



Designation: D6845 – 22

# Standard Test Method for Silica, Precipitated, Hydrated—CTAB (Cetyltrimethylammonium Bromide) Surface Area<sup>1</sup>

This standard is issued under the fixed designation D6845; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the measurement of the specific surface area of precipitated silicas exclusive of area contained in micropores too small to admit hexadecyltrimethylammonium bromide (cetyltrimethylammonium bromide, commonly referred to as CTAB) molecules. This test method is suitable for characterizing rubber-grade silicas of all types.

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:*<sup>2</sup>

[D1193 Specification for Reagent Water](#)

[D1799 Practice for Carbon Black—Sampling Packaged Shipments](#)

[D1900 Practice for Carbon Black—Sampling Bulk Shipments](#)

[D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries](#)

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

Current edition approved Nov. 1, 2022. Published November 2022. Originally approved in 2002. Last previous edition approved in 2020 as D6845 – 20. DOI: 10.1520/D6845-22.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 3. Summary of Test Method

3.1 The isotherm for adsorption of an aqueous solution of CTAB on silicas has a long horizontal plateau corresponding to a monolayer coverage of the substrate surface from which the adsorbate is not sterically excluded. The CTAB adsorption by silica is independent of functional groups containing hydrogen and oxygen, etc. Rapid equilibration is achieved by using mechanical stirring. Titration with dioctyl sodium sulfosuccinate (Aerosol OT<sup>3</sup>) solution to a turbidity maximum end point is used to determine the unadsorbed CTAB after removal of the colloiddally dispersed silica by ultrafiltration.

3.2 Titration of the unadsorbed CTAB with Aerosol OT solution is accomplished by automatic titration.

## 4. Significance and Use

4.1 The CTAB molecule is relatively large; so it is not adsorbed in micropores or on surface roughness. Thus, the CTAB surface area reflects only the surface of the silica that is available for interaction with rubber molecules.

## 5. Apparatus

5.1 *Analytical Balance*, 0.1 mg sensitivity.

5.2 *Centrifuge*, capable of 67 rev/s (4000 r/m).

5.3 *Magnetic Spinbars*,<sup>4</sup> chemically resistant covered (polychlorotrifluoroethylene or TFE-fluorocarbon), 6.4- or 4.8-mm ( $\frac{1}{4}$ - or  $\frac{3}{16}$ -in.) diameter, and length nearly equal to the diameter of 50 or 100 cm<sup>3</sup> Berzelius beakers, glass vials, or other glass vessels.

5.4 *Glass Funnel*, small.

5.5 *Glass Vials*, with plastic screw caps, 28 mm in outside diameter, about 40 cm<sup>3</sup> capacity.

5.6 *Buret*, 50 cm<sup>3</sup>, 0.1 cm<sup>3</sup> divisions, Class A, preferably of automatic refilling and zeroing type (see **Note 1**) with reagent reservoir.

<sup>3</sup> Aerosol OT is a registered trademark of the American Cyanamid Co., Process Chemicals Dept., Wayne, NM 07470.

<sup>4</sup> Examples of suitable spinbars are Catalog No. 9235-U7, A. H. Thomas Co., Philadelphia, PA 19105, or Catalog No. S-76497-30, Sargent Welch Scientific Co., Skokie, IL 60076.

NOTE 1—Automatic burets are usually not certified to Class A tolerance. Such burets should be checked for accuracy, and if in error by more than 0.05 cm<sup>3</sup> at any point, a calibration curve should be prepared and used to correct observed buret readings. Burets with TFE-fluorocarbon manostat valves offer some advantage in ease of stopcock manipulation in delivering small increments of titrant.

5.7 *Dispenser-Type Pipet*,<sup>5</sup> 50 cm<sup>3</sup>, attached to a suitable reservoir for CTAB solution.

5.8 *Pipet*, 10 cm<sup>3</sup>, Class A.

5.9 *Erlenmeyer Flasks*, or beakers, 50 or 100 cm<sup>3</sup>.

5.10 *Dropping Bottle*, 60 cm<sup>3</sup> amber.

5.11 *Jar*, wide-mouth, plastic screw cap, 120 or 240 cm<sup>3</sup> (4- or 8-oz) size.

5.12 *Gravity Convection Drying Oven*, capable of maintaining 105 ± 5°C.

5.13 *Magnetic Stirrer*.

5.14 *Containers*, suitable for preparation and storage of reagent solutions.

