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.

Designation: D5604 – 96 (Reapproved 2017)

Standard Test Methods for Precipitated Silica—Surface Area by Single Point B.E.T. Nitrogen Adsorption[1]

This standard is issued under the fixed designation D5604; 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 a procedure to measure the surface area of precipitated hydrated silicas by, a single point approximation of the Brunauer, Emmett, and Teller (B.E.T.)[2] theory of multilayer gas adsorption. These test methods specify 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 These test methods are used to determine the single point nitrogen surface areas in the range of 10 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 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.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D1993 Test Method for Precipitated Silica-Surface Area by Multipoint BET Nitrogen Adsorption

3. Summary of Test Methods

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

3.2 These test methods measure the estimated quantity of nitrogen in the monomolecular layer formed by adsorption at liquid nitrogen temperature and at a fractional saturation pressure of 0.30 ± 0.01.

3.3 Before a surface area determination can be made it is necessary that any material which may already be adsorbed on the surface of the silica be removed. Removal of adsorbed foreign material (by heating under vacuum or in a steam of non-adsorbing gas) eliminates two potential errors. The first error is due to the mass of the foreign material. The second error is due to interference by the foreign material to access by nitrogen the silica surface.

4. Significance and Use

4.1 These test methods measure the approximate surface area of precipitated hydrated silicas that is available to the nitrogen molecule using an approximation of the B.E.T. method. While the multi-point version of the B.E.T. method is generally accepted as being less prone to errors arising from the varying surface properties of individual samples, the single-point approximation is often adequate due to the shorter time per test and relative simplicity of the instrumentation needed. Quality control applications and comparative tests on

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and are 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] 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.

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