Designation: D 718 – 86 (Reapproved 1999)

Standard Test Methods for Analysis of Aluminum Silicate Pigment[1]

This standard is issued under the fixed designation D 718; 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 aluminum 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 Oil[2]

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments[3]

D 717 Test Methods for Analysis of Magnesium Silicate Pigment[2]

D 1193 Specification for Reagent Water[3]

D 1208 Test Methods for Common Properties of Certain Pigments[2]

D 2448 Test Methods for Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

3. Significance and Use

3.1 These test methods may be used to confirm the stated aluminum oxide and SiO_2 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 ($NH_4C\ _2H_3O_2$) in 1 L of distilled water.

7.2 *Ammonium Chloride Solution* (20 g/L)—Dissolve 20 g of ammonium chloride ($NH\ _4Cl$) in water and dilute to 1 L.

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

7.4 *Diphenylamine Indicator Solution* (1 g/100 mL)—Dissolve 1 g of diphenylamine in 100 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84).

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* (H_2O_2, 3 %, freshly prepared)—Mix 1 volume of H_2O_2 (30 %) with 9 volumes of distilled water.

7.9 *Hydroxylamine Hydrochloride* (10 %)—Dissolve 10 g

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[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.

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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.

of $NH_2OH \cdot HCl$ 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* ($HgCl_2$), 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 ($K_2Cr_2O_7$) 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* (H_3PO_4, 85 %)—Concentrated phosphoric acid.

7.15 *Potassium Pyrosulfate* ($K_2S_2O_7$).

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 ($SnCl_3$, $2H_2O$) 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 (H_2SO_4, sp gr 1.84) to 1 volume of distilled water.

7.19 *Sulfuric Acid* (1+9)—Add carefully 1 volume of concentrated H_2SO_4 (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 $K_2S_2O_7$ 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 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 NH_4OH (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 NH_4Cl.

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 NH_4Cl 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 $Al_2O_3 + TiO_2 + Fe_2O_3$.

8.4 Fuse the combined oxides from 8.3 with 9 to 10 g of $K_2S_2O_7$ 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 H_2SO_4 (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 TiO_2 and Fe_2O_3 determinations.

8.5 Determine the percent of TiO_2 as follows: Pipet an aliquot containing 0.2 to 3.0 mg of TiO_2 into a 100-mL volumetric flask. For samples containing 0.5 to 3 % TiO_2, a 20-mL aliquot is suitable. Add 5 mL of H_2O_2 (3 %), dilute to volume with H_2SO_4 (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 TiO_2 standards similarly treated and read on the same instrument.

8.6 Determine the percent of Fe_2O_3 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 $NH_2OH \cdot HCl$ (10 %), 10 mL of $NH_4C_2H_3O_2$ (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 NH_4OH 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 Fe_2O_3 standards similarly treated and read on the same instrument.

8.7 Alternatively the Fe_2O_3 may be determined by titration as follows: Fuse 1 g of sample with 10 g of $K_2S_2O_7$ 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 H_2SO_4 (1+1) and 15 mL of HF (48 %), and heat until fumes of H_2SO_4 appear. Bring the residue into solution with the addition of a few drops more of H_2SO_4 (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 $SnCl_2$ 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 $HgCl_2$ solution (saturated). Stir the solution vigorously for 1 min, add 5 mL of phosphoric acid (85 %) and 3 drops of diphenylamine indicator solution. Titrate with 0.05 *N* $K_2Cr_2O_7$ solution to an intense deep-blue end point that remains unchanged on further addition of $K_2Cr_2O_7$ solution.

8.9 Calculate the percent of Fe_2O_3 as follows:

$$Fe_2O_3\,\% = [(N \times V \times 0.0798)/S] \times 100 \quad (1)$$

where:

S = sample used, g,
V = $K_2Cr_2O_7$, mL, and
N = normality of the $K_2Cr_2O_7$.

8.10 Calculate the percent of Al_2O_3 as follows:

$$Al_2O_3, \% = 100(P/S) - C \tag{2}$$

where:

P = weight of combined oxides $Al_2O_3 + TiO_2 + Fe_2O_3$,
S = specimen weight, g, and
C = $Fe_2O_3 + TiO_2$, %.

LOSS ON IGNITION

9. Procedure

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

MOISTURE AND OTHER VOLATILE MATTER

10. Procedure

10.1 Determine moisture and other volatile matter in accordance with Test Methods D 280.

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

11. Apparatus

11.1 *Sieve*—A No. 325 (45-µm) 3-in. (76 mm) diameter high form sieve conforming to Specification E 11, shall be used.

11.2 *Stirring Apparatus*, consisting of:

11.2.1 *Mixing Cup*—A stainless steel cup, about 7 in. (178 mm) deep, and slightly tapered from an outside diameter at the top of about 4 in. (102 mm) to about 2¾ in. (70 mm) at the bottom, such as is characteristic of a usual malted-milk type mixing cup.

11.2.2 *Mechanically Operated Stirring Device*, in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 r/min without load. The shaft shall be equipped with a replaceable stirring button not more than 1 in. (25 mm) in diameter, and of such length that the stirring button shall operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38 mm) above the bottom of the dispersion cup. A common malted milk-type mixer of high quality meets this specification.

12. Procedure

12.1 Dry a No. 325 (45-µm) sieve in an oven at 105± 2°C, cool, and weigh accurately. Transfer 100 ± 0.5 g of the specimen to the mixing cup containing approximately 250 mL of water to which has been added about 2 drops of 0.5 % solution sodium silicate, and stir for about 10 min at high speed. Pour the resulting slurry slowly through the sieve and wash out the mixing cup carefully (a polyethylene squeeze-type wash bottle is very convenient) to ensure complete transfer of the specimen to the sieve. Spray the sieve surface gently with a low pressure fan-shaped spray (tap water at about 5 psig (30 kPa) back pressure) and continue washing until all the pigment has been washed through and the water passing the sieve is clear. Dry the sieve for 1 h at 105 to 110°C, cool and weigh.

12.2 Calculation:

$$\text{Percent of coarse particles} = (\text{increase in weight of sieve/specimen weight}) \times 100 \tag{3}$$

MATTER SOLUBLE IN WATER

13. Procedure

13.1 Determine the matter soluble in water by Test Method D 2448.

COLOR

14. Standard Pigment

14.1 *Standard Extender Pigment*.

14.2 *Standard Zinc Oxide*.

15. Procedure

15.1 Carefully weigh out the proportion 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 to present them on an even plane, and judge the color immediately.

PRECISION AND BIAS

16. Precision and Bias

16.1 Data are not available to determine the precision and bias of these methods. There are no plans at present to obtain such information. The methods have been in use for several years and are considered acceptable.

17. Keywords

17.1 aluminum oxide in aluminum silicate pigment; aluminum silicate pigment, analysis of; pigment, aluminum silicate