



Standard Test Method for Precipitated Silica-Surface Area by Multipoint BET Nitrogen Adsorption¹

This standard is issued under the fixed designation D 1993; 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 This test method covers a procedure which is used to measure the surface area of precipitated hydrated silicas by the conventional Brunauer, Emmett, and Teller (BET)² theory of multilayer gas adsorption behavior using multipoint determinations, similar to that used for carbon black in Test Methods D 4820. This test method specifies the sample preparation and treatment, instrument calibrations, required accuracy and precision of experimental data, and calculations of the surface area results from the obtained data.

1.2 This test method is used to determine the nitrogen surface area of precipitated silicas with specific surface areas in the range of 1 to 50 hm^2/kg (10 to 500 m^2/g).

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

1.4 *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.* The minimum safety equipment should include protective gloves, sturdy eye and face protection, and means to deal safely with accidental mercury spills.

2. Referenced Documents

2.1 ASTM Standards:

D 1799 Practice for Carbon Black—Sampling Packaged Shipments³

D 1900 Practice for Carbon Black—Sampling Bulk Shipments³

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries³

D 4820 Test Methods for Carbon Black—Surface Area by

Multipoint B.E.T. Nitrogen Adsorption⁴

3. Significance and Use

3.1 This test method is used to measure the surface area of precipitated, hydrated silicas that is available to the nitrogen molecule using the multipoint (B. E. T.) method.

3.2 Solids adsorb nitrogen and, under specific conditions, the adsorbed molecules approach a monomolecular layer. The quantity in this hypothetical monomolecular layer is calculated using the BET equation. Combining this with the area occupied by the nitrogen molecule yields the total surface area of the solid.

3.3 This test method measures the estimated quantity of nitrogen in the monomolecular layer by adsorption at liquid nitrogen temperature and at several (at least five) partial pressures of nitrogen.

3.4 Before a surface area determination can be made it is necessary that the silica be stripped of any material which may already be adsorbed on the surface. The stripping of adsorbed foreign material eliminates two potential errors. The first error is associated with the weight of the foreign material. The second error is associated with the surface area that the foreign material occupies.

4. Apparatus

4.1 Commercial instruments are available⁵ for the measurement of nitrogen surface area by the multipoint BET method. These may be of the “flowing gas” or the “vacuum-volumetric” type.

NOTE 1—Automated instruments will provide results equivalent to the procedure described herein if careful calibration of the instrument, equivalent sample preparation, adherence to manufacturer’s instruction for instrument operation, and equivalent data handling and calculations are performed.

4.2 *Sample Cells*, and other peripheral equipment as recommended by the manufacturer for the instrument used.

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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² Brunauer, Emmett, and Teller, *Journal of the American Chemical Society*, Vol. 60, 1938, p. 309.

³ *Annual Book of ASTM Standards*, Vol. 09.01.

⁴ Discontinued—see 1999 *Annual Book of ASTM Standards*, Vol. 09.01.

⁵ Commercial automated instruments found satisfactory may be obtained from Micromeritics Instrument Corporation, One Micromeritics Drive, Norcross, GA 30093-1877, website: www.micromeritics.com, and Quantachrome Instruments, 1900 Corporate Drive, Boynton Beach, FL 33426, website: www.quantachrome.com.