Designation: ~~D6556 – 19~~ D6556 – 19a

Standard Test Method for Carbon Black—Total and External Surface Area by Nitrogen Adsorption[1]

This standard is issued under the fixed designation D6556; 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 the determination of the total surface area by the Brunauer, Emmett, and Teller (B.E.T. NSA) theory of multilayer gas adsorption behavior using multipoint determinations and the external surface area based on the statistical thickness surface area method.

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.* (The minimum safety equipment should include protective gloves, sturdy eye and face protection).

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:*[2]

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D3765 Test Method for Carbon Black—CTAB (Cetyltrimethylammonium Bromide) Surface Area (Withdrawn 2007)[3]

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 The total and external surface areas are measured by evaluating the amount of nitrogen adsorbed, at liquid nitrogen temperature, by a carbon black at several partial pressures of nitrogen. The adsorption data is used to calculate the NSA and STSA values.

4. Significance and Use

4.1 This test method is used to measure the total and external surface area of carbon blacks based on multipoint nitrogen adsorption. The NSA measurement is based on the B.E.T. theory and it includes the total surface area, inclusive of micropores, pore diameters less than 2 nm (20 Å). The external surface area, based on the statistical thickness method (STSA), is defined as the specific surface area that is accessible to rubber.

4.2 CTAB Surface Area (formerly Test Method D3765) has been withdrawn. The CTAB value may be estimated from the STSA value using Eq 1. The equation is based on a linear regression of the STSA and CTAB measured values of the SRB 5 standards.

$$CTAB = STSA * 1.0170 + 2.6434 \quad (1)$$

[1] This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.21 on Carbon Black Surface Area and Related Properties.

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[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] The last approved version of this historical standard is referenced on www.astm.org.

5. Apparatus

5.1 *Multipoint Static-Volumetric Gas Adsorption Apparatus,* with Dewar flasks and all other accessories required for operation.

5.2 *Sample Cells,* that when attached to the adsorption apparatus, will maintain isolation of the sample from the atmosphere equivalent to a helium leak rate of $<10^{-5}$ cm^3/min, per atmosphere of pressure difference.

5.3 *Balance, Analytical,* with 0.1 mg sensitivity.

5.4 *Heating Mantle or Equivalent,* capable of maintaining a temperature of 300 ± 10°C.

5.5 *Oven, Gravity Convection,* capable of maintaining a temperature of 125 ± 10°C.

6. Reagents

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

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

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

7. Sampling

7.1 Samples may be taken in accordance with Practices D1799 and D1900.

8. Sample Preparation Procedure

8.1 Dry a portion of carbon black at 125°C for 1 h. If the carbon black is known to be substantially free of moisture, or subsequent preparation steps are known to be adequate for moisture removal, then this step may be omitted.

8.2 Condition an empty sample cell for a minimum of 10 min at the same conditions intended for degassing the sample. Weigh the empty sample cell to the nearest 0.1 mg and record the mass.

8.3 Weigh approximately 0.4 g of the carbon black into the sample cell.

NOTE 1—For carbon black powder samples, add enough carbon black to give a depth of approximately 2 in. in straight wall sample tubes, or approximately 0.4 g for bulb-type sample cells.

8.4 *Flow Degassing:*

8.4.1 Open the gas control valve and insert the delivery tube into the sample tube, through the sample bed, and allow purging with ~~either helium or~~ nitrogen for a minimum of 1 min.

8.4.2 Place a heating mantle or other source of heat around the sample cell and degas the sample at 300 ± 10°C for ½ h or longer to ensure that all traces of moisture condensing in the top of the tube are absent. The minimum degassing time that gives a stable surface area (that is, a surface area that does not increase with additional degassing) ~~may be~~ shall be the minimum time used for degassing. Failure to do so will lead to underreporting of the NSA and STSA values for the sample under test.

NOTE 2—For carbon blacks at their moisture pickup equilibrium, like standard reference ~~blacks,~~ blacks that have been exposed to atmospheric conditions, longer degassing times likely will be required in order to achieve stable results. It has been found that for ~~SRB B8 and SRB C8,~~ SRB-8B, SRB-8C, and SRB-9C, it is necessary to extend the degassing time to at least 60 min in order to reliably obtain the target NSA and STSA values. This may be true for other high structure and high porosity carbon blacks. Again, failure to fully degas the carbon black will result in underreporting of NSA and STSA values.

8.4.3 Once the typical degassing times have been determined, future samples can be degassed on the basis of time alone, if desired, allowing a reasonable margin of excess time. Some samples will be found to require less than ½ h, especially if moisture exposure has been minimal. In these cases, the minimum time that gives a stable surface area ~~may be~~ shall be the minimum time used for degassing. It will be necessary to determine these minimum times for each grade of carbon black.

8.4.4 After degassing, the sample tube may be moved directly to the analyzer. Otherwise, remove the sample tube from the heat source and continue the flow of purging gas until it is ready for analysis.

8.4.5 Go directly to Section 9 and continue the remaining steps of the procedure.

8.5 *Vacuum Degassing:*

8.5.1 With the apparatus at atmospheric pressure, place the sample cell containing the carbon black onto the degassing apparatus.

8.5.2 Begin the degassing procedure as appropriate for the apparatus.

8.5.3 Place a heating mantle or other source of heat around the sample cell and degas the sample at 300 ± 10°C for ½ h or longer as required to obtain and hold a pressure less than 1.4 Pa (10 μm Hg).

NOTE 3—Attention! One-half hour vacuum degassing may be inadequate for some grades and may result in statistically different results to flow degassing.

8.5.4 Once the typical degassing times have been determined, future samples can be degassed on the basis of time alone, if desired, allowing a reasonable margin of excess time. Some samples will be found to require less than ½ h, especially if moisture