AMERICAN NATIONAL STANDARD
ANSI/ASTM C 819 - 77

Standard Test Method for SPECIFIC SURFACE AREA OF CARBON OR GRAPHITE[1]

This Standard is issued under the fixed designation C 819; 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.

1. Scope

1.1 This method covers the determination of the specific surface area of carbon or graphite by the Brunauer, Emmett, and Teller (BET)[2] method. The BET method is based on the adsorption of a monolayer of nitrogen gas at its normal boiling point (−195.8°C) on the carbon or graphite surface.

1.2 This method is intended for the determination of the specific surface areas of solid bodies, but can be applied to granular specimens.

2. Summary of Method

2.1 A known amount of nitrogen gas is introduced into a measured volume containing a specimen of known weight which has been cooled to liquid nitrogen temperature.

2.2 A monolayer of nitrogen atoms is adsorbed on the surfaces of the sample and the amount of nonadsorbed gas is calculated from pressure-volume relationships.

2.3 The specific surface area is calculated in square metres per gram of specimen from the amount of nitrogen adsorbed and the surface area occupied by each nitrogen molecule.

3. Significance

3.1 The specific surface area that is accessible to gas molecules is an important parameter for evaluating the oxidation rate of a carbon or graphite body.

3.2 The BET method provides a direct and reproducible means of measuring the accessible surface area.

4. Apparatus

4.1 Apparatus for specific surface area measurements can be constructed from materials readily available in a typical laboratory.

4.2 Figure 1 is a schematic drawing of a BET surface area apparatus. All piping, stopcocks, and storage bulbs are made of glass.

4.3 Equivalent apparatus is also available from commercial sources.

5. Calibration

5.1 The volume of each chamber of the mercury buret must be determined accurately prior to assembly of the apparatus. This can be accomplished by weighing the empty buret, filling a buret volume with mercury, and reweighing.

6. Procedure

6.1 Where commercial apparatus is used, follow the detailed procedure established by the manufacturer.

6.2 *Preparation:*

6.2.1 Outgas the system and sample at 10^{-5} torr (1.33 mPa) or less with the specimen at a temperature of about 375 ± 25°C.

NOTE 1—Completion of outgassing can be detected by isolating the specimen chamber and observing the rate of pressure increase.

6.2.2 With S_1 through S_5 and S_9 closed, fill the helium and nitrogen storage bulbs to

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[1] This method is under the jurisdiction of ASTM Committee C-5 on Manufactured Carbon and Graphite Products, and is the direct responsibility of Subcommittee C05.05 on Nuclear Applications.

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[2] Brunauer, S., Emmeth, P. H., and Teller, E., "Adsorption of Gases in Multimolecular Layers," *Journal of the American Chemical Society*, JACSA, Vol 60, 1938, pp. 309-319.

slightly above atmospheric pressure. Remove oxygen from the helium and nitrogen by passing the gas over copper wire in the furnace at 350°C; water is condensed in the water traps which are cooled with liquid nitrogen. Trace impurities, except the inert gases, are removed from the helium by the charcoal absorber which is cooled by liquid nitrogen.

6.3 *Measurement of Line Volume*—The line volume is that volume between the zero reference of the buret, the zero reference of the manometer (M_1), and S_4 through S_7.

6.3.1 With S_4, S_6, and S_7 closed, slowly leak helium into this volume from the helium storage bulb.

6.3.2 Adjust the mercury level in M_1 to zero reference and record the gas pressure, P_1.

6.3.3 Expand the gas by lowering the mercury level in the gas buret and adjust the mercury level in M_1 to zero reference.

6.3.4 Record the buret volume, V_{B1}, the gas pressure, P_2, the buret temperature, T_{B1}, and the room temperature, T_{R1}.

6.4 *Measurement of Sample Chamber Volume*—The free volume is the sum of the line volume plus the volume in the sample tube, which includes the space contained in the pores of the sample open to helium.

6.4.1 With the sample tube immersed in liquid nitrogen, return the mercury level of the buret to zero reference and slowly open S_6.

6.4.2 Determine the liquid nitrogen temperature by the following procedure:

6.4.2.1 With S_8 turned to vacuum, slowly bleed nitrogen through S_1 until the compression chamber is emptied of mercury.

6.4.2.2 Open S_9 and, controlling the flow rate at the tank, slowly bubble nitrogen through the liquid nitrogen in the Dewar flask.

6.4.2.3 Slowly open S_8 to the atmosphere, compressing the nitrogen in the compression chamber.

6.4.2.4 Record the pressure, P_N, of the compressed (condensed) nitrogen on manometer M_2.

6.4.2.5 Record the liquid nitrogen temperature, T_c, as determined from Fig. 2.

6.4.3 Adjust the mercury level in M_1 to zero reference and record the room temperature, T_{R2}, and the pressure of gas, P_3.

6.5 *Addition of Nitrogen:*

6.5.1 Slowly open S_7 and evacuate the free volume to 10^{-5} torr (1.33 mPa).

6.5.2 Close S_6 and S_7, return the mercury level of the buret to zero reference, and slowly add nitrogen from the storage bulbs.

6.5.3 Expand the gas by lowering the mercury level in the gas buret and adjust the mercury level in M_1 to zero reference.

6.5.4 Record the buret volume, V_{B1}, the gas pressure, P_4, the buret temperature, T_{B4} and the room temperature, T_{R4}.

6.6 *Nitrogen Adsorption:*

6.6.1 With the sample tube immersed in liquid nitrogen, return the mercury level of the buret to zero reference and slowly open S_6.

6.6.2 Adjust the mercury level in M_1 to zero reference and record the buret temperature, T_{B5}, the room temperature, T_{R5}, and the pressure of the gas, P_5.

6.6.3 Expand the gas by lowering the mercury level in the gas buret and adjust the mercury level in M_1 to zero reference.

6.6.4 Record the buret volume, V_{B2}, the gas pressure, P_5, the buret temperature, T_{B5}, and the room temperature, T_{R5}.

6.6.5 Repeat steps 6.6.3 and 6.6.4 to obtain a minimum of three measurements within the range of pressure ratios:

$$(P_5/P_N) = 0.05 \text{ to } 0.35$$

NOTE 2—Volumes and amounts of sample should be chosen carefully so as to obtain pressure ratios covering most of this range. In order to obtain a reasonable estimate of the precision of fitting the line plotted in accordance with 7.5.1.1, six or more measurements are needed within this range.

7. Calculation

7.1 Calculate the line volume, V_1, as follows:

$$V_1 = \left(\frac{P_2}{P_1 - P_2}\right)\left(\frac{T_{R1}}{T_{B1}}\right)V_{B1}$$

NOTE 3—Temperatures are in kelvins.

7.2 Calculate the sample chamber volume, V_C, as follows:

$$V_C = \left(\frac{P_1 - P_3}{P_3}\right)\left(\frac{T_C}{T_{R2}}\right)V_1$$

7.3 Calculate the total volume (V_{STP}) of nitrogen introduced at standard temperature and pressure, as follows: