



Designation: D717 – 86 (Reapproved 2020)

Standard Test Methods for Analysis of Magnesium Silicate Pigment¹

This standard is issued under the fixed designation D717; 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 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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D234 Specification for Raw Linseed Oil (Withdrawn 2007)³

D280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments

D718 Test Methods for Analysis of Aluminum Silicate Pigment

D1193 Specification for Reagent Water

D1208 Test Methods for Common Properties of Certain Pigments

D2448 Test Method for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment

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 Subcommittee 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 The last approved version of this historical standard is referenced on www.astm.org.

E97 Method of Test for Directional Reflectance Factor, 45-Deg 0-Deg, of Opaque Specimens by Broad-Band Filter Reflectometry (Withdrawn 1991)³

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.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.

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

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).*

⁴ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

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

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 Na_2CO_3 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 1.19); 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 H_2SO_4 (sp gr 1.84), and 15 mL of HF. Evaporate to dryness on a steam bath, heat gently until H_2SO_4 has been expelled, and ignite at 1200°C for 5 min. Cool and weigh. The loss in weight represents the SiO_2 .

8. Calculation

8.1 Calculate the percent of silica as follows:

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

where:

P = SiO_2 , 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 (NH_4Cl) in 100 mL of water.

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

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*—($\text{K}_2\text{S}_2\text{O}_7$).

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 $\text{K}_2\text{S}_2\text{O}_7$ 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 NH_4OH and add an excess of 2 drops. Bring to a boil adding NH_4OH 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 NH_4Cl 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 NH_4Cl . 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 R_2O_3 (aluminum and iron oxides).

11. Calculation

11.1 Calculate the percent of R_2O_3 as follows:

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

where:

P_2 = R_2O_3 , g, and
 S = sample used, g.

CALCIUM OXIDE

12. Reagents

12.1 *Ammonium Hydroxide (1+1)*—Mix 1 volume of concentrated NH_4OH (sp gr 0.90) with 1 volume of water.

12.2 *Ammonium Oxalate Solution*—($(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$), saturated.

12.3 *Ammonium Oxalate Solution (1 g/L)*—Dilute 1.0 g of ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$) 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 (NH₄OH).

15.2 *Ammonium Hydroxide (5+95)*—Mix 5 volumes of concentrated NH₄OH (sp gr 0.90) with 95 volumes of water.

15.3 *Diammonium Phosphate Solution (250 g/L)*—Dissolve 250 g of diammonium phosphate (NH₄)₂HPO₄ 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 (NH₄)₂HPO₄ (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

NH₄OH 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 NH₄OH (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 (NH₄)₂HPO₄ solution, and then add concentrated NH₄OH drop by drop, while stirring constantly, until the precipitate is again formed as described in the NH₄OH 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 NH₄OH (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 = Mg₂P₂O₇, 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 SiO₂ as combined magnesium and calcium silicate.

LOSS ON IGNITION

18. Procedure

18.1 Determine the loss on ignition in accordance with Test Methods D1208.

MOISTURE AND OTHER VOLATILE MATTER

19. Procedure

19.1 Determine the moisture and other volatile matter by Test Methods D280.

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 D718, 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.