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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 D 5604; 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:*

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[3]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[3]

D 1993 Test Method for Precipitated Silica—Surface Area by Multipoint BET Nitrogen Adsorption[3]

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 near-identical samples of close chemical and micro-structural composition are likely to be the applications of greatest value.

5. ASTM D11 Standard Reference Silicas

5.1 *None Required*—This test method is used to determine surface area of candidate silicas. Reference silicas are available[4] for determining agreement with data obtained in the interlaboratory test used for multi-point procedure Test Method D 1993.

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[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] Precipitated samples are available from Forcoven Products, P.O. Box 1556, Humble, TX 77338. Samples are available in three surface areas: A138; B.57; and C.168 X10^3 m^2/kg.

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TEST METHOD A — SURFACE AREA BY STATIC VOLUMETRIC APPARATUS

6. Apparatus

6.1 *Static-Volumetric Gas Adsorption Apparatus*, with dewar flasks and all other accessories required for operation.

6.2 *Oven*, vacuum-type, capable of temperature-regulation to ±5°C at 110°C. Pressure should be less than 13.5 Pa (0.1 mmHg).

6.3 *Sample Cells*, which, when attached to the adsorption apparatus, will maintain isolation of the sample from the atmospheric pressure equivalent to a helium leak rate of $\leq 10^{-5}$ standard cubic centimeters per minute, per atmosphere of pressure difference.

6.4 *McCleod Gage*, or equivalent means to measure the pressure. (May be part of the adsorption apparatus.)

6.5 *Pressure Gage or Transducer*, known to be accurate to ±0.25 % of reading or ±0.067 kPa (±0.5 mmHg), whichever is greater and covering the 0 to 101.3 kPa (760 mmHg) pressure range. (May be part of the adsorption apparatus.)

6.6 *Analytical Balance*, with 0.1 mg sensitivity.

6.7 *Glass Vials*, small (30 cm^3) glass vials with caps for oven drying samples.

6.8 *Heating Mantle*, or equivalent, capable of maintaining a temperature of 160 ± 5°C.

6.9 *Volumetric Calibration Apparatus*, with valve or stopcock and 6.4 mm tubing adapter to gas adsorption sample connector. See Fig. 1.

7. Reagents

7.1 *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.[5] Other grades may be used, provided it is established that they are of sufficiently high purity to use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water (and ice prepared from it) shall be understood to mean distilled water or water of equal purity.

7.3 *Liquid Nitrogen*, 98 % or higher purity.

7.4 *Ultra-High Purity Nitrogen Gas*, cylinder, or other source of prepurified nitrogen gas.

7.5 *Ultra-High Purity Helium Gas*, cylinder, or other source of prepurified helium gas.

8. Sampling

8.1 No separate practice for sampling silicas is available. However, samples may be taken in accordance with Practices D 1799 or D 1900, whichever is appropriate.

[5] *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.

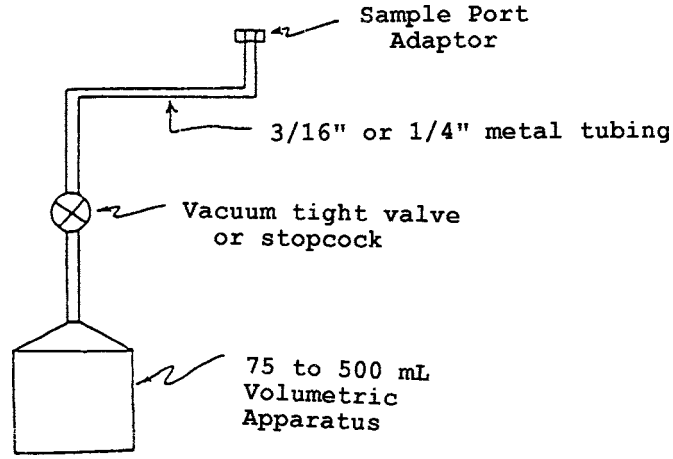

FIG. 1 Volumetric Apparatus

9. Preparation and Verification of Calibration of Static-Volumetric Apparatus

NOTE 1—Perform this procedure for initial calibration, periodically for quality control, and following repairs or adjustments. If a commercial apparatus is used, consult the user's manual for specific instructions in carrying out the following steps.

9.1 Attach the very low and atmospheric pressure gages or transducers (see 6.4 and 6.5) to the apparatus and evacuate it, the manifold, and all internal pressure/vacuum sensors to 2.7 Pa (20μ mHg) or below.

9.2 Verify that the internal vacuum sensor(s) are reading correctly and that the internal pressure sensor(s) are reading correctly in the vicinity of zero pressure subject to the expected resolution and stability limits. Make adjustments as needed.

9.3 Close the vacuum path and admit nitrogen gas to build 101.3 kPa ± 1 % (760 mmHg ± 7.6 mmHg) of pressure. Verify that the pressure sensors read the correct pressure to within ±0.25 %. Make adjustments as needed.

9.4 Thoroughly clean and dry an empty sample holder. Attach it to the apparatus and evacuate it to 2.7 Pa (20 μmHg). Apply a 160°C heating mantle and continue evacuation for at least 1 h and until the rate of pressure rise upon temporarily closing off the vacuum path is under .4 Pa (3 μmHg) per minute.

9.5 Perform a sample analysis on this clean empty sample tube at 0.30 ± 0.01 P/P_o. Use a P_o of 101.3 kPa (760 mmHg) and a sample mass of 1 g.

9.6 Examine the volume adsorbed quantity obtained. Ideally it should be zero. An error amount exceeding 0.25 standard cubic centimeters is unacceptable and requires correction. An error amount of 0.125 standard cubic centimeter or less is acceptable.

9.7 Obtain a cylindrical or spherical calibration volume made of glass or corrosion resistant metal and having an internal volume between 75 cm^3 and 500 cm^3. It must have a tubing connection and an in-line valve or stopcock as shown in Fig. 1.

9.8 Determine the internal volume below the valve or stopcock by the mass difference when first empty and then when filled completely with distilled water. Measure the water temperature and correct for the water density to obtain the

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