Methods D 1208.

MOISTURE AND OTHER VOLATILE MATTER

19. Procedure

19.1 Determine the moisture and other volatile matter by Test Methods D 280.

COARSE PARTICLES

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

20. Procedure

20.1 Determine the coarse particles in accordance with Test Methods D 718, except that the specimen weight should be adjusted to provide appropriate sensitivity for the residual weight determination. Any difficulty encountered in wetting-out the specimen can be overcome by prewetting with a 50+50 mixture of acetone or alcohol and water.

MATTER SOLUBLE IN WATER

21. Procedure

21.1 Determine the matter soluble in water by Test Method D 2448 or alternatively by Test Methods D 1208.

COLOR

22. Standard Pigment

22.1 *Standard Extender Pigment.*

22.2 *Standard Zinc Oxide.*

23. Procedure

23.1 Carefully weigh out the proportions of the standard extender pigment and standard zinc oxide mutually agreed upon, and rub up to a fairly stiff paste with a glass muller on a glass plate or stone slab with raw linseed oil conforming to Specification D 234. Note the volume of the oil required. Prepare a similar paste with the sample, using the same weight of pigment, volume of oil, and number of strokes of the glass muller as used in the preparation of the paste of the standard pigments. Spread the pastes adjacently on a microscope slide, draw a scraper lightly across them so as to present them on an even plane, and judge the color immediately.

24. Dry Brightness

24.1 The test sample and reference sample shall be prepared into suitable smooth, dry, packed surfaces in accordance with

accepted practice and tested for reflectance using the green filter in accordance with Test Method E 97.

obtain such information. The methods have been in use for several years and are considered acceptable.

PRECISION

25. Precision and Bias

25.1 Data are not available to determine the precision and bias of these test methods. There are no plans at present to

26. Keywords

26.1 calcium oxide in magnesium silicate; magnesium oxide in magnesium silicate; magnesium silicate pigment, analysis of; silicon dioxide, analysis of

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