



Designation: D 4487 – 90 (Reapproved 2003)

Standard Test Methods for Analysis of Calcium Borosilicate¹

This standard is issued under the fixed designation D 4487; 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 the pigment commercially known as calcium borosilicate.

1.2 The test methods appear in the following order:

Test Methods	Sections
Silicon Dioxide (SiO ₂)	6-9
Iron Oxide (Fe ₂ O ₃)	10-13
Boron Trioxide (B ₂ O ₃)	17-20
Calcium Oxide (CaO)	21-23
Moisture and Volatile Matter	24
Water of Hydration	25 to 26
Coarse Particles	27
Oil Absorption	28

1.3 Individual specimens may be used for the direct determinations of SiO₂, B₂O₃, and CaO. SiO₂ and Fe₂O₃ should be removed before the determination of the B₂O₃ and CaO.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- D 185 Test Methods for Coarse Particles in Pigments, Pastes, and Paints
- D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments
- D 281 Test Method for Oil Absorption of Pigments by Spatula Rub-Out
- D 1193 Specification for Reagent Water

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.

Current edition approved Dec. 1, 2003. Published December 2003. Originally approved in 1985. Last previous edition approved in 1990 as D 4487 – 90 (1999).

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. Significance and Use

3.1 These test methods compile in one place, recommended procedures for analysis of the pigment known commercially as calcium borosilicate. This pigment is used extensively in paints and the composition is important to the user and producer.

4. Reagents

4.1 *Purity of Reagents*—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.³ 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

4.3 *Concentration of Acids and Ammonium Hydroxide*—When acids and ammonium hydroxide are specified by name or chemical formula only, it should be understood that concentrated reagents of the following specific gravity are intended:

Hydrochloric acid (HCl)	1.19
Nitric acid, (HNO ₃)	1.42
Sulfuric acid (H ₂ SO ₄)	1.84
Ammonium hydroxide (NH ₄ OH)	0.90

5. Preparation of Samples

5.1 Thoroughly mix and comminute the sample before taking portions for analysis.

SILICON DIOXIDE

6. Apparatus

- 6.1 *Evaporating Casserole*, 250-mL capacity.
- 6.2 *Hot Plate*.

³ *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.

6.3 *Porcelain Filtering Crucible*, medium porosity, 30-mL capacity.

6.4 *Drying Oven*, maintained at $100 \pm 5^\circ\text{C}$.

6.5 *Muffle Furnace*.

7. Reagents

7.1 *Hydrochloric Acid* (1+1).

7.2 *Hydrochloric Acid* (1+19).

7.3 *Hydrochloric Acid* (1+99).

8. Procedure

8.1 Introduce a 1-g specimen, weighed to the nearest 0.1 mg into an evaporating casserole. Add 50 mL of HCl (1+1) and thoroughly mix.

8.2 Place the casserole on a hot plate and evaporate carefully to dryness.

8.3 Place the casserole in the oven at 100°C for 2 h. Do not allow the oven temperature to exceed 105°C at any time. Remove the casserole and allow to cool for 10 min.

8.4 Completely wet the residue with 25 mL of HCl (1+1) and cover the casserole with a watch glass. Warm just to boiling on a hot plate and maintain for 10 min.

8.5 Add 25 mL of water, free any material from the sides of the casserole with a stirring rod, and immediately filter through a tared porcelain crucible of medium porosity.

8.6 Wash the residue with two 5-mL portions of hot HCl (1+19), one 5-mL portion of hot HCl (1+99), and finally with two 5-mL portions of hot water. Save the combined filtrates for the determination of iron oxide (Sections 10-12).

8.7 Place the crucible containing the precipitate in the muffle furnace from 600 to 800°C and heat to constant weight (± 4 mg). Cool in a desiccator and weigh.

9. Calculation

9.1 Calculate the percent of SiO_2 , A , as follows:

$$A = R/S_1 (100) \quad (1)$$

where:

R = weight of residue, g, and

S_1 = weight of original specimen, g.

IRON OXIDE

10. Apparatus

10.1 *Volumetric Flasks*, 250-mL and 1000-mL capacity.

10.2 *Buret*, 10-mL capacity.

11. Reagents

11.1 *Potassium Iodate* (0.01878 N)—Dry 1.0 g of KIO_3 at 120°C for 2 h in a drying oven. After cooling, weigh 0.6700 g and dissolve it in 100 mL of water. Dilute the solution to 1 L in a volumetric flask. 1 mL = 0.001500 g Fe_2O_3 .

11.2 *Potassium Iodide* (KI)—Iodate free.

11.3 *Starch Indicator Solution*—Make a homogeneous paste of 10 g of soluble starch in cold water. Add to this 1 L of boiling water, stir rapidly, and cool. Salicylic acid (1.25 g/L) may be added to preserve the indicator. If long storage is required, the solution should be kept in a refrigerator at 4 to

10°C . Prepare fresh indicator when the end point of the titration from blue to colorless fails to be sharp.

11.4 *Sulfuric acid* (H_2SO_4) (1+18).

11.5 *Sodium Thiosulfate, Standard Solution* (0.025 N)—Dissolve 1.5 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 50 mL of water and dilute to 250 mL. Standardize as follows: Pipet 10 mL of the KIO_3 solution into each of three 150-mL beakers. Dilute each to 100 mL with water, add 2 g of KI and 5 mL of H_2SO_4 (1+18), and dissolve the KI with stirring. Titrate the liberated iodine with 0.025 N $\text{Na}_2\text{S}_2\text{O}_3$ solution until the color of the solution becomes pale yellow. Add 2 mL of starch indicator and continue the titration dropwise until the color changes from blue to colorless.

$$I = 1 \text{ mL } \text{Na}_2\text{S}_2\text{O}_3 = 0.01500/V_1 \text{ g } \text{Fe}_2\text{O}_3 \quad (2)$$

where:

I = iron oxide equivalent of $\text{Na}_2\text{S}_2\text{O}_3$ solution,

V_1 = $\text{Na}_2\text{S}_2\text{O}_3$ required for titrations, mean, mL, and

0.01500 = $(10.00 \text{ mL } \text{KIO}_3) \times (0.001500 \text{ g } \text{Fe}_2\text{O}_3/\text{mL } \text{KIO}_3)$.

12. Procedure

12.1 Dilute the solution obtained from the procedure in 8.6 to 100 mL with water. Add 10 mL of HCl and 5 g KI. Dissolve the KI with stirring.

12.2 Titrate with 0.025 N $\text{Na}_2\text{S}_2\text{O}_3$ solution until the color becomes a pale yellow. Add 2 mL of starch indicator solution and continue the titration until the color changes from blue to colorless.

13. Calculation

13.1 Calculate the percent of iron oxide, D , as follows:

$$D = (I \times V_2)/S_1 (100) \quad (3)$$

where:

V_2 = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration, mL, and

S_1 = weight of original specimen, g.

SOLUTION OF PIGMENT FOR THE DETERMINATION OF BORON TRIOXIDE AND CALCIUM OXIDE

14. Apparatus

14.1 *Boiling Flask*—300-mL capacity with ground glass connection.

14.2 *Büchner Funnel*, 56-mm diameter.

14.3 *Filter Paper*, 55-mm diameter.⁴

14.4 *Filter Flask*, 250-mL capacity.

14.5 *Hot Plate/Stirrer*.

14.6 *pH Meter*.

14.7 *Reflux Condenser*, with ground glass connection, water cooled.

⁴ The sole source of supply of Whatman No. 50 Filter Paper, known to the committee at this time is Whatman, Inc. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.