Designation: D 5816 – 99

Standard Test Methods for Carbon Black—External Surface Area by Multipoint Nitrogen Adsorption[1]

This standard is issued under the fixed designation D 5816; 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 the determination of the external surface area of carbon blacks by the statistical thickness surface area (STSA) 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.*

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 4820 Test Methods for Carbon Black—Surface Area by Multipoint B.E.T. Nitrogen Adsorption[2]

3. Significance and Use

3.1 These test methods are used to measure the external surface area of carbon black. These techniques are based on the statistical thickness method using a carbon black model. The external surface area is defined as the specific surface area that is accessible to rubber. The external surface area excludes the micropore area, pore diameters less than 20 Å.

4. Apparatus

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

4.2 *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.

4.3 *Analytical Balance*, with 0.1 mg sensitivity.

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

4.5 *Oven, Gravity Convection*, capable of maintaining a temperature of 125 ± 5°C.

5. Reagents

5.1 *Liquid nitrogen*, 98 % or higher purity.

5.2 *Ultra high-purity nitrogen gas*, cylinder, or other source of prepurified nitrogen gas.

5.3 *Ultra high-purity helium gas*, cylinder, or other source of prepurified helium gas.

6. Sampling

6.1 Samples shall be taken in accordance with Practice D 1799 and Practice D 1900.

7. Sample Preparation Procedure

7.1 Dry a portion of the carbon black to be tested 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.

7.2 Weigh a sample cell to the nearest 0.1 mg and record the mass.

7.3 Weigh into the cell approximately 0.4 g of the carbon black to be tested.

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 fill the sample bulb for bulb-type sample cells.

7.4 *Flow Degassing*:

7.4.1 Open the helium control valve and insert the delivery tube into the sample tube, and allow to purge for a minimum of 1 min.

7.4.2 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.

7.4.3 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 used for degassing.

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[1] These test methods are under the jurisdiction of ASTM Committee D-24 on Carbon Black and are 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.