



Designation: D 718 – 86 (Reapproved 2003)

Standard Test Methods for Analysis of Aluminum Silicate Pigment¹

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1. Scope

1.1 These test methods cover the analysis of aluminum 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 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 Pigments³

D 717 Test Methods for Analysis of Magnesium 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 11 Specification for Wire Cloth and Sieves for Testing Purposes⁴

3. Significance and Use

3.1 These test methods may be used to confirm the stated aluminum oxide and SiO₂ content of aluminum silicate for quality control.

4. Apparatus

4.1 *Platinum Crucible.*

4.2 *Electric furnace* (or gas burner), capable of 1050 to 1100°C.

4.3 *Volumetric flask*, 100 and 250 mL.

4.4 *Colorimeter*, with transmission range from 400 to 550 nm.

4.5 *High Silica Crucible.*

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,⁵ where such specifications are available. 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. Procedure

6.1 Determine the silicon dioxide content in accordance with Test Methods D 717.

ALUMINUM OXIDE

7. Reagents

7.1 *Ammonium Acetate (20 %)*—Dissolve 200 g of ammonium acetate (NH₄C₂H₃O₂) in 1 L of distilled water.

7.2 *Ammonium Chloride Solution (20 g/L)*—Dissolve 20 g of ammonium chloride (NH₄Cl) in water and dilute to 1 L.

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

7.4 *Diphenylamine Indicator Solution (1 g/100 mL)*—Dissolve 1 g of diphenylamine in 100 mL of concentrated sulfuric acid (H₂SO₄, sp gr 1.84).

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 *Annual Book of ASTM Standards*, Vol 06.03.

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

⁴ *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 Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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

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

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

7.8 *Hydrogen Peroxide* (H₂O₂, 3 %, freshly prepared)—Mix 1 volume of H₂O₂ (30 %) with 9 volumes of distilled water.

7.9 *Hydroxylamine Hydrochloride* (10 %)—Dissolve 10 g of NH₂OH · HCl in 100 mL of distilled water. Prepare fresh weekly.

7.10 *Iron Standard*—Dissolve 0.1 g of analytical grade iron wire in 10 mL of HCl (1+1) and dilute to 1 L. Each millilitre contains 0.1 mg of Fe.

7.11 *Mercuric Chloride Solution* (HgCl₂), saturated.

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

7.13 *Potassium Dichromate, Standard Solution* (0.05 N)—Dissolve 2.457 g of potassium dichromate (K₂Cr₂O₇) in water and dilute to 1 L. Standardize against National Bureau of Standards' standard sample No. 27b of Sibley iron ore, using such an amount as to give approximately the same titration as the sample to be analyzed.

7.14 *Phosphoric Acid* (H₃PO₄, 85 %)—Concentrated phosphoric acid.

7.15 *Potassium Pyrosulfate* (K₂S₂O₇).

7.16 *o-Phenanthroline* (0.1 %)—Dissolve 1.0 g of *o*-phenanthroline in 1 L of hot distilled water.

7.17 *Stannous Chloride Solution* (5 g/100 mL)—Dissolve 5 g of stannous chloride (SnCl₂ · 2H₂O) in 10 mL of concentrated HCl (sp gr 1.19) and dilute to 100 mL with water. Add scraps of iron-free granulated tin, and boil until the solution is clear. Keep the solution in a closed dropping bottle containing metallic tin.

7.18 *Sulfuric Acid* (1+1)—Add carefully 1 volume of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) to 1 volume of distilled water.

7.19 *Sulfuric Acid* (1+9)—Add carefully 1 volume of concentrated H₂SO₄ (sp gr 1.84) to 9 volumes of distilled water.

8. Procedure

8.1 If an appreciable residue remains after the treatment with HF in accordance with 7.4 of Test Methods D 717, 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 or Test Methods D 717.

8.2 Bring the volume of the combined solution to 250 mL, and, if necessary, add HCl in order to ensure a total of 10 to 15 mL of HCl, add a few drops of methyl red indicator solution, and heat to boiling. Add concentrated NH₄OH (sp gr 0.90) dropwise until 1 drop changes the color of the solution to a distinct yellow. Reheat the solution containing the precipitated hydroxides to boiling, boil for 1 or 2 min, and filter. Wash the precipitate once by decantation and then slightly on the filter with hot NH₄Cl.

8.3 Transfer the precipitate and paper to the original beaker in which the precipitation was made. Dissolve the precipitate in

hot HCl (1+3), dilute to 100 mL, and precipitate again as described in 8.2. After filtering, wash the precipitate ten times with small portions of hot NH₄Cl solution. Transfer the precipitate to a weighed platinum crucible, heat slowly until the paper is charred, and finally ignite to constant weight at 1050 to 1100°C in an electric furnace or over a burner, taking care to avoid reduction. Weigh the precipitate as Al₂O₃ + TiO₂ + Fe₂O₃.

8.4 Fuse the combined oxides from 8.3 with 9 to 10 g of K₂S₂O₇ in a platinum crucible, starting at low temperature and increasing the heat gradually until the oxides have all dissolved. Take up the melt with 5 mL of H₂SO₄ (1+1) in 150 mL of water and warm to effect solution. Transfer the solution to a 250-mL volumetric flask, dilute to volume, and mix well. Reserve for TiO₂ and Fe₂O₃ determinations.

8.5 Determine the percent of TiO₂ as follows: Pipet an aliquot containing 0.2 to 3.0 mg of TiO₂ into a 100-mL volumetric flask. For samples containing 0.5 to 3 % TiO₂, a 20-mL aliquot is suitable. Add 5 mL of H₂O₂ (3 %), dilute to volume with H₂SO₄ (1+9) and mix well. Obtain the colorimetric reading in a suitable colorimeter using a filter with transmission limits of 400 to 450 nm or at 410 nm with a prism or grating spectrometer. Compare the readings to a curve plotted from a set of TiO₂ standards similarly treated and read on the same instrument.

8.6 Determine the percent of Fe₂O₃ as follows: Pipet a 25-mL aliquot into a 100-mL volumetric flask. Add the following in the order given, mixing well after each addition: 2 mL of NH₂OH · HCl (10 %), 10 mL of NH₄C₂H₃O₂ (20 %), and 10 mL of *o*-phenanthroline (0.1 %). Roll a small piece of congo red paper into a ball and introduce into the flask. Add concentrated NH₄OH dropwise until indicator turns red and 1 drop in excess. Dilute to volume and let stand for 10 to 20 min. Obtain the colorimetric reading in a suitable colorimeter using a filter with transmission limits 485 to 550 nm, or at 510 nm with a prism or grating spectrometer. Compare the readings to a curve plotted from a set of Fe₂O₃ standards similarly treated and read on the same instrument.

8.7 Alternatively the Fe₂O₃ may be determined by titration as follows: Fuse 1 g of sample with 10 g of K₂S₂O₇ in a high-silica crucible starting at low temperature and increasing the heat gradually until the crucible glows with a dull red color and decomposition is judged to be complete. Leach the melt with 10 mL of concentrated HCl in 100 mL of water and digest at low heat to disintegrate the cake. Filter and wash free of chlorides with hot water. Save filtrate. Ignite the insoluble residue in a platinum crucible. Treat with 5 mL of H₂SO₄ (1+1) and 15 mL of HF (48 %), and heat until fumes of H₂SO₄ appear. Bring the residue into solution with the addition of a few drops more of H₂SO₄ (1+1) and combine the solution with the bulk of the iron. Add 5 mL of HCl to the combined solutions and evaporate to 50-mL volume.

8.8 To the hot solution add SnCl₂ solution, dropwise, while stirring, until the solution is decolorized, and then add 2 to 3 drops more. Cool the solution to room temperature, wash down the inside of the beaker with water, and add at one stroke 10 mL of cool HgCl₂ solution (saturated). Stir the solution vigorously for 1 min, add 5 mL of phosphoric acid (85 %) and