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Designation: D 6556 – 00a

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

This standard is issued under the fixed designation D 6556; 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 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[2]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[2]

D 3324 Practice for Carbon Black—Improving Test Reproducibility Using ASTM Reference Blacks[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

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 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.

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 Practice D 1799 and Practice D 1900.

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 minutes 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

[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 Adsorptive Properties of Carbon Black.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

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