Designation: ~~D717 – 86 (Reapproved 2008)~~ D717 – 86 (Reapproved 2014)

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

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 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:*[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D234 Specification for Raw Linseed Oil (Withdrawn 2007)[3]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
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of Test for Directional Reflectance Factor, 45-Deg 0-Deg, of Opaque Specimens by Broad-Band Filter Reflectometry (Withdrawn 1991)[3]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 SiO_2, 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.

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[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~~standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] *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.

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* (Na_2CO_3).

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:

$$SiO_2, \% = (P/S) \times 100 \quad (1)$$

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*—($K_2S_2O_7$).

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 $K_2S_2O_7$ 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.

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.