5.15 *Automatic Titration Equipment with Photometric Sensor*,<sup>6</sup> with instruction manual.

5.16 *Beakers*, 100 cm<sup>3</sup> Berzelius, tall form (one furnished with automatic titration equipment).

5.17 *Thermometer*, to measure temperature in a range from 20 to 40°C.

5.18 *Polyethylene Tubing* (do not use vinyl tubing).

5.19 *Wire mesh sieve*, 75 μm (200 mesh).

NOTE 2—All apparatus must be kept chemically clean. Contamination of equipment, water supply, or chemicals by ionic surfactants must be particularly avoided.

## 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>7</sup> 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 in Specification **D1193**.

6.3 *Sodium Hydroxide Solution (1M)*—Dissolve 40.0 g NaOH in a 1 dm<sup>3</sup> flask containing 0.5 dm<sup>3</sup> reagent water. After dissolution fill to mark.

<sup>5</sup> This test is based on the use of the Universal Repipet, a registered trademark of Labindustries, 1802 Second St., Berkeley, CA 94710.

<sup>6</sup> This test is based on the use of automatic titration equipment with photometric sensor. Appropriate devices are commercially available.

<sup>7</sup> *Reagent Chemicals, American Chemical Society Specifications*, Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagent Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia*.

6.4 *Buffer Solution of pH 9.6 (0.05 M)*—Prepare buffer solution by dissolving 3.101 g of orthoboric acid (H<sub>3</sub>BO<sub>3</sub>), 3.708 g of potassium chloride (KCl) and 36.85 cm<sup>3</sup> of sodium hydroxide (NaOH) solution in a 1 dm<sup>3</sup> flask containing 0.5 dm<sup>3</sup> reagent water. After dissolution fill to mark. Verify that the pH of the solution is 9.6 ± 0.1.

6.5 *CTAB Standard Solution*—Dissolve 5.50 g of reagent-grade CTAB<sup>8</sup> in a 1 dm<sup>3</sup> flask containing 350 cm<sup>3</sup> of buffer solution and 500 cm<sup>3</sup> water. After dissolution fill to mark. Homogenize on a magnetic stirrer for 10 h. Allow to stand 24 h before use. Verify that the pH of the solution is 9.6 ± 0.1 before use. If not, adjust the pH with either 0.1 N NaOH solution (too low) or 0.1 N HCl solution (too high). (**Warning**— Storage of the solution below 22°C will result in slow crystallization of the CTAB.)

6.6 *Aerosol OT<sup>9</sup> Solution 100 % Grade* (approximately 0.00389 M)—Dissolve 1.73 g/dm<sup>3</sup> of Aerosol OT solution (100 % solids) in 500 cm<sup>3</sup> distilled or deionized water. Stir vigorously with a magnetic stirrer for 10 h. Allow to stand an additional twelve days before standardization and use. The solution should be capped tightly and stored in a cool place. (Once opened, 100 % Aerosol OT should be stored in a desiccator.)

6.7 *Distilled or Deionized Water*.

## 7. Sampling

7.1 No separate practice for sampling silicas is available. However, samples may be taken in accordance with Practices **D1799** or **D1900**.

## 8. Standardization of Reagents

8.1 Duplicate blanks are determined and averaged.

8.2 Pipet 5 cm<sup>3</sup> of CTAB solution into a beaker.

8.3 Add 50 cm<sup>3</sup> of distilled or deionized water.

8.4 Titrate with Aerosol OT using automatic titrator until endpoint is reached.

8.5 Use average of both repetitions when calculating CTAB of silica sample in Section 10.

## 9. Procedure

9.1 Dry an adequate portion of sample of silica at 105°C for 2 h and cool in a desiccator.

NOTE 3—If the heating loss of the silica is known, the drying step may be omitted and the observed sample mass corrected for heating loss to obtain the mass of dry sample, *W*.

9.2 Grind an appropriate quantity of silica sample in a laboratory grinder for 60 s.

9.3 Pass ground sample through a wire mesh sieve 75 μm (200 mesh). This can be accomplished manually or mechanically. Discard fraction retained on sieve. The fraction <75 μm (<200 mesh) is used for the determination of the CTAB surface area.

<sup>8</sup> Available from J. T. Baker Chemical Co., 222 Red School Lane, Phillipsburgh, NJ 08865, Catalog No. 7N121.

<sup>9</sup> Available from American Cyanamid Co., Process Chemicals Dept., Wayne, NM 07470. Also available as Catalog No. A-349 from Fisher Scientific Co